Détermination de la broyabilité des plaquettes et des pellets de bois

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Résumé

La littérature concernant la consommation énergétique du broyage dans l'industrie qui traite les minerais montre que cette problématique a été, et est encore, étudiée par de nombreux auteurs. Différents modèles mathématiques décrivant le broyage sont donc disponibles pour exprimer l'énergie nécessaire à la réduction granulométrique des minerais. Le broyage de la biomasse, préalable à une utilisation énergétique, quant à lui a été bien moins étudié. Quelques études ont néanmoins porté sur la mesure des consommations énergétiques engendrées par le broyage de matières végétales particulières, dans des systèmes particuliers. Rares sont les auteurs qui prennent en compte suffisamment de paramètres relatifs à la matière entrante et sortante (granulométrie, humidité, origine…). De plus, les méthodes utilisées pour mesurer les propriétés des matières considérées sont généralement différentes d'une étude à l'autre. En conséquence, pratiquement aucun modèle de broyage n'a été proposé pour la biomasse et concernant le broyage des pellets de bois aucune donnée ne semble disponible.

Cette étude s'inspire des théories du broyage utilisées par de l'industrie traitant les minerais (théories de Von Rittinger, Kick et Bond, rassemblées en une relation unique grâce aux travaux de Charles et Hukki) pour définir une méthode de caractérisation de la consommation énergétique induite par le broyage des plaquettes et des pellets de bois. De même, les caractéristiques de la matière qui sont à prendre en considération et les méthodes pour les mesurer, sont définies en vue de leur utilisation dans un modèle de la consommation du broyage de plaquettes et de pellets de bois.

Il est montré que, dans un broyeur de laboratoire, la consommation énergétique des plaquettes de bois dépend de trois facteurs principaux : l'humidité de la matière, la différence de granulométrie entre alimentation et produit broyé et la matière elle-même. Cette dernière peut être caractérisée par un paramètre de broyabilité unique, basé sur une adaptation de la constante de Von Rittinger.

Une relation caractérisant la consommation énergétique des pellets de bois en fonction des distributions granulométriques des constituants des pellets et des pellets broyés est proposée également. Elle est caractéristique de chaque type de pellets pour chaque humidité considérée.

Michaël Temmerman (2013). Grindability determination for wood chips and pellets (Philosophical dissertation in French). University of Liege, Gembloux Agro-Bio Tech.

Abstract

The literature about energy requirements for product milling in mining industry shows the subject has been, and still is, considered by numerous authors. Several milling theories have been proposed for these industries, especially concerning ores milling. Biomass milling, prior to its use as biofuel, has been, by far, less studied. Nevertheless, few measurements

are available about energy needed for milling of particular biomass, in particular systems. But studies taking into account enough characteristic of the milled material (origin, moisture content, particle size distribution) are scarce. Moreover, the methods used to measure these properties are generally different. In consequence, nearly none biomass milling model has been proposed. Concerning wood densified product (pellets and briquettes) apparently no data are available yet.

This study draws upon the milling theories developed for the ore processing industry (Von Rittinger, Kick and Bond theories which have been merged by Charles and Hukki) in order to define a method for characterizing wood chip and pellet energy consumption during milling. Thus it proposes parameters and measurement methods that have to be taken into account when milling modelling comes to an end for biomass or densified biomass.

Energy consumption during wood milling depends on three main factors: the material moisture content, the particle size difference between the feed and the milled product, and the material itself. The latter may be characterized by a single grindability parameter based on an adaptation of Von Rittinger's constant.

A relation characterizing wood pellet energy consumption as a function of the particle size distribution of the pellet ingredients and the milled pellets is proposed as well. This is characteristic of each type of pellet for each moisture content value considered.

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Chapitre 1
Introduction générale

Le présent document se compose de trois parties principales. La première consiste en un état de l'art qui présente le contexte de l'étude et permet de délimiter ses objectifs. Ce volet synthétise et analyse les informations scientifiques disponibles à propos de la consommation énergétique induite par le broyage de minerais et par celle liée au broyage de la biomasse. Cette consommation énergétique, ramenée à l'unité de masse de matière broyée, est appelée broyabilité. La caractérisation des méthodes de mesure des principales propriétés de la biomasse qui peuvent influencer son broyage fait l'objet de la seconde partie du document. Enfin, dans la troisième partie, ces données sont utilisées pour modéliser la consommation énergétique du broyage des plaquettes et des pellets de bois.

La première partie de ce document débute par le recensement des lois et des modèles qui expliquent la consommation énergétique du broyage des minerais. L'analyse identifie ensuite les concepts qui pourraient être transposés à la caractérisation du broyage de la biomasse. Ensuite, cet état de l'art recherche des lois ou des modèles similaires qui pourraient caractériser le broyage de la biomasse, du bois ou des pellets. En réalité, ces références se limitent souvent à des mesures ponctuelles. En outre, elles sont bien moins nombreuses, plus disparates et difficilement exploitables, que celles publiées à propos des minerais. La diversité des données recensées pour la biomasse provient notamment de la diversité des propriétés considérées pour expliquer la consommation énergétique du broyage. Certaines de ces propriétés apparaissent néanmoins essentielles, ce sont, pour la biomasse en général, l'humidité, la granulométrie de l'alimentation, la granulométrie du produit et la matière elle-même. À celles-ci s'ajoutent, pour les pellets, la durabilité, la masse volumique nette et la granulométrie des constituants.

À la diversité des propriétés considérées pour expliquer la consommation énergétique du broyage de la biomasse s'ajoute la diversité des méthodes utilisées, par les auteurs, pour les caractériser. En conséquence, la seconde partie de cette thèse s'attache à sélectionner les méthodes les mieux adaptées à la mesure des propriétés identifiées, au terme de l'analyse bibliographique, pour leur impact probable sur la consommation énergétique du broyage. En effet, la même propriété peut être déterminée à l'aide de méthodes de mesure ou de procédures différentes qui conduisent généralement à des résultats différents en valeur ou en variabilité.

Ainsi, l'humidité des biocombustibles solides est traditionnellement mesurée à l'étuve, un échantillon est placé dans des conditions de température et de ventilation constantes jusqu'à ce que sa masse n'évolue plus significativement et qu'il puisse être considéré comme anhydre. Cette procédure prend du temps, un délai minimum de 24 h est en général nécessaire pour obtenir la mesure de l'humidité. Cependant, des méthodes de mesure plus rapides, quasi instantanées, existent. Elles sont basées sur l'analyse du spectre proche infrarouge ou sur la mesure de la conductivité électrique de l'échantillon.

La granulométrie des biocombustibles se détermine généralement par tamisage. Un échantillon dont l'humidité s'est équilibrée avec celle de l'air est placé au sommet d'une série de tamis empilés, eux-mêmes placés sur un système vibrant. Les fractions

obtenues au terme de la séparation sont exprimées sous forme de distribution de fréquences relatives, ou de distribution de fréquences relatives cumulées, obtenues sur base des fractions massiques. La moyenne et les différents quantiles des distributions obtenues peuvent être calculés. Plusieurs principes de tables vibrantes sont disponibles dans le commerce, affichant des vibrations à une, deux ou trois dimensions. Il existe également des tamis rotatifs (à l'état de prototype) et des systèmes de mesure de la granulométrie par analyse d'image. Ces différentes alternatives ont été comparées entre elles et à une méthode de classification de référence (par mesure manuelle des trois dimensions des particules).

La masse volumique nette des pellets se heurte à la difficulté de mesurer avec précision le volume des pellets constituant l'échantillon. Différentes méthodes ont été comparées : mesures manuelles au pied à coulisse, détermination par déplacement de liquides et par déplacement de solides. Pour les méthodes nécessitant de plonger les pellets dans un liquide, l'opportunité de les hydrofuger à l'aide de paraffine a été envisagée, de même que l'intérêt d'ajouter un agent mouillant à l'eau dans laquelle seront plongés les échantillons.

La durabilité des pellets est une expression qui caractérise la résistance de ces produits densifiés lorsqu'ils sont soumis à des chocs répétés. Dans ce cas, deux principes de mesures principaux ont été comparés. Dans le premier cas, les pellets sont soumis à des chocs répétés par la mise en rotation, autour d'un axe, de la partie d'équipement qui les contient. Dans le second cas, les pellets sont mis en mouvement par soufflerie.

Lorsqu'ils sont plongés dans l'eau, les pellets se désagrègent en particules dont la distribution granulométrique peut être caractérisée, après séchage et mise à l'équilibre, à l'aide d'un jeu de tamis identique à celui utilisé pour déterminer la distribution granulométrique des particules d'autres biocombustibles solides. Pour certains pellets, la désagrégation dans de l'eau à température ambiante est lente ou incomplète. Des méthodes alternatives ont donc été comparées.

Afin de sélectionner, sur une base chiffrée et objective, les méthodes les plus adaptées à la caractérisation des échantillons constitutifs de l'expérimentation sur le broyage, des essais inter-laboratoires ont été organisés, en partenariat avec plusieurs unités de recherche, dans différents pays européens. Ces tests circulaires ont permis de déterminer la répétabilité et la reproductibilité des méthodes testées. Ces critères sont en effet essentiels à une sélection raisonnée de méthodes de mesure à utiliser. Même s'il reste nécessaire de considérer la faisabilité pratique des mesures.

La répétabilité est l'étroitesse de l'accord entre des résultats (d'essais ou de mesures) indépendants obtenus par la même méthode sur des individus identiques, dans des conditions de répétabilité. Ces conditions imposent que les essais soient réalisés sur la même installation (dans le cadre de cette thèse il s'agit du laboratoire), par le même opérateur, sur le même équipement, avec la même procédure et pendant un intervalle de temps court. La répétabilité peut alors s'exprimer quantitativement, par exemple sous forme d'écart-type de répétabilité, obtenu sur base des résultats d'essais, ou de mesures, pratiqués dans ces conditions. Cet écart-type permet de calculer une limite de répétabilité (r) qui est la valeur au-dessous de laquelle est située, avec une probabilité de 95 %, la valeur absolue de la différence entre deux résultats d'essais obtenus sous les conditions de répétabilité.

La reproductibilité se définit et s'exprime, *mutatis mutandis*, de la même manière que la répétabilité, mais dans des conditions de reproductibilité. Celles-ci stipulent que les résultats d'essais sont obtenus par la même méthode sur des individus identiques, sur

différentes installations d'essais (ici, laboratoires), avec différents opérateurs et utilisant des équipements différents. La limite de reproductibilité est notée *R*.

Fort de ces différentes méthodes, il est possible de caractériser les propriétés des plaquettes et des pellets de bois à utiliser dans la troisième partie de cette thèse. Cette dernière partie a pour objectif d'identifier le modèle de consommation énergétique du broyage utilisé dans l'industrie traitant des minerais, le plus adapté aux plaquettes et aux pellets de bois. Pour ce faire, les théories de Von Rittinger, de Kick et de Bond (unifiées par la relation de Charles) sont considérées. Ces modèles présentent l'avantage de ne considérer qu'un nombre limité de paramètres : la matière et sa granulométrie, avant et après broyage. De plus, ces paramètres sont associés à une consommation énergétique par unité de masse grâce à une relation simple.

L'objectif de cette étude est aussi de proposer une méthode de mesure de la broyabilité de la biomasse. Cette méthode est conçue afin de pouvoir fournir aux praticiens de la biomasse des valeurs de référence et les indications nécessaires au dimensionnement d'installations nouvelles ou à la comparaison d'installations existantes. La nécessaire souplesse exigée par l'expérimentation implique un équipement de petite échelle, un broyeur de laboratoire sera donc préféré à des mesures en production. Toutefois le principe de fonctionnement du broyeur utilisé est similaire à ceux utilisés en pratique : un broyeur à marteaux, de faible puissance, aux grilles interchangeables à mailles de diamètres différents. Ce broyeur est équipé d'un dispositif de mesure de la consommation électrique et d'un dispositif de récolte de la matière broyée.

Chapitre 2
Énergie nécessaire au broyage de la biomasse et des produits densifiés[1]

La littérature concernant la consommation énergétique du broyage dans les industries minières montre que cette problématique a été, et est encore, étudiée par de nombreux auteurs. Différents modèles de broyage sont donc disponibles pour ces industries, en particulier pour le broyage des minerais. Les principaux modèles et certaines de leurs évolutions sont repris dans la présente revue. Le broyage de la biomasse quant à lui a été bien moins étudié. Quelques études ont néanmoins porté sur la mesure des consommations énergétiques engendrées par le broyage de biomasses particulières, dans des systèmes particuliers. Rares sont celles qui prennent en compte suffisamment de paramètres relatifs à la matière entrante et sortante (granulométrie, humidité, origine, etc.) ou les caractéristiques du système de broyage. Pratiquement, aucun modèle de broyage de la biomasse n'a donc été proposé. Concernant le broyage des produits densifiés, aucune donnée ne semble disponible. À la lueur des théories de broyage de l'industrie minière, cette étude propose donc les paramètres à prendre en compte pour une éventuelle modélisation du broyage de la biomasse et des produits densifiés.
Mots-clés : Biomasse, broyage, fragmentation, pellet, briquette, théories de broyage, énergie.

Milling energy needs for biomass and densified products. The literature about energy requirements for product milling in mining industry shows the subject has been, and still is, considered by numerous authors. Several milling theories have been proposed for these industries, especially concerning ores milling. The main mining milling theories and some of their evolutions are described in this paper. Biomass milling has been, by far, less studied. Nevertheless, few measurements are available about energy needed for milling of particular biomass, in particular systems. But studies taking into account enough characteristics of the milled material (origin, moisture content, particle size distribution) are scarce. In consequence, nearly none biomass milling model has been proposed. Concerning densified products (pellets and briquettes) apparently no data are available yet. Considering the milling theories, this study selects parameters that have to be taken into account when milling modeling comes to an end for biomass or densified biomass.
Keywords: Biomass; Milling; Comminution; Pellets; Briquettes; Milling theories, Energy.

2.1. Introduction

La volonté internationale de réduire les émissions de gaz à effet de serre, en particulier de gaz carbonique fossile, a encouragé de nombreux producteurs d'électricité à convertir certaines de leurs installations utilisant du charbon pulvérisé, ou du gaz, en centrales à co-combustion biomasse. Il existe même un exemple de conversion d'une centrale à l'utilisation exclusive de pellets broyés (Ryckmans et al., 2006). En effet, l'incorporation de biomasse aux combustibles fossiles présente de nombreux avantages. D'un point de vue environnemental, cette option permet, à court terme et sans risque majeur pour l'utilisateur, de réduire les émissions de CO_2 fossile, de SO_x et de NO_x. De plus, il apparait que, techniquement, l'efficacité des

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chaudières peut s'en trouver accrue et que la réduction du cout du combustible améliore la rentabilité des installations (Demirbas, 2003 ; Baxter, 2005 ; Demirbas, 2005).

Les quantités de biomasse nécessaires à l'approvisionnement de telles centrales sont colossales et leur disponibilité à l'échelle locale souvent insuffisante. Ainsi, lorsque sont pris en compte les couts engendrés pour atteindre les objectifs de l'Union européenne en matière de réduction des émissions, la dépendance du continent vis-à-vis des ressources biomasse intercontinentales devient indéniable (Bjerg, 2004 ; Hamelinck et al., 2005). Il demeure cependant rentable, tant économiquement que du point de vue environnemental, de transporter des particules de bois sous forme densifiée, même sur de longues distances (Wahlund et al., 2004 ; Hamelinck et al., 2005).

Les centrales électriques utilisent la biomasse (en général du bois) sous forme de poudre, ce qui nécessite un broyage avant utilisation afin d'obtenir des particules de bois aux qualités aérodynamiques suffisantes pour être utilisées dans le process. Dans ces systèmes, les caractéristiques des particules de biomasse sont d'une importance capitale, elles influencent en effet l'alimentation, la cinétique de combustion, la quantité d'imbrûlés et la température du foyer (Tmej et al., 2000 ; Paulrud et al., 2004 ; Nishiyama et al., 2007 ; Rhén et al., 2007).

Pour la plupart de ces unités de co-combustion, le cahier des charges impose que la totalité des particules passent au travers d'une maille de 6,34 mm et que la majorité soient d'une taille de 3 mm (Baxter, 2005). Cependant, les spécifications de certains consommateurs sont parfois plus strictes (Esteban et al., 2006a ; Ryckmans et al., 2006).

Néanmoins, le broyage des pellets peut s'avérer problématique. En effet, les données manquent pour pouvoir dimensionner le poste broyage lors de la conception des chaines d'approvisionnement des centrales électriques. Inversement, dans les chaines existantes, l'acceptation de matières nouvelles passe par des essais pilote en vraie grandeur. D'une manière générale, c'est le broyage de la biomasse dans son ensemble qui reste peu étudié.

Cette étude tente donc de dégager les pistes qui serviront à caractériser les besoins énergétiques du broyage de biomasse solide, en particulier des pellets. Pour ce faire, les connaissances en matière de broyage de la biomasse sont évaluées à la lumière de ce qui est pratiqué dans d'autres domaines d'activités, comme les industries agro-alimentaires et l'industrie minérale. Enfin, l'intérêt d'établir une relation entre les propriétés physiques et mécaniques des biocombustibles solides et l'énergie nécessaire à leur broyage sont évalués.

2.2. Les théories du broyage de l'industrie minérale

2.2.1. Les aspects qualitatifs

Les théories de broyage ont avant tout été développées grâce aux travaux effectués par les industries minérales. La variabilité des matières issues du monde vivant, de la biomasse, pourrait être plus importante que celle des matériaux pour lesquels ces théories ont été élaborées. Cependant, certaines applications ont été développées pour des industries agro-alimentaires (Chamayou et al., 2003) qui permettent de penser qu'elles seront également applicables aux biocombustibles solides.

Le broyage vise d'abord à fragmenter la matière pour en réduire la taille afin de lui conférer une forme utilisable. Les théories de broyage considèrent que des forces de contact sont appliquées sur un volume solide et créent dans celui-ci un réseau de

fissures qui conditionneront la taille et la forme des fragments résultant de la rupture du matériau. Le résultat d'une opération de broyage est donc la conséquence d'une part, des propriétés du solide broyé et d'autre part, de la nature, de la répartition et de l'intensité des contraintes que lui applique le broyage.

Le choix d'un broyeur dépend principalement de trois facteurs : la nature de la matière première, la distribution granulométrique du produit que l'on cherche à obtenir et le volume de production que l'on recherche. Dans l'industrie minérale, cinq caractéristiques principales sont généralement retenues pour caractériser la matière à broyer : la dureté classiquement représentée sur l'échelle de Mohs, l'abrasivité, l'adhésivité, la forme et la distribution granulométrique.

2.2.2. Les lois de broyage

La théorie de la fragmentation repose sur la relation liant l'énergie consommée et la taille des particules du produit obtenu à partir d'une taille donnée de l'alimentation. Dans ce qui suit, E est l'énergie de broyage nécessaire par unité de masse broyée (l'énergie spécifique) ; x une caractéristique granulométrique (par exemple q_{80} – la taille de maille laissant passer 80 % de la matière mesurée – le quartile 80 de la distribution), n est un exposant exprimant la grandeur du processus, C est une constante caractérisant le matériel à broyer dont les unités équilibrent l'équation.

Historiquement, en matière de broyage, de nombreuses théories ont été proposées (Masson, 1960). Cependant, trois d'entre elles méritent une attention particulière pour avoir traversé les générations et être encore citées, à l'heure actuelle, dans bien des publications.

Les trois piliers de la théorie de broyage

Loi de Von Rittinger (1867). C'est la plus ancienne. Elle établit que l'aire de la nouvelle surface produite par le broyage est directement proportionnelle à l'énergie nécessaire au broyage. L'aire de la surface d'une masse de particules de diamètre uniforme x étant proportionnelle à $1/x$, l'énergie nécessaire au broyage y est donc proportionnelle également. Elle est exprimée par la formule générale :

$$E_{1\text{-}2} = C_{VR} \left(\frac{1}{x_2} - \frac{1}{x_1} \right) \qquad (1)$$

où x_1 et x_2 sont respectivement l'état granulométrique de la matière avant et après broyage et C_{VR} une constante caractéristique de la matière.

Loi de Kick (1885). Kick, pour sa part, émet le postulat que le travail requis est proportionnel à la réduction de volume des particules en cause. L'équation qu'il propose est la suivante :

$$E_{1\text{-}2} = C_K \left(\ln (x_1) - \ln (x_2) \right) \qquad (2)$$

où x_1 et x_2 sont respectivement l'état granulométrique de la matière avant et après broyage et C_K une constante caractéristique de la matière. Le rapport x_1/x_2 est parfois appelé le rapport de réduction.

Loi de Bond (1952). D'un point de vue physique, la théorie de Bond (Bond, 1952 ; Bond, 1961) suppose que l'énergie transmise à un corps par effort de compression se répartit d'abord dans la masse et est proportionnelle à x_1^3, mais que dès le début de la fissuration en surface, cette énergie se concentre sur les fissures et devient alors proportionnelle à x_1^2. Elle conduit donc à admettre que le travail de broyage proprement dit est intermédiaire entre x_1^2 et x_1^3.

D'après cette théorie, pour des particules de forme semblable, la longueur de fissure est équivalente à la racine carrée de la surface. L'énergie nécessaire au broyage étant proportionnelle à cette longueur de fissure, elle s'écrit comme suit :

$$E_{1\text{-}2} = C_k \left(\frac{1}{\sqrt{x_2}} - \frac{1}{\sqrt{x_1}} \right) \quad (3)$$

où x_1 et x_2 sont respectivement l'état granulométrique de la matière avant et après broyage et C_K une constante caractéristique de la matière.

En pratique, ce sont les quartiles 80 (aussi appelés d_{80}) qui sont utilisés pour décrire les états granulométriques x_1 et x_2. Cette valeur peut être définie comme la dimension de la maille du tamis qui autorise 80 % de passant. Si P et F sont respectivement les quartiles 80 du produit broyé et de l'alimentation, l'énergie consommée en kWh par unité de masse peut s'exprimer par l'équation :

$$W = W_i \left(\frac{10}{\sqrt{P}} - \frac{10}{\sqrt{F}} \right) \quad (4)$$

où W est l'énergie spécifique réellement consommée par un circuit de broyage et W_i est l'indice de Bond ou « *Work index* » caractéristique de la matière et du circuit de broyage. Le succès de la loi de Bond s'explique notamment parce que ces paramètres sont aisément mesurables en industrie. Mais W_i peut aussi être calculé à partir de mesures réalisées dans un broyeur de laboratoire dimensionné par Bond à cet effet.

La synthèse et l'évolution des lois empiriques

Les lois de Von Rittinger, Kick et Bond seront rassemblées par Charles (Charles, 1957) qui constate que les relations établies entre l'énergie E et l'état granulométrique x par ces trois auteurs sont des formes particulières d'une même équation différentielle :

$$dE = - C \frac{dx}{x^n} \quad (5)$$

l'exposant n de x prenant respectivement pour les lois de Von Rittinger, Bond et Kick les valeurs 2, 1,5 et 1.

Hukki (1962) montrera que chacune de ces valeurs correspond à une plage granulométrique relativement restreinte et que l'exposant n'est pas constant, mais est dépendant du niveau granulométrique x lui-même, comme exprimé par l'équation :

$$dE = - C \frac{dx}{x^{f(x)}} \quad (6)$$

Morell (2004) rappelle les travaux cités ci-dessus et ceux de Griffith (1920), Weibull (1939) et Rumph (1973) qui montrent que pour les minerais, lorsque la taille des particules diminue, le nombre et la densité des imperfections et des fissures diminuent aussi. Il postule ensuite que la constante C est elle-même dépendante de la granulométrie. Morrell montre aussi que cette fonction, notée $g(x)$, n'est pas la même pour toutes les roches :

$$dE = - C\, g(x) \frac{dx}{x^{f(x)}} \quad (7)$$

Or, en pratique, la détermination de ces deux fonctions se révèle particulièrement difficile. Morrell propose donc une formulation alternative pour la détermination de la consommation énergétique au broyage :

$$W = M_i K \left(x_2^{f(x2)} - x_1^{f(x2)} \right) \quad (8)$$

où W est l'énergie spécifique (en $kWh \cdot t^{-1}$), K une constante, M_i un indice relatif à la propriété de fracture de la matière, x_1 et x_2 les quartiles 80 de l'alimentation et du produit.

L'auteur observe que la forme de $f(x)$ donnant les meilleurs résultats est :

$$f(x) = - (a + x^b) \quad (9)$$

où a et b sont des constantes et x le quartile 80.

Les lois empiriques restent d'actualité

Malgré l'évolution des théories du broyage, l'indice de Bond fait encore couler beaucoup d'encre, notamment lorsqu'il s'agit d'estimer l'efficacité du circuit de broyage (Sachihito et al., 2002 ; Coello Velazquez et al., 2008 ;

Tromans, 2008 ; Ahmadi et al., 2009). En effet, dans l'industrie minérale, le rapport entre le W mesuré sur site et le W_i mesuré en laboratoire est souvent utilisé pour estimer l'efficacité d'un circuit de broyage, même si cela peut conduire à des approximations.

Stambioliadis (2002) propose une relation où l'influence de la distribution granulométrique des particules est exprimée sous la forme d'une distribution théorique de fréquence. C'est celle de Gates Gaudin Schumann qui est utilisée. Cette relation permet donc de déterminer l'énergie nécessaire au broyage à partir d'une distribution granulométrique théorique et de la relation de Charles (5). L'auteur montre également, à partir de données disponibles dans la littérature, que plus la granulométrie est fine, plus la consommation énergétique est importante.

Dans l'optique de leur application au broyage de la biomasse, ces travaux présentent l'intérêt d'estimer la consommation d'énergie d'un circuit de broyage à partir d'un petit nombre de paramètres. Elles sont applicables à des matières très diverses, du moins du point de vue dureté. Par contre, elles ont été développées pour des broyeurs caractéristiques de l'industrie minérale, les broyeurs à boulets et à barre, principalement. Il n'en reste pas moins qu'elles indiquent qu'une relation entre l'énergie consommée par le broyage d'une matière et la granulométrie du produit et de la matière première peut être recherchée.

2.2.3. Les méthodes de mesure des indices de broyabilité

De nombreux tests ont été développés pour caractériser la broyabilité, jusque récemment, chaque test étant généralement développé pour une application particulière. Werner et al. (1999) développent un broyeur de laboratoire destiné à mesurer le comportement au broyage de différentes origines de charbon. Lecoq et al. (1999) utilisent un broyeur à jet d'air pour évaluer la relation entre l'énergie d'impact et la broyabilité des solides projetés.

Plus classiquement, Stambioliadis (2002) recense plusieurs types de tests pour déterminer la relation entre l'énergie de broyage et la granulométrie : les broyeurs sans friction, décrits par Hukki en 1943 ; les tests de chute ou les impacts de projectiles, décrits par Charles en 1957 et les tests de compression développés par Tavares et al. (1998). La relation entre l'énergie de broyage et la distribution granulométrique peut aussi être étudiée à l'aide de broyeurs à barre ou à boulets tels que ceux développés par Bond en 1952 et Charles en 1957.

Ces tests permettent d'estimer l'énergie dépensée pour le broyage à partir du calcul de l'énergie mécanique mise en œuvre.

Pris à la lettre, ces tests sont généralement représentatifs de situations de broyage très particulières. À titre d'exemple, pour son importance historique et sa polyvalence, la détermination en laboratoire du « *Work index* » de Bond suit une procédure très stricte et de nombreux facteurs correctifs sont ensuite nécessaires pour déterminer la puissance du broyeur industriel qui sera installée en pratique (Bond, 1961).

Par contre, le succès de cette théorie s'explique par le fait que le « *Work index* » de Bond peut être déterminé directement sur le site de production. En effet, connaissant les caractéristiques de l'équipement, la puissance consommée, le débit d'alimentation et la granulométrie des produits entrants et sortants, il est possible d'en déduire le coefficient d'aptitude au concassage et au broyage. Connaissant les rendements électriques et mécaniques des différents postes du circuit de broyage concernés, il est possible de calculer W_i.

2.2.4. L'expression de la granulométrie

Bien que d'autres techniques puissent être appliquées (analyse d'image, diffraction laser, etc.), la mesure de la granulométrie de la biomasse se fait encore principalement par tamisage (Hartmann et al., 2006).

Les résultats s'expriment en diagrammes de fréquences et de fréquences cumulées qui sont les représentations les plus intuitives. La distribution cumulée est définie comme la proportion d'observation soit inférieure, soit supérieure à une limite de classe d(i). Si le cumul des proportions s'effectue à partir de la classe de plus petite dimension (en dessous d'une valeur limite), la distribution est dite en passant cumulé. Si, au contraire, le cumul s'effectue à partir de la classe de plus grande dimension (au-dessus d'une valeur limite), la distribution est dite en refus cumulé. L'emploi de distributions en passant ou en refus cumulés est indifférent. De même, les représentations fréquentielles ou cumulées peuvent être utilisées conjointement (Novalès et al., 2003).

Les distributions n'étant pas toujours aisées à manipuler dans leur intégralité, l'information est généralement synthétisée par des paramètres qui décrivent la tendance centrale et la dispersion des distributions, comme en statistique classique.

Le paramètre de tendance centrale a pour objectif de décrire la taille des particules majoritaires dans la population. Les notions de mode, médiane et moyenne sont donc aussi utilisées en granulométrie. Pour exprimer des résultats de tamisage, c'est principalement la médiane qui est utilisée. Elle correspond à la dimension pour laquelle la valeur de la distribution cumulée est de 50 %. Elle est parfois appelée diamètre médian et notée d_{50}.

Les paramètres de dispersion permettent d'obtenir une estimation de la variabilité de la taille. Les intervalles interquartiles sont obtenus à partir de la courbe des fréquences cumulées, de manière similaire à la médiane. On rencontre couramment les valeurs d_{10}-d_{90}, d_{75}-d_{25} et d_{84}-d_{16}. Ce dernier écart correspond à un intervalle de ± 1 écart-type, dans le cas où la distribution suit une loi normale. L'écart peut être donné en valeur relative par rapport à la tendance centrale.

Dans le cas du broyage, le calcul de quartiles, le d_{80}, est particulièrement utile vu qu'il est utilisé pour la détermination du « *Work index* » de Bond.

Les paramètres de tendance centrale et les paramètres de dispersion ne permettent pas de décrire la forme globale de la distribution, c'est pourquoi un ajustement des distributions observées à des modèles de référence est parfois envisagé.

Quatre modèles granulométriques adaptés pour les distributions unimodales sont couramment utilisés : les lois normales, log-normale, de Rosin Rammler et de Gaudin Schuhmann. Les deux premières proviennent de la statistique usuelle, tandis que les deux dernières sont obtenues à partir de modèles construits pour décrire des distributions de produits broyés ou concassés. Elles sont, par exemple, intégrées dans certains modèles de simulation de broyage (Austin, 1964 ; Nikolov, 2002 ; Stambioliadis, 2002 ; Austin, 2004). Cependant, ce mode d'expression de la granulométrie reste très peu utilisé pour caractériser les produits biomasse solide.

2.2.5. La modélisation

Plus récemment, des modèles de broyage ont été proposés, qui estiment la distribution granulométrique du produit à partir des caractéristiques de la matière et du circuit de broyage. Dans des cas simples, la définition d'une fonction de sélection et d'une fonction de broyage permet de modéliser le broyage par une approche de type bilan de population (Chamayou et al., 2003).

Ces modèles ont d'abord pour intérêt d'être construits sur des bases physiques et de permettre la caractérisation granulo-

métrique des produits et des circuits pour lesquels ils sont paramétrés. En effet, les fonctions de broyage et de classification comptent plusieurs facteurs caractéristiques de la matière et ne sont validés que sur certains cas particuliers. Le but principal de ces modèles étant de déterminer la granulométrie du produit sortant, l'énergie nécessaire au broyage n'est considérée que comme un paramètre du modèle, parmi les autres.

2.3. Le broyage de la biomasse

2.3.1. Les broyeurs et la forme de la biomasse

En fonction de la forme sous laquelle se présente la biomasse, plusieurs types de broyeurs peuvent être envisagés (Temmerman et al., 2005). Les broyeurs à disques, à tambours et à vis sont surtout utilisés pour le broyage de grosses branches ou d'arbres entiers. Les broyeurs à dents et à cisaille sont utilisés pour le broyage de déchets biomasse, comme des palettes ou des bois de démolition non traités. À l'exception du broyage d'arbres entiers, les très polyvalents broyeurs à marteaux sont utilisés pour la préparation de la matière, quelle que soit sa forme de départ. Le choix d'un broyeur est également fonction de la quantité de matière à broyer et de la qualité du produit désiré. L'utilisation de grille permet de calibrer la granulométrie du produit, mais influence le débit du broyeur.

2.3.2. La consommation énergétique du broyage de la biomasse

Les publications relatives à la consommation énergétique du broyage de la biomasse sont relativement rares. Mani et al. (2004) citent quelques auteurs ayant étudié le sujet dans les années 1980. Plus récemment, Vigneault et al. (1992) et Rothwell et al. (1992) publient des consommations énergétiques pour le broyage du maïs. Depuis, les publications relatives au sujet sont surtout le fait de trois auteurs, Mani et al. (2004), Esteban et al. (2006a ; 2006b) et Laskowski et al. (1997 ; 1998 ; 1999). Par ailleurs, le développement de la technique de torréfaction du bois et son impact annoncé sur la broyabilité de la biomasse (Arias et al., 2008) offrent quelques mesures de consommation énergétique au broyage de bois (Bergman et al., 2005). Les données publiées par ces différents auteurs sont synthétisées dans le tableau 2.1.

Les études de Vigneault et al. (1992) et Rothwell et al. (1992) portent sur la comparaison des consommations énergétiques du broyage de maïs induites par la forme des marteaux et le type de crible montés sur le broyeur d'une meunerie commerciale. L'étude montre l'influence significative de la forme des marteaux et de leur vitesse sur la consommation énergétique. Toutes autres choses restant égales, les consommations mesurées varient de 10 à 12 $kWh \cdot t^{-1}$ pour des marteaux fins et standards, respectivement. Le type de crible utilisé influence lui aussi la consommation énergétique du broyage.

Laskowski et al. (1997 ; 1998 ; 1999) font la relation entre le comportement de grains en compression et les consommations énergétiques d'un broyeur de laboratoire. La faible taille des échantillons utilisés pour le broyage et le nombre important de variables prises en compte dans le modèle de régression rendent les résultats de ces études difficilement utilisables en pratique. Cependant, ces travaux mettent clairement en évidence l'influence de l'humidité et de la matière broyée sur la consommation énergétique. Pour une matière donnée, plus le grain est humide, plus élevée sera sa consommation énergétique au broyage. Les consommations mesurées s'étendent de 15 à 144 $kWh \cdot t^{-1}$.

Dans son étude ayant pour but d'évaluer les avantages et les inconvénients à combiner

Tableau 2.1. Énergie nécessaire au broyage de diverses biomasses — *Energy needed for different biomass milling*.

Matière	PSD 1 (mm)	PSD 2 (mm)	H1 (%)	H2 (%)	P	En Sp ($kWh \cdot t^{-1}$)	Source
Paille de froment	GM : 7,67	Gd : 0,8 ; 1,6 ; 3,2	NS	8,3	N	11,4 - 51,6	Mani et al., 2004
	GM : 7,67	Gd : 0,8 ; 1,6 ; 3,2	NS	12,1	N	24,7 - 45,3	
Paille d'orge	GM : 20,52	Gd : 0,8 ; 1,6 ; 3,2	NS	6,9	N	13,8 - 53,0	Mani et al., 2004
	GM : 20,52	Gd : 0,8 ; 1,6	NS	12,0	N	27,1 - 99,5	
Tige de maïs	GM : 12,48	Gd : 0,8 ; 1,6 ; 3,2	NS	6,2	N	7,0 - 22,1	Mani et al., 2004
	GM : 12,48	Gd : 0,8 ; 1,6 ; 3,2	NS	12,0	N	11,0 - 34,3	
Panic érigé	GM : 7,15	Gd : 0,8 ; 1,6 ; 3,2	NS	8,0	N	23,8 - 62,6	Mani et al., 2004
	GM : 7,15	Gd : 0,8 ; 1,6 ; 3,2	NS	12,0	N	27,6 - 58,5	
Peuplier	Q_{50} : 8,0	Q_{95} : 1 mm	11,8	8,1	G	82,6 - 89,1	Esteban et al., 2006a
Pin	Q_{50} : 9,2	Q_{95} : 1 mm	Δ H1 - H2 = 34		G	113,2 - 119,1	Esteban et al., 2006a
Écorces de pin	Q_{50} : 8,25	Q_{95} : 1 mm	Δ H1 - H2 = 9		G	18,1 - 23,6	Esteban et al., 2006a
Peuplier	NS	MS : 0,2 - 1,2	13	NS	N	20,0 - 75,0	Bergman et al., 2005
Peuplier	NS	MS : 0,2 - 1,2	< 1	NS	N	10,0 - 30,0	Bergman et al., 2005
Haricot	NS	Gd : 1	10-18	NS	NS	27,0 - 64,0	Laskowsky et al., 1999
Pois	NS	Gd : 1	10-18	NS	NS	23,0 - 50,0	Laskowsky et al., 1999
Lupin	NS	Gd : 1	10-18	NS	NS	70,0 - 144,0	Laskowsky et al., 1999
Vesce	NS	Gd : 1	10-18	NS	NS	15,0 - 36,0	Laskowsky et al., 1999
Maïs	NS	GM : 0,49 - 0,51	NS	NS	NS	10,0 - 12,4	Vigneault et al., 1992
Chips de pin	8	Gd : 3 ; 4 ; 6	10 - 15	8,6 - 9,3	G	36,0 - 53,0	Esteban et al., 2006b

PSD : distribution granulométrique — *article size distribution* ; H : humidité — *moisture content* ; P : mesure de l'énergie relative à — *energy measurement related to* ; En Sp : quelques données de la littérature — *data from litterature* ; indice 1 : alimentation — *feed* ; indice 2 : produit — *product* ; GM : moyenne géométrique — *geometric mean* ; Gd : grille — *grid* ; Q_{50} : quantile 50 — *quantile 50* ; Q_{95} : quantile 95 — *quantile 95 ;* MS : moyenne — *mean* ; N : biomasse nette — *net biomass* ; G : circuit de broyage global — *global milling circuit* ; NS : non spécifié — *not specified.*

torréfaction du bois avant densification (pelletisation), Bergman et al. (2005) comparent les consommations énergétiques du broyage de bois sec et humide avec celles du bois torréfié, dans un broyeur à couteaux. Selon la granulométrie (dimension moyenne des particules de 0,2 à 1,2 mm), le broyage de bois anhydre consomme de 10 à 30 $kWh \cdot t^{-1}$, tandis que le bois un peu plus humide nécessite une gamme de consommation de 20 à 75 $kWh \cdot t^{-1}$, pour la même gamme de granulométrie. Ces mesures mettent en outre en évidence une tendance nette à de plus fortes consommations énergétiques pour les broyages les plus

fins. Le broyage de bois torréfié demande, pour sa part, moins d'énergie que le bois non traité, toujours sous les 10 kWh·t^{-1}, quel que soit le procédé de torréfaction utilisé et la granulométrie du produit. Dans ce cas aussi, plus la granulométrie est fine, plus le broyage consomme de l'énergie.

Le broyage du bois a été également étudié par Esteban et al. Dans une première étude, concernant l'efficacité énergétique d'une chaine de production de pellets, Esteban et al. (2006b) réalisent des tests de broyages. La matière première est constituée de plaquettes de rémanents forestiers de pin sylvestre conditionnés à une humidité homogène comprise entre 10 et 15 % et ayant subi un premier broyage en broyeur à marteaux équipé d'une grille perforée de maille au diamètre de 8 mm. Cette matière est ensuite reprise pour être affinée alternativement à l'aide du même broyeur équipé de grilles de 6, 4 ou 3 mm. Les consommations énergétiques globales de chaque broyage sont mesurées, elles s'étendent de 29 à 53 kWh·t^{-1}.

Dans une seconde étude, Esteban et al. (2006a) comparent l'efficacité de différents circuits de broyage, notamment du point de vue de leur consommation énergétique. Trois matières aux distributions granulométriques très semblables sont testées : des chips de peuplier (Q_{50} : 8 mm), des chips de pin (Q_{50} : 9,3 mm) et de l'écorce de pin (Q_{50} : 8,25 mm). Douze circuits de préparation de la matière (en une ou plusieurs étapes) sont testés avec l'objectif de produire des particules utilisables en brûleur à combustibles pulvérisés (Q_{95} : 1 mm et Q_{12} : 0,125 mm). Les consommations énergétiques globales des différentes alternatives sont mesurées et comparées entre elles. Il faut noter que parmi les alternatives testées, peu permettent d'atteindre l'objectif granulométrique fixé. Cependant, la consommation énergétique des différentes combinaisons matière – process de préparation varie davantage d'une matière à l'autre que d'un procédé à l'autre. Ainsi, la moyenne des consommations énergétiques globales de tous les procédés testés pour le broyage de peuplier est de 85,4 kWh·t^{-1} (avec un écart-type de 4,9 kWh·t^{-1}), celle relative au chips de pin est de 118,5 kWh·t^{-1} (l'écart-type est de 6,2 kWh·t^{-1}) et celle de l'écorce de pin est de 19,7 kWh·t^{-1} (l'écart-type est de 3,9 kWh·t^{-1}). D'autre part, les auteurs notent une forte réduction de l'humidité de la matière due au broyage, pour les trois matières testées.

Mani et al. (2004) étudient les consommations énergétiques liées au broyage de biomasse. Ces auteurs considèrent différents facteurs comme ayant une influence possible sur la consommation énergétique au broyage. Parmi les propriétés de la matière, sont citées, la matière elle-même, l'humidité, la granulométrie (avant et après broyage), la masse volumique en vrac, la masse volumique nette. L'installation elle-même peut avoir une influence également, le type de broyeur utilisé, ses réglages, le débit d'alimentation, etc.

À l'aide d'un broyeur de petite puissance, Mani et al. étudient la consommation énergétique nette du broyage de quatre matières (la paille de froment, la paille d'orge, les tiges de maïs et le panic érigé) dont la granulométrie est exprimée au moyen de la moyenne géométrique (panic érigé : 7,15 mm ; paille de froment : 7,67 mm ; tige de maïs : 12,48 mm ; paille d'orge : 20,52 mm). Trois diamètres de grille sont utilisés pour le broyage de ces biomasses, elles sont conditionnées avant broyage à deux niveaux d'humidité (environ 8 et 12 %). Les consommations mesurées varient de 11,04 kWh·t^{-1} à 62,55 kWh·t^{-1} en fonction des différents facteurs d'influence considérés ci-dessus. Ces résultats sont illustrés en figure 2.1. Sur base de ces données, les auteurs définissent des relations entre dimension de la grille du broyeur et consommation énergétique, elles sont linéaires pour les matières les plus sèches et polynomiales pour le niveau d'humidité le plus élevé.

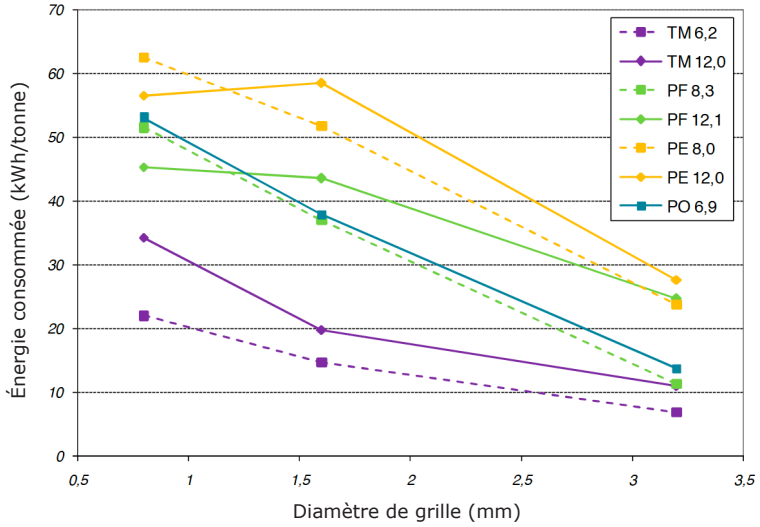

Figure 2.1. Consommation énergétique nécessaire au broyage de différentes biomasses, à deux niveaux d'humidité et trois diamètres de grille de broyeur (données d'après Mani et al., 2004) — *Energy consumption needed for biomass milling, at two moisture content levels, for three hammermill grid diameters (data from Mani et al., 2004).*

PF : Paille de Froment — *Wheat Straw* ; PO : Paille d'Orge — *Barley Straw* ; TM : Tiges de Maïs — *Corn Stover* ; PE : Panic Érigé — *Switchgrass* ; ces notations sont suivies de la valeur de l'humidité de la matière — *notes are followed by material moisture content value.*

Mani et al. sont probablement les premiers à formaliser la relation entre l'énergie consommée et une expression de la granulométrie du produit (le diamètre de grille), pour du broyage biomasse. Cette publication est donc une référence pour de nombreux auteurs et est même qualifiée de « *… probably the best available in the literature …* » par Hosseini et al., 2009. Pourtant, ces résultats soulèvent quelques questions.

Premièrement, pourquoi deux des matières humides consomment-elles moins d'énergie pour le broyage fin humide que pour le sec ? Ces deux valeurs vont à l'encontre de la tendance dessinée par les autres mesures qui semblent indiquer que le broyage d'une biomasse humide est plus énergivore que celui de la biomasse sèche. Tendance par ailleurs confirmée par Bergman et al. (2005).

Ensuite, pourquoi la relation qui semble unir diamètre de grille et consommation énergétique est-elle linéaire ? Pourtant, les théories du broyage issues de l'industrie minérale indiquent une tendance exponentielle de la consommation énergétique en fonction de la diminution de la granulométrie de produit. Tendance également observée dans Bergman et al. (2005) et Esteban et al. (2006b), une partie de réponse réside probablement dans l'étroitesse de la gamme de granulométrie testée.

De plus, il semble réducteur d'estimer la granulométrie du produit sur base du diamètre de grille du broyeur. Enfin, pourquoi la granulométrie de départ semble-t-elle ne pas influencer la consommation énergétique au broyage ?

2.4. Quelles propriétés de la biomasse pour caractériser son comportement au broyage ?

Le tableau 2.2 indique, pour chaque facteur d'influence potentiel, son importance au regard des théories du broyage de l'industrie minérale et par rapport aux informations disponibles à propos du broyage de biomasse. En fonction de ces deux points de vue, l'importance présumée pour le broyage de la biomasse et des pellets est hiérarchisée. Des scores sont accordés à chaque propriété, ils s'étendent de 0 à 3, en fonction de leur importance au regard du broyage. Ils sont fixés de manière empirique en fonction des éléments exposés ci-dessus.

La matière broyée, son origine, influence le broyage, elle conduit à une relation entre consommation énergétique et granulométrie qui lui est propre, pour un système donné. Ceci a été montré pour les minerais, peu pour la biomasse. En effet, le faible nombre de données publiées et la diversité des critères considérés, pour caractériser la biomasse broyée, pourraient être la cause de différences attribuées à ce facteur. Par exemple, la granulométrie de la matière de départ n'est pas toujours mesurée et lorsqu'elle l'est, son écart par rapport à la granulométrie du produit n'est pas pris en compte dans l'expression des résultats. Or, cet écart granulométrique entre l'état initial et final de la matière est la base même des théories de broyage de l'industrie minérale. En conséquence, une étude du broyage de la biomasse devra prendre en compte cet aspect. Un coefficient 3 est donc attribué à ce facteur, quel que soit le secteur de broyage considéré.

Les broyeurs à marteaux équipés de grilles se prêtent bien au broyage de la biomasse et constituent donc l'essentiel des données de la littérature. Cette situation semble conduire la majorité des auteurs à exprimer la granulométrie du produit par la dimension des grilles utilisées pour broyer la matière entrante. Strictement considérées, ces données sont représentatives de l'énergie consommée avec un diamètre de grille, mais pas d'une granulométrie donnée, même si le

Tableau 2.2. Estimation du degré d'importance des facteurs d'influence sur la consommation énergétique au broyage pour les minerais, la biomasse et les pellets — *Ranking estimation of factors influencing the milling energy consumption for ores, biomass and pellets.*

Propriété	Industrie minérale	Broyage de biomasse	Broyage des pellets
L'origine / la matière	3	3	3
L'humidité	0	3	3
La masse volumique en vrac	1	1	1
La masse volumique unitaire	2	1	2
Granulométrie de l'alimentation	3	1	3
Granulométrie du produit	3	3	3
Résistance à la compression	0	1	1
La durabilité	NA	NA	3
Granulométrie des constituants des pellets	NA	NA	3
Type de broyeur	1	1	1
Type de grille	1	3	3
Débit d'alimentation	1	1	1

0, 1, 2, 3 : score (sans unité) indiquant un degré croissant d'influence sur la consommation énergétique du broyage — *score (dimensionless) indicating an increasing influence of the parameter on the milling energy consumption* ; NA : sans objet — *not applicable.*

diamètre des mailles d'une grille influence évidemment la distribution granulométrique. Ce facteur se voit néanmoins attribué le score de 3, pour le broyage de la biomasse et des pellets.

L'humidité est probablement le premier facteur d'influence additionnel à prendre en compte lorsque l'utilisation de matières biologiques, de biomasse, est étudiée. Le broyage ne fait pas exception à la règle. Peu de données sont pourtant disponibles à ce sujet et elles sont parfois contradictoires. Il semble néanmoins justifié de considérer que plus une matière est humide, plus elle consommera d'énergie pour être broyée, *a fortiori* si cette énergie est rapportée à la matière sèche broyée. Pour le broyage de la biomasse et celui des pellets, le score 3 a donc été attribué.

Si la masse volumique en vrac est souvent mesurée dans les travaux étudiant le broyage, c'est rarement pour montrer son influence sur la consommation énergétique. C'est davantage son rapport au diamètre des grilles du broyeur qui est mis en avant. La variabilité de la méthode de mesure (Böhm et al., 2004) rend très aléatoire l'utilisation de cette propriété dans un modèle. Le score attribué à cette propriété est donc de 1.

Aucune information n'est par contre disponible concernant l'influence dans la masse volumique unitaire sur le broyage de la biomasse, un score 1 a donc été attribué. Son utilisation dans les modèles de simulation de broyage de l'industrie minérale justifie un score 2 pour ce secteur de broyage, score qui a influencé celui attribué pour le broyage des pellets.

Parmi les autres propriétés physiques et mécaniques de la biomasse qui pourraient être considérées, la résistance à la compression doit être citée, pour avoir été étudiée. Cependant, le nombre de facteurs à prendre en compte pour caractériser la relation qui l'unit au broyage rendent cette option très peu réaliste en pratique. Le score 1 a donc été attribué à cette propriété.

Aucune donnée n'est disponible dans la littérature au sujet de l'énergie consommée lors du broyage de pellets. Il est probable qu'elle soit influencée par les mêmes facteurs que le broyage de la biomasse. Cependant, des propriétés propres aux pellets de biocombustibles sont à prendre en considération, la durabilité et la granulométrie des consistants, toutes deux reprises dans le cahier des charges des principaux utilisateurs de pellets en centrale.

Avant utilisation en centrale, les constituants des pellets subissent une ultime réduction granulométrique. Cette réduction est probablement très consommatrice d'énergie au regard de celle qui pourrait être exprimée à partir de la durabilité. En effet, la cohésion de la matière au sein des particules constitutives des pellets est vraisemblablement supérieure à la cohésion entre ces particules. Ces deux propriétés se sont donc vues attribuer un score 3.

Le dernier facteur d'influence de la consommation énergétique d'un broyage, c'est le circuit de broyage lui-même. Trois facteurs principaux sont ici à prendre en compte : le type de broyeur ou de broyage, le débit d'alimentation et enfin la dimension des mailles de criblage utilisée. Ce dernier point a été abordé lors de la discussion de l'influence de la granulométrie. Son influence est manifeste et justifie le score 3. Idéalement, une relation entre la grille utilisée et la granulométrie produite est à identifier.

Le débit d'alimentation d'un broyeur influence probablement le rendement du broyage. Cependant, le fait de rapporter la consommation à la masse de matière broyée, du moins si le broyeur est utilisé à son débit optimum, clarifie l'influence réelle de ce paramètre. Un score 1 a donc été attribué.

Reste l'influence du circuit de broyage dont il n'est pas aisé de s'affranchir, pourtant l'industrie minérale caractérise les matières qu'elle traite sans tenir compte de ce facteur. La caractérisation de la matière est faite à partir d'une méthode de mesure stricte

et clairement définie (index de Bond, par exemple), utilisant un modèle réduit des broyeurs utilisés en industrie. La relation entre les données mesurées au laboratoire et en industrie est faite grâce à l'utilisation de facteurs correctifs. D'après la littérature, le circuit de broyage de la biomasse influence assez peu la consommation énergétique du broyage, s'il est comparé au facteur matière par exemple, un score 1 a donc été attribué à ce paramètre.

2.5. Conclusions

Le gouffre entre les lois énergétiques de broyage des matières minérales et l'état des connaissances concernant la biomasse à ce sujet est flagrant. La mise en pratique de ces lois, certes empiriques, semble quotidienne dans l'industrie minière, alors que les utilisateurs de biomasse peinent à évaluer la granulométrie de l'alimentation et du produit, pour les relier à l'énergie qui aura été consommée par le broyage de quelques matières particulières, principalement agricoles.

Même si sa théorie a été souvent remise en question, il demeure que la loi de Bond est un standard de l'industrie minérale. Vraisemblablement, cela est-il dû au nombre de matières caractérisées par l'auteur qui, aujourd'hui encore, offre une référence aux praticiens des industries minérales ? Son application, à la lettre, au broyage de la biomasse n'est sans doute pas souhaitable, ne fut-ce que parce que le broyeur utilisé pour déterminer le «*Work index*» de Bond est un broyeur à boulets qui n'est pas adapté au broyage de la biomasse. La notoriété de la loi de Bond cache ses évolutions ultérieures, en particulier l'unification avec les lois de Von Rittinger et Kick. Mais au final, il reste un principe, simple, basé sur très peu de paramètres : la matière, sa granulométrie avant et après broyage, une consommation énergétique et une relation qui les unifie.

La simulation, utilisant une approche de type bilan de population, s'intéresse moins à la consommation énergétique liée au broyage qu'à la prévision de la granulométrie qui sera obtenue à l'issue d'un broyage. Ces modèles sont, par ailleurs, généralement conçus pour décrire un procédé existant et pouvoir simuler, valider ou invalider des options de ce procédé. Cette approche est donc prématurée pour caractériser le broyage de biomasse.

La consommation énergétique du broyage des pellets lors de leur utilisation en centrale électrique semble n'avoir encore fait l'objet d'aucune publication. Plus globalement, ce sont les données relatives à la consommation énergétique du broyage de la biomasse qui sont rares. Elles suffisent cependant à montrer que la gamme de consommation engendrée par cette opération est vaste et qu'une formulation globale du procédé est souhaitable, du point de vue technique, mais aussi, économique. Car, actuellement, les données publiées sont, dans leur grande majorité, relatives à des campagnes de mesures réalisées sur des situations et des circuits de broyage particuliers.

Les données existantes sont donc difficiles à exploiter et à globaliser. De plus, le nombre de matières testées reste limité et certains résultats sont parfois contradictoires ou vont à l'encontre des théories générales du broyage. Au-delà de la diversité des matériaux d'origine biologique, ce manque d'homogénéité des résultats doit aussi être recherché dans les multiples méthodes utilisées pour caractériser la matière première, les produits et la consommation au broyage.

L'analyse de la littérature disponible met aussi en lumière la nécessité d'une méthode de référence pour la mesure de la broyabilité de la biomasse. Méthode qui, idéalement, serait indépendante du système de broyage. Or, même dans la diversité des méthodes développées par les industries minérales, rares sont celles qui parviennent totalement à s'affranchir de cette influence. Et lorsqu'elles y

parviennent, les données obtenues s'éloignent de la réalité du terrain. Cependant, ces valeurs sont utilisées comme référence et donnent des indications utilisables pour dimensionner de nouvelles installations de broyage ou comparer entre elles des installations existantes.

L'objectif du développement de cette méthode est donc de fournir aux praticiens de la biomasse des valeurs de référence, comparables à celles disponibles dans les industries minérales. Pour ce faire, l'utilisation d'un appareillage à l'échelle laboratoire semble la plus avantageuse. Elle permettra, plus aisément qu'en production, la caractérisation d'un grand nombre de matière. De plus, un équipement à petite échelle offre suffisamment de souplesse pour réaliser le nombre de mesures nécessaires à l'identification de l'influence de l'état granulométrique sur la consommation. Enfin, le principe de fonctionnement de ce broyeur devra être similaire à ceux utilisés en pratique : un broyeur à marteaux de petite puissance, aux grilles interchangeables à mailles de diamètres différents, équipé d'un dispositif de mesure de la consommation électrique et d'un dispositif de récolte de la matière broyée.

Les facteurs d'influence identifiés et retenus au regard de la consommation énergétique au broyage de la biomasse sont la matière elle-même, la granulométrie de la matière à broyer, la granulométrie du produit, l'humidité avant et après broyage.

L'objet de cette étude est aussi le broyage des pellets et si le broyage de la biomasse, en général, est tant décrit dans ce qui précède, c'est d'une part par manque d'études relatives au broyage des pellets eux-mêmes et d'autre part, parce que les enseignements tirés de l'étude du broyage de la biomasse seront utiles à celle du broyage des pellets. Vraisemblablement, des propriétés supplémentaires, propres à ces biocombustibles particuliers, seront à prendre en compte : la masse volumique unitaire, la durabilité et la granulométrie des constituants des pellets.

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(47 réf.)

Chapitre 3

Définition des méthodes de mesure des propriétés influençant le broyage

3.1. Moisture content determination in solid biofuels by dielectric and NIR reflection methods

One near infrared (NIR) reflectance and five dielectric moisture meters were tested for their capability of measuring moisture content (MC) in solid biofuels. Ninety-eight samples were tested at up to eight moisture levels covering the MC range from fresh fuel to approximately 10% MC (w.b.). The fuel types ranged from typical solid biofuels such as coniferous and deciduous wood chips over short rotation coppice (SRC) to sunflower seed and olive stones. The most promising calibrations were obtained with the NIR reflection method and two dielectric devices where the sample is placed in a container integrated in the device. The calibration equations developed show that there is a profound influence from both laboratory and fuel type. It is suggested that individual calibrations that are based on the specific fuel types used at the individual heating plant could be applied.
Keywords: Moisture content; Solid biofuel; Fuel chips; On-site measurement; Dielectric measurement; NIR.

Détermination de l'humidité des biocombustibles solides par des méthodes diélectrique et réflexion NIR. Une méthode de réflexion proche infrarouge et cinq humidimètres diélectriques ont été testés pour déterminer leur capacité à mesurer l'humidité des biocombustibles solides. Nonante-huit échantillons ont été testés jusqu'à huit niveaux d'humidité couvrant l'éventail des humidités du bois frais jusque 10 % environ (sur base humide). Les combustibles s'étendent des biocombustibles solides caractéristiques comme des plaquettes de bois de conifères et de feuillus, jusqu'aux graines de tournesol et aux noyaux d'olives, en passant par le taillis à très courte rotation (TtCR). Les calibrations les plus prometteuses ont été obtenues avec la méthode de réflexion NIR et deux appareils diélectriques pour lesquels l'échantillon est placé dans un contenant intégré à l'appareil. Les équations de calibration développées montrent qu'à la fois le laboratoire et le combustible ont une profonde influence. Il est suggéré qu'au niveau de chaque installation de chauffage, les calibrations individuelles soient déterminées pour les combustibles spécifiques utilisés.

3.1.1. Introduction

Solid biofuels are by nature heterogeneous with respect to moisture content (MC), bulk density and particle size. The fuels range from well-defined upgraded materials such as wood or straw pellets used in domestic burners to wood chips produced from thinnings and residues from logging operations. Pellets typically have a MC below 12% (w.b.), a narrow bulk density range and a uniform size distribution. In contrast, the MC of forest chips varies substantially, from approximately 10% to around 60% (w.b.), it has a widerange bulk density and a size distribution that largely depends on tree species, raw material and type of chipper used.

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The price of the fuel is often based on the MC. Combustion optimisation, storage management and handling properties are influenced by the MC [1–4]. Traditionally, MC is measured as weight loss by convection drying until constant mass in hot air at 103 or 105 ± 2°C in a drying cabinet. The method is widely accepted and described in a number of standards used for moisture determination of solid biofuels [5–7]. It is considered as being accurate and independent from other parameters such as bulk density or ambient temperature.

However, moisture determination in a drying oven is time and labour consuming. The potential for saving time and obtaining information on the MC prior to incineration is therefore a motivation for using rapid test methods for moisture determination. As reviewed by Nyström and Dahlquist [8], several methods for rapid measurement of MC of solid biofuels exist. Among these, the dielectric- and near infrared (NIR) methods are presently the best alternative to convective drying [9–12]. Common for the devices are that they measure a characteristic of the fuel that which is correlated with the MC.

The dielectric methods are based on electric field changes. The changes are due to the dielectric properties of the material matrix upon radiation with an alternating current. The dielectric properties of any material are expressed as the relative permittivity, $\varepsilon = \varepsilon' - \varepsilon''$, where ε' is the dielectric constant of the material which expresses the materials capability to polarise and ε'' is the loss factor of electrical energy within the field due to polarisation of the material. Water has a high dielectric constant ($\varepsilon' \approx 80$) compared to wood ($\varepsilon' \approx 4-5$) [13]. In its simplest version, a moisture meter consists of two plates connected to an alternating voltage supply. The volume between the plates contains the dielectric material e.g. wood chips. When an alternating current passes from one plate (emitter) to the other (receiver), the decrease in the effective electric field compared to a situation with air or vacuum between the plates is a measure of the moisture in the sample. Besides MC, temperature and bulk density also influence dielectric measurements [14–16]. Influence from bulk density and temperature may be reduced by calibrating the moisture meters to fuel types within a suitable bulk density range and by defining the ambient temperature.

If the applied wavelength is within the radio frequency (RF) area (0.3 GHz), the moisture meter is of the capacitance type. A widespread alternative to radio frequencies are microwaves (0.3 – 20 GHz). Similar to the capacitance meters, the microwave technique measures the MC by sending a wave of a defined frequency through the material. The waves are either absorbed or reflected by the dipole water molecules, which is measured as a transmission or reflection factor. This technique is particularly attractive since influences from ion conductivity are negligible at high frequencies as compared to the dielectric losses.

The NIR reflectometric method is based on the principle that a material illuminated by monochromatic light of a defined wavelength absorbs, transmits or reflects the light. Water has two distinct absorption bands in the NIR part (800 – 2500 nm) of the electromagnetic spectrum at around 1475 and 1940 nm. This is used in NIR-reflectometry where monochromatic light of either of the two wavelengths is absorbed by the water molecules of the sample. The absorption difference between the absorbed bands and reference bands using wavelengths that are almost entirely reflected provide a quantitative measure of the functional O–H groups of the material (primarily water). Thygesen [17] found no literature describing influence of bulk density, whereas Fassio and Cozzolino [18] showed that NIR could be sensitive to variances in the colour of the material.

This paper summarises the results of the testing of five rapid moisture meters mea-

suring dielectric properties in the radio and microwaves frequencies and on one optical device based on NIR reflectance. All devices tested are commercially available and two of them are developed particularly for moisture determination of solid biofuels. The study was carried out as a round robin with participants from 11 European laboratories as described in Section 3.1.2.3.

3.1.2 Materials and methods

3.1.2.1 Rapid moisture meters

An overview on the optical and five dielectric moisture meters tested in the study is given in Table 3.1.1.

The Pandis and Schaller devices belong to a type where the sample is placed in a container during the measurements (container type). The emitter and receiver are orientated in a way that allows an electrical field to build up within the sample (transmission measurements). Both devices are commercially available only as pre-calibrated versions. The Pandis consists of a cylindrical container (640 x 500 mm) with a filling volume of approximately 60 l. It is designed to measure MC ranging from 0% to 55% (w.b.) on a mixture of fine and medium chips as described in the ÖNORM M7133 [19]. The sample is compacted during filling by shaking the container twice when it is half full. The Schaller has a cubic sample container (150 x 190 x 400 mm) which requires a sample size of 1700 ± 10 g. It is designed to measure fine and medium chips [19], at a MC range from 0% to 40% (w.b.) and an optimum measuring range between 10% and 25% (w.b.). With the Schaller, the influence from bulk density is reduced due to the application of a defined sample volume and mass.

ACO, Wile and the microwave type Moist are all handheld devices. The Wile has a probe (length ≈ 190 mm) that is inserted into the sample, while ACO and Moist have a flat sensor head (ACO, Ø = 50 mm; Moist, Ø = 80 mm), that is placed on the surface of the sample during measurements (reflective measurements). The sensor heads of the hand-held devices thus unite emitter and receiver.

The ACO and Wile compensate for bulk density differences by pressing the sensor head with a defined force onto the material during measurements. The ACO and Moist are available only in non-calibrated versions, whereas the Wile is pre-calibrated for MC measurements in silage. Except the Schaller, only non-calibrated versions of the dielectric methods were tested.

The optical Mesa equipment is developed for on-line measurements. It comprises a gauge with a filter wheel that releases monochromatic light of the desired wavelength (measuring beam) onto the sample and measures the reflected light. A reference beam

Table 3.1.1. Description of the tested rapid moisture meters.

Method	Principle	Manufacturer type	Name	Range	MC range (%)[a]
Dielectric	Capacitive	Pandis: FMG 3000	Pandis	<0.3 GHz	0–55
		ACO: MMS-0-1-2-0	ACO		0–80
		Farmcomp: Wile 25	Wile		13–85
		Schaller: FS 2002-H	Schaller		0–40
	Microwave	hf sensor: Moist 100	Moist	0.3–20 GHz	0–80
Optical	NIR reflection	Mesa: MM710	Mesa	800–2500 nm	10–40

[a] As recommended by the supplier.

with a wavelength, which is not absorbed by free water, is used to eliminate variations caused by the environment (e.g. distance to sample or colour differences between the particles). Furthermore, a correction beam, which follows the normal radiation paths of the sensor head without leaving the system, enables corrections for any measuring drift (e.g. caused by ageing of the light source, changes in sensitivity of the detector, temperature changes). In this study, the gauge was equipped with a filter for measuring in the range from 10% to 40% MC. For the testings, the sample was placed in a cylindrical steel container that was rotating horizontally during the measurements, thus imitating a moving stream of fuel on a conveyor belt.

For reference MC determination triplicates of 500 g from each sub-sample were measured in drying ovens at 105 °C [20]. The ovens of all partners were tested for temperature accuracy and distribution prior to the round robin. This was done using thermocouples connected to a Testo 177 data logger. Areas in the drying ovens where the temperature exceeded the tolerance of 105 ± 2 °C were avoided for the moisture determinations.

3.1.2.2. Fuel conditioning and sample preparation

A broad variety of 98 solid biofuel types were used for this study (Table 3.1.2). From each fuel type, samples of minimum 1 m^3 were collected and divided into sub-samples of equal size (eight for wood chips, up to five for the others) using the coning and quartering technique. Approximately equidistant moisture steps were produced for each sample ranging from fresh fuel to approximately 10% moisture (w.b.). The target moisture steps were achieved by determining the MC of the fresh sample [20] followed by floor drying of the sub-samples until the pre-defined MC step was achieved. During the drying process, the moisture level of the sub-samples was monitored on small samples (500–1000 g), which were dried parallel to the sub-samples. When the small sample reached a pre-defined weight corresponding to a desired MC level, the drying of the sub-sample was terminated. The floor drying was performed by spreading the sub-samples on a layer of wooden plates covered by plastic or fine meshed net. Using fine net allowed forced ventilation, which accelerated the drying process. The sub-sample had a height of approximately 5–7 cm and was turned and mixed frequently to achieve a uniform drying process. The conditioning procedure secured that all fuel samples were represented by subsamples covering the widest possible moisture range. Furthermore, it secured that one of the sub-samples had an MC of 30 ± 3%; these samples were for determine bulk density. After conditioning, the sub-samples were stored in airtight plas-

Table 3.1.2. Applied fuels for the test of rapid moisture meters.

Fuel type	Bulk density range (kg/m^3)	No. of samples
Wood chips		
Coniferous	111.7–228.6	30
Deciduous	154.2–248.2	25
Mixed	177.8–213.3	2
Short rotation coppice	129.9–156.5	6
Bark	121.57–219.2	6
Sawdust	91.5–162.4	9
Shavings	95.2	1
Pellets		
Wood	600.5–668.0	6
Straw	511.5–636.0	3
Stones/shells	212.34–522.5	5
Olive cake	622.4	1
Grain		
Triticale	647.4	1
Energy grass		
Miscanthus	82.0–138.4	2
Peat	142.0	1

tic bags at a cool, dry and dark location for a minimum of 2 weeks, in order to ensure a uniform moisture distribution within the sub-samples.

3.1.2.3. Measuring procedure

The 11 participating European laboratories were divided into two groups. Each laboratory prepared up to 10 test fuels as described above (3.1.2.2). Two sets of the rapid-MC equipment were procured and each circulated within a group of laboratories. Pre-tests were performed on the dielectric equipment to identify optimal settings during the round robin. Prior to the round robin tests, detailed guidelines describing the measuring procedure were developed to ensure uniform trial conditions. Further, the handling of all devices tested was demonstrated at a workshop which was attended by representatives of all participating laboratories.

The MC of the sub-samples of each fuel was measured with the six rapid test devices starting with the driest moisture level and proceeding with increasing moisture levels until all levels were measured. Prior to the measurements, the sub-sample was poured on a clean surface (e.g. a plastic sheet), homogenised twice and split into four portions by coning and quartering. Three of the newly obtained portions were put together in a transparent plastic bag. The fourth portion was split into three small plastic bags, which were closed airtight to avoid moisture changes.

The measurements started with the Moist device. In 20 replicate measurements, the measuring head was held at the exterior of the bag at the middle of the filling height. Then 10 replicate measurements with the Wile were performed, plunging the probe into the material from the top of the open bag. Subsequently, three replicate measurements with the Pandis device were made and then the three replicates for reference moisture were sampled and measured according to CEN/TS 14774-1 [20]. Proceeding with the content of three small plastic bags, the measurements were then performed with the Schaller (3 times one measurement), the ACO (3 times five replicates on the surface of a 7 cm sample layer) and the Mesa (3 times five replicates).

The bulk density was determined in triplicates on the sub-sample which had a MC of 30 ± 3% using a 50-l cylindrical steel container. Forced compaction was applied by dropping the container 3 times from 150 mm height [21]. For fuel types with a maximum MC below 30%, primarily straw and wood pellets, the bulk density was measured at the MC at reception.

3.1.2.4. Statistics

All analyses were performed using SAS version 8.2. In order to exclude erroneous data due to sample pretreatment, all data from samples that varied more than 1% (t-test) within the three reference sample measurements were excluded from the analysis. Tests for outliers from measurements with the devices tested were performed using the interactive data analysis program in SASs, which compares the studentised residuals to a critical value. Only those outliers that could be explained as handling or procedure errors were excluded from the analysis.

The following model was used to analyse the data:

$$MC_{ijk} = \alpha_0 + \beta_1 \text{ value} + \beta_2 BD + A_i + B_j + (AB)_{ij} + \varepsilon_{jk} \quad (1)$$

where α_0, β_1, β_2 are the parameters for fixed effects, A_i, B_j, AB_{ij} the parameters for random effects, i the Lab, j the Fuel type, k the replication, ε_{ijk} the ~ $N(0,\sigma^2)$, DB the bulk density, Value the reading on device and MC the moisture contents.

Significance levels of fixed and random effects were tested by t-test and χ^2, respectively.

A joint data set from fuel chips of coniferous, deciduous and short rotation coppice was applied to the SAS® PROC MIXED procedure to test the variance due to fuel type and laboratory. The contributions of the dimensionless reading from the test device and the bulk density of the fuels were treated as fixed effects whereas the variance due to laboratory and fuel type were considered to be random effects (bulk density is only considered for the dielectric devices). With PROC MIXED, the variance components due to the random factors "lab" (laboratory) and "fuel type" was quantified. The variance that is explained by laboratory and fuel type respectively in Tables 3.1.4–3.1.6 indicates the magnitude of the bias for the predicted MC that is expected if the devices are not calibrated specifically to the laboratory (or person) and/or fuel type. This is important, as a bias only can be eliminated by calibration. The remaining part of the variance (residual) describes the ability of the specific device to measure MC under the given circumstances. Since the fuels were not circulated among the laboratories each fuel was unique to the laboratory. Consequently, the variance due to fuel type may include both that of general fuel type and of the laboratory specific fuel. Finally calibrations on selected devices were made by general linear regression methods (SAS® PROC GLM), fitting the data to Eq. (1), but excluding the random effects, fuel-type and laboratory, from the model since the calibration was made specifically for each laboratory and fuel type.

The minimum number of replications required to measure MC within a range of ±5%-point at a certain significance level were determined according to Cochran [22]:

$$CL \geq t_{1-\alpha/2} S/\sqrt{nn} \geq t^2_{1-\alpha/2} S^2/CL^2, \qquad (2)$$

where *CL* is the confidence limit (5%-point), *a* the significance level, *s* the standard deviation, *n* the number of replicates and *t* the *t*-value with *n*-1 degrees of freedom.

3.1.3. Results and discussion

Significance levels for fixed and random effect tested for the chipped fuels are shown in Table 3.1.3. In all cases, the interaction between laboratory and fuel type were significant and thus the effect 'fuel type' was always included in the model despite its main effect in some cases was insignificant.

An initial analysis including all fuel samples showed that the variance due to fuel type constitutes a considerably large share of the variance (see "var fuel type" in Table 3.1.4). In order to reduce the influence due to fuel type heterogeneity, further analyses were performed on a reduced data set of wood chips from coniferous, deciduous and short rotation coppice. The result from the analysis on the reduced data set is given in Table 3.1.5.

The residual variance shown in Table 3.1.5 indicates that the NIR reflectance method is able to measure moisture with a higher accuracy than the dielectric methods. No statistically significant influence from the variance due to laboratory and fuel type was found in the reduced data set. For the dielectric devices, the residuals are considerably lower for the container type equipment compared to the probe/sensor type equipment. Pandis was more sensitive to inconsistent handling than Schaller (see "var lab" in Table 3.1.5). A reason might be that in contrast to Schaller, Pandis prescribes compaction of the sample introducing an additional source of error. Extending the MC range to 0–55% for the Schaller results in higher residual variance (33.45; see Table 3.1.6) compared to Pandis (20.73; see Table 3.1.5). This indicates that the Schaller is less suitable for measuring within the MC range of 40–55% MC, which is common for wood fuels used in heating plants [11,23].

The analyses of ACO, Wile and Moist were all performed on wider moisture ranges than the container types (Table 3.1.5). Thus,

Table 3.1.3. Significance levels of fixed and random effects used in Eq. (1).

Device	MC range (%)[a]	Fixed effects		Random effects		
		Value	Bulk density	Lab	Fuel type	Interaction
Pandis	0–55	<0.0001	0.0005	0.12700	0.01165	<0.0001
Moist	0–80	<0.0001	<0.0001	0.03076	0.38786	<0.0001
Wile	13–85	<0.0001	<0.0001	0.02084	0.06814	<0.0001
Schaller	0–40	<0.0001	0.0004	1.0	0.00113	<0.0001
ACO	0–80	<0.0001	<0.0001	0.00508	0.10980	<0.0001
MESA	10–40	<0.0001	a	0.97227	0.75643	<0.0001

[a] Bulk density not relevant.

Table 3.1.4. Sum of the joint variance components of laboratory and fuel type and the interaction between laboratory and fuel type.

Method	Type	Device	Var lab[a]	Var fuel type[b]	Residual[c]	*n*
Optical	Moving	Mesa MM710	2.94	15.58	9.55	6225
Dielectric	Container	Pandis FMG3000	8.60	34.25	33.64	1613
		Schaller FS 2002-H	5.27	15.32	13.32	1171
	Probe/sensor	ACO MMS 0112	9.13	88.19	83.13	9915
		Wile25	22.43	54.14	84.68	5437
		Moist 100	11.48	11.91	65.37	13200

Data originating from the six moisture meters at the full set of fuels and the MC ranges given in Table 3.1.1.
[a] Sum of variance from lab and the interaction of lab and fuel type.
[b] Sum of variance from fuel type and the interaction of lab and fuel type.
[c] Residual variance.

Table 3.1.5. Sum of the joint variance components of laboratory and fuel type and the interaction between laboratory and fuel type.

Method	Type	Device	Var lab[a]	Var fuel type[b]	Residual[c]	*n*
Optical	Moving	Mesa MM710	2.88	3.50	7.84	4470
Dielectric	Container	Pandis FMG3000	13.09	11.38	20.73	1202
		Schaller FS 2002-H	3.94	10.25	12.04	895
	Probe/sensor	ACO MMS 0112	15.99	8.34	32.45	6780
		Wile25	31.72	9.87	36.70	3868
		Moist 100	10.22	4.96	65.99	9020

Data originating from the six moisture meters and the reduced set of fuel types (coniferous, deciduous and short rotation coppice chips) and the MC ranges given in Table 3.1.1.
[a] Sum of variance from lab and the interaction of lab and fuel type.
[b] Sum of variance from fuel type and the interaction of lab and fuel type.
[c] Residual variance.

some variance obtained may be caused by the presence of large proportions of free water. In order to test whether smaller MC ranges of 0–40% and 0–55% reduced the residual variation, the three devices were also analysed at these MC ranges. However, only a reduction of the upper MC range to 40% leads to a detectable decrease of the residual variance compared to analyses on a wider MC range, whereas the 0–55% range gave similar variances as compared to the entire moisture ranges (Tables 3.1.5 and 3.1.6). The variance related to fuel type does not vary much between Pandis, Schaller, ACO and Wile, whereas the influence from the laboratories varies among the devices (Table 3.1.5). In pre-tests, it was found that the inclination of the ACO towards the horizontal plane of the sample influenced the measurement. Furthermore, the results obtained with the ACO and Wile were influenced by the variable forces that were probably applied when the samples were compressed. The high residual variance obtained using Moist (Table 3.1.5) reflects its limited ability to measuring MC's of particulate material such as biofuels. The device was originally developed for measurements on materials such as bricks or wooden panels, which offer a plane solid surface for the measurements. This conclusion is supported by the comparably low variance related to laboratory and fuel type, which means that the major part of the total variance is due to the device itself. The relatively low residual variance of the container type devices compared to the probe/sensor type indicates that the first type is superior to the second.

The results from the calibrations of Mesa, Pandis, Schaller and ACO on individual fuel types and laboratories using PROC GLM in SASs are shown in Table 3.1.7. To facilitate the comparisons between the two types of dielectric meters, the ACO is calibrated for two moisture ranges (0–40% and 0–55% MC, respectively). Based on the R^2-value, the regressions confirm the smaller variance obtained with the NIR reflectance method (R^2 = 0.84–0.99) and the dielectric method using the Pandis (R^2 = 0.89–0.995). Furthermore, it is seen that the dielectric devices are sensitive towards bulk density variations. This can be read from the number of regressions, marked with an asterix in Table 3.1.7, where a significant bulk density ($\alpha < 0.05$) influence had been determined. Also for the Schaller device, acceptable results were obtained, although two of the regressions resulted in poor R^2-values of only 0.75 and 0.31.

The sensitivity of ACO towards variable handling procedures is indicated by the high variation of R^2-values and standard errors between the 11 laboratories. However, some of the laboratories reach similar regression values as for the container type devices, e.g. labs 4 and 8. Any technical solution, which reduces inconsistent handling, should therefore improve the accuracy of the moisture estimation. As shown by Hartman and Böhm [10], arranging the ACO sensor head steadily and horizontally to the sample surface, e.g. in a test rig that simulates a moving sample stream could considerably reduce the variance in the measurements.

Calibrating the Mesa MM710 to the combined set of three fuel types increased the variation in the measurements compared to a calibration for only one fuel type (Fig. 3.1.1, upper plots). For the dielectric devices, it was observed that the variance of the measurements varied for different fuel types and increased with higher MC. This trend was particularly evident above the fibre saturation point, where free water in the cell vacuoles leads to inconsistent measurement (shown for Pandis and ACO in Fig. 3.1.1, middle and lower plots) [13].

The analyses of the Pandis data from Fig. 3.1.1 exemplify that acceptable calibrations with high R^2-values are possible with this principle. To measure the MC shown in Fig. 3.1.1

Table 3.1.6. Sum of the joint variance components of laboratory and fuel type and the interaction between laboratory and fuel type.

Device	MC range (%)	Var lab[a]	Var fuel type[b]	Residual[c]	*n*
Schaller FS 2002-H	0–55	10.40	26.28	33.45	1149
ACO	0–40	8.67	1.62	15.69	4980
MMS 0112	0–55	14.48	7.69	29.98	6465
Wile 25	0–40	10.35	4.97	21.00	3318
	0–55	24.26	10.98	41.84	4298
Moist 100	0–40	6.40	1.68	36.33	6640
	0–55	7.66	3.13	59.75	8600

Data originating from ACO, Wile and Moist, a reduced set of fuel types (coniferous, deciduous and short rotation coppice chips) and the moisture range of 0–40% and 0–55%. Further, the Schaller was tested at a moisture range of 0–55%.
[a] Sum of variance from lab and the interaction of lab and fuel type.
[b] Sum of variance from fuel type and the interaction of lab and fuel type.
[c] Residual variance.

with 75%-points accuracy at a probability level where 99% (α = 0.01) of the measurements is within the 5%-points require at least five replicates for both one fuel type and for the three fuel types. Reducing the probability level to 95% (α = 0.05) decreases the minimum number of measurements to 3 for one fuel type and 5 for the three fuel types (Eq. (2)). The bulk density influences the determination with dielectric devices e.g. for the Pandis a bulk density of 100, 150 and 200 kg/m^3, respectively, the resulting MC will be biased in the order of 2–3%-point between the consecutive bulk density levels.

When comparing the R^2-values and standard errors of the calibrations from Fig. 3.1.1 with similar statistics from other laboratories (Table 3.1.7) confirmed that within a specific fuel type the performance of the laboratory is quite decisive for the overall quality of the calibration. This is true for all tested combinations of moisture ranges and fuel types for both the NIR reflectance and the dielectric methods. Thus, the decision on the number of fuel types and the applied moisture range for the calibration will always be made as a trade-off between the intended accuracy of the subsequent measurements and the workload of providing and applying individual calibrations for each fuel type. If such a calibration regards bulk density as an additional input parameter for the MC estimation, it should be measured along with the MC. Furthermore, it should be determined for all unknown fuels to ensure that the tested samples are within the range of bulk density for which the device was calibrated.

3.1.4. Conclusions

Rapid moisture meters enable instantaneous moisture determination of solid biofuels. Among the instruments tested (one NIR reflectance and five dielectric devices), several promising applications were identified. The main conclusions are as follows:

- The NIR reflectance method (Mesa MM710) and the dielectric devices of the container type (Pandis FMG3000 and Schaller FS2002-H) have clear advantages compared to the other systems tested. They can be operated at higher accuracy with less sensitivity towards variable fuel types or calibration procedures.
- For the dielectric devices, it was shown that the fuel bulk density influences the measurements. This variable should therefore

Table 3.1.7. Statistical figures from regression analyses of NIR reflectance method (Mesa MM710), the dielectric container type (Pandis FMG3000 and Schaller FS 2002-H) and the probe/sensor type with the lowest variance given in Table 5 (ACO).

Device	MC (%)	Chips type	Statistics	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6	Lab 7	Lab 8	Lab 9	Lab 10	Lab 11
Mesa MM710	10-40	Coniferous	R^2	0.88	0.91	0.82	0.99	0.97	0.92	0.93	0.89	0.97	0.99	0.89
			n	285	165	345	75	360	30	285	240	90	90	150
			s^2	11.74	3.73	16.86	1.65	2.22	1.80	4.65	5.43	1.95	1.44	8.20
		Deciduous	R^2	0.97	0.84	0.86	0.99	0.91		0.91	0.94		0.96	0.96
			n	135	345	360	75	240		465	285		75	90
			s^2	3.69	10.51	11.59	1.05	5.38		5.48	4.83		2.78	1.89
		SRC	R^2		0.93			0.98			0.91		0.99	0.98
			n		90			75			165		60	45
			s^2		4.80			1.67			3.87		0.85	1.16
Pandis FMG3000	0-55	Coniferous	R^2	0.89	0.88	0.93	0.96	0.96	0.98	0.91[a]	0.93[a]	0.98	0.98	
			n	108	63	117	15	84	12	92	66	20	24	
			s^2	23.92	22.90	14.92	4.29	3.98	1.59	16.14	12.44	2.51	5.01	
		Deciduous	R^2	0.98	0.89	0.93	0.99	0.98[a]		0.96[a]	0.96		0.96	
			n	45	95	95	18	66		111	72		24	
			s^2	3.79	18.91	10.82	1.27	2.33		4.20	5.54		5.90	
		SRC	R^2		0.97			0.995			0.93		0.97	
			n		24			21			48		21	
			s^2		4.51			0.89			9.26		9.12	
Schaller FS2002-H	0-40	Coniferous	R^2	0.94[a]	0.86	0.90		0.92	0.89	0.94[a]	0.97	0.96	0.94	0.90[a]
			n	69	33	84		72	6	63	21	18	18	27
			s^2	8.47	5.78	12.11		5.44	3.62	5.85	3.17	2.89	6.32	9.46
		Deciduous	R^2	0.97[a]	0.75	0.89[a]	0.97	0.95[a]		0.96	0.93		0.94	0.98
			n	39	78	72	11	45		99	63		21	21
			s^2	5.38	25.14	9.14	2.01	5.74		2.82	7.53		6.14	1.69

		SRC	R^2		0.93			0.97			0.31		0.97	0.98
			n		15			15			12		14	6
			s^2		3.80			2.46			6.11		3.80	1.76
ACO	0-40	Coniferous	R^2	0.88	0.77	0.81[a]	0.96	0.85	0.56	0.90[a]	0.90[a]	0.90	0.78	0.75[a]
			n	345	165	420	75	270	30	315	270	90	90	165
			s^2	15.40	9.20	22.08	4.75	10.52	10.53	8.54	7.39	7.35	21.54	24.18
		Deciduous	R^2	0.87[a]	0.77[a]	0.84	0.95	0.90[a]		0.87[a]	0.92[a]		0.94	0.85
			n	195	420	375	75	180		495	315		105	105
			s^2	16.63	24.01	15.31	3.88	6.42		8.96	7.63		5.77	10.68
		SRC	R^2		0.88			0.90			0.90[a]		0.93	0.97
			n		90			75			180		75	60
			s^2		8.65			6.72			7.67		8.92	4.45
ACO	0-55	Coniferous	R^2	0.83[a]	0.88[a]	0.83[a]	0.96	0.84[a]	0.86	0.90[a]	0.93[a]	0.88	0.67	0.77[a]
			n	540	315	585	75	330	60	465	330	105	120	225
			s^2	38.47	23.41	38.40	4.75	19.33	18.10	17.59	10.82	13.41	72.11	40.23
		Deciduous	R^2	0.89[a]	0.84[a]	0.85[a]	0.95	0.92[a]		0.90[a]	0.94[a]		0.94	0.87
			n	225	480	480	90	225		555	360		120	120
			s^2	22.27	26.42	22.51	6.29	8.86		10.02	8.70		7.35	12.04
		SRC	R^2		0.84			0.94			0.91[a]		0.95	0.97
			n		120			105			240		105	90
			s^2		26.62			10.37			11.88		13.32	7.50

Data from the 11 participating laboratories and three fuel chips types; coniferous, deciduous and short rotation coppice (SRC).
[a] Bulk density significant in the calibration.

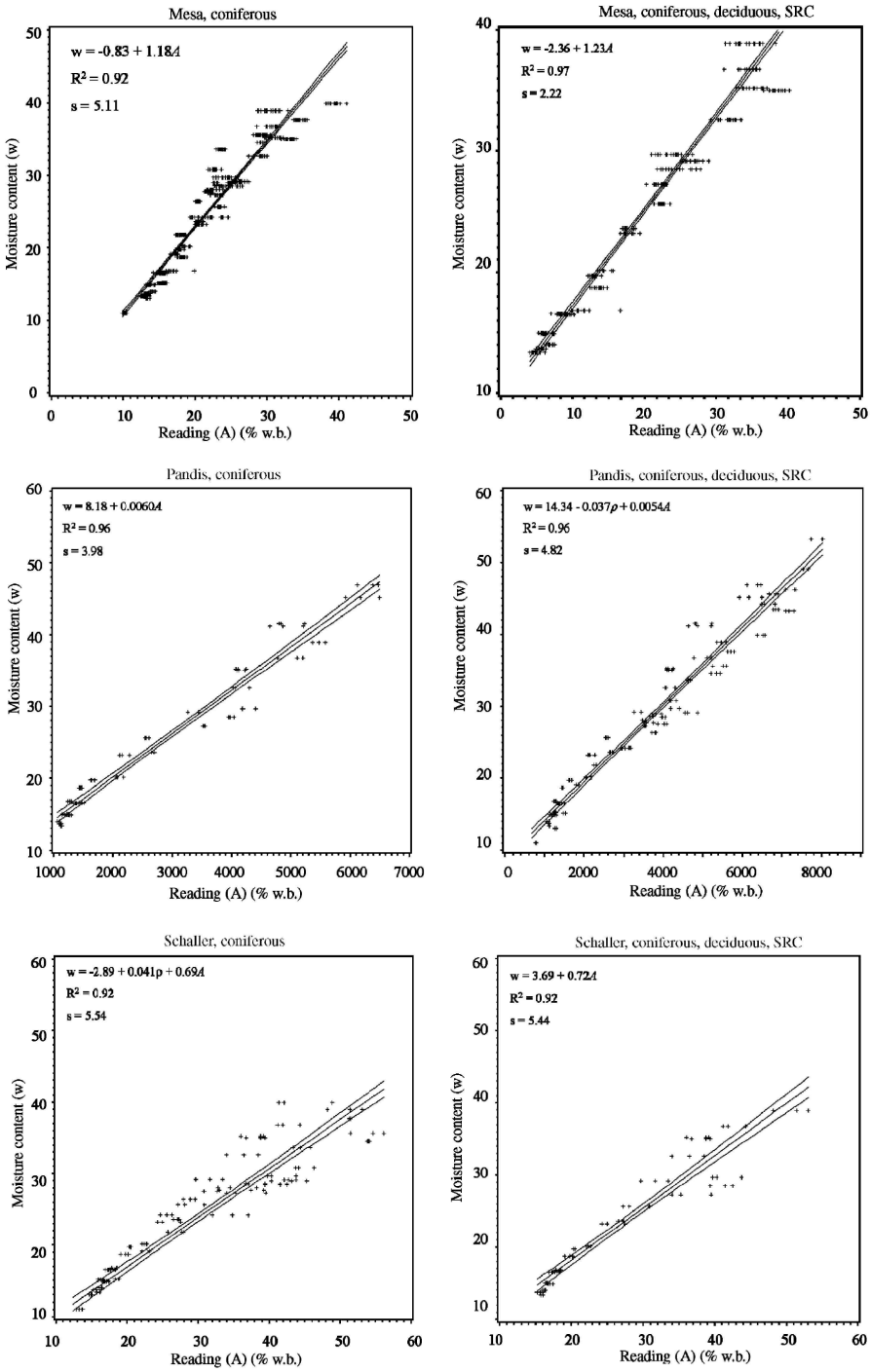

Fig. 3.1.1. Example of calibration functions for Mesa MM710, Pandis FMG3000 and Schaller FS 2002-H. Data shown are from Lab 5 in Table 7. The upper and lower lines in each plot represent the confidence intervals (α = 0.05). The black dots represent the original data pairs.

always be considered in calibrations for dielectric devices when several fuel types are involved.
- Due to the usually higher precision it is advisable to apply an individual calibration for the prevailing fuel base at a site, rather than using a global calibration function for all wood fuels of different properties and origin.
- The application of calibrations from other laboratories implies the risk of non-suitable data fit, as shown by the significant laboratory influences.

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- Forest Contracting Association, UK (B. Hudson),
- EC Baltic renewable energy centre, Poland (M. Pisarek and L. Jamowics),
- AFOCEL, France (M. Lecourt),
- INETI, Portugal (D.H. Boavida),
- Comitado Termotecnico Italiano, Italy (J.A. Calzoni).

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3.2. Methods for size classification of wood chips

Methods for size classification of wood chips were analysed in an international round robin using 13 conventional wood chip samples and two specially prepared standard samples, one from wood chips and one from hog fuel. The true size distribution of these two samples (according to length, width and height) had been determined stereometrically (reference method) using a digital calliper gauge and by weighing each of the about 7000 wood particles per sample. Five different horizontal and three rotary screening devices were tested using five different screen hole diameters (3.15, 8, 16, 45, 63 mm, round holes). These systems are compared to a commercially available continuously measuring image analysis equipment.

The results show that among the devices of a measuring principle—horizontal and rotary screening—the results are quite comparable, while there is a severe incompatibility when distributions are determined by different measuring principles. Highest conformity with the reference values is given for measurements with an image analysis system, whereas for all machines with horizontal screens the median value of the size distribution only reached between one-third to half of the reference median value for the particle length distribution. These deviations can be attributed to a higher particle misplacement, which is particularly found in the larger fractions. Such differences decrease when the particle's shape is more roundish (i.e. sphericity closer to one). The median values of length distributions from screenings with a rotary classifier are between the measurements from an image analysis and horizontal screening devices.
Keywords: Wood chips; Hog fuel; Size classification; Particle size distribution; Horizontal screening; Rotary screening; Image analysis; Sphericity.

Méthodes pour la classification granulométrique des plaquettes de bois. Des méthodes de classification granulométrique des plaquettes de bois ont été analysées lors d'un test interlaboratoire international qui a utilisé 13 échantillons de plaquettes de bois conventionnelles et deux échantillons standardisés spécialement préparés, un de plaquettes de bois, l'autre de déchets de bois. Les distributions granulométriques réelles de ces deux échantillons (en longueur, largeur et hauteur) ont été déterminées de manière stéréométrique (méthode de référence) en utilisant un pied à coulisse digital et en pesant chacune des environ 7000 particules par échantillon. Cinq tamis horizontaux différents et trois tamis rotatifs ont été testés en utilisant des tamis aux mailles de diamètres différents (3, 15, 8, 16, 45, 63 mm, mailles rondes). Ces systèmes sont comparés à un équipement de mesure par analyse d'image en continu, disponible commercialement.

Les résultats montrent que parmi les appareils ayant un même principe de mesure – tamisage horizontal et rotatif – les résultats sont assez comparables, alors qu'il y a une incompatibilité sévère lorsque les distributions granulométriques sont déterminées à l'aide de principes de mesure différents. La meilleure conformité avec les valeurs de référence est obtenue pour le système d'analyse d'image, alors que pour toutes les machines à tamis horizontal, la valeur médiane des distributions granulométriques n'atteint pas plus du tiers à la moitié de la valeur médiane de référence obtenue pour une distribution en longueur des

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particules. Ces déviations peuvent être attribuées à davantage de particules allouées à des classes erronées, ce qui se produit particulièrement dans les fractions les plus importantes. Ces différences diminuent lorsque la forme de la particule est plus ronde (c'est-à-dire une sphéricité proche de 1). Les valeurs médianes des distributions en longueur issues de tamisages réalisés avec un tamis rotatif sont intermédiaires entre les mesures issues de l'analyse d'image et les appareils de tamisage horizontaux.

3.2.1. Introduction

Particle size distribution influences the handling and combustion characteristics of solid particulate biofuels. Storage and ventilation properties are affected [1]. Bridging tendency, which is critical for an undisturbed fuel conveying, increases with long and thin particles [2,3]. Energy conversion and emissions are also influenced by the particle sizes, particularly for sawdust [4].

For fuel chips, mechanically or manually operated screening devices are commonly applied and there is a large variety of applicable systems. The screens are usually arranged in a tower of several sieves, which are clamped to each other, starting with the largest aperture size on top (Fig. 3.2.1). They are operating in a one-, two- or three-dimensional screen shaking movement sorting the particles by decreasing size. This type of determination of particle size distribution of wood chips is described in a number of standards [5-8].

Recently some measuring alternatives to the common flat screens have been discussed for use in wood fuel quality determination [9]. One of the options is an arrangement of several cylindrical sieves, which form a rotary screen starting with the lower aperture size and sorting the particles by increasing size (Fig. 3.2.1). The horizontal screening and the rotary classifier belong to the gravimetric method, which all have the problem that long and thin particles can pass small aperture widths while being in a vertical orientation, and they are thus wrongly allocated, if a separation by length is desired.

Such disadvantages are overcome by the application of a dynamic online image analysis system (Fig. 3.2.1). This technique is particularly interesting as it can sort the particle sizes according to more than just one size parameter. However, the image analysis

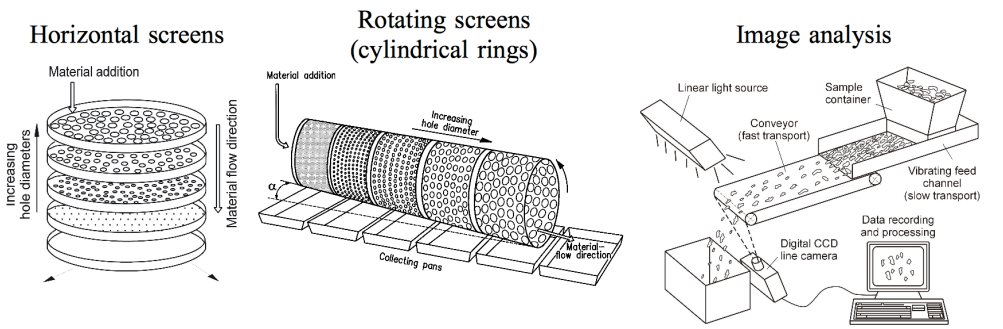

Fig. 3.2.1. Functioning principles of screening and image analysis classification methods for wood chips classification.

is sensitive towards particle overlappings, resulting in overestimation of particle sizes [9,10]. A proper separation of the particles is thus an important prerequisite for an accurate size determination.

A reliable way of size determination is by hand, using callipers. However, such a stereometric measurement is generally too time consuming for routine operations and it is therefore only applicable for measurements on reference materials.

The measuring principles described are also based on different physical assumptions. The gravimetric methods—and also the stereometric method—are indirect methods where all particles are initially separated to size fractions before they are converted to mass fractions by weighing. The measurement therefore implies the hypothesis that the density of smaller and larger particles is more or less constant in order to allow conclusions on the size fractions by weighing. Density differences or inconsistent moisture contents would disturb the accuracy of the measurement. For the optical method, however, density differences are irrelevant, as the size measurement is made directly by regarding the projection of each particle shape.

In order to achieve comparable measurements, it is important to know to what degree the determination methods are compatible and which are the influencing factors. Such knowledge is particularly required for the currently ongoing elaboration of European biofuel standards. In this research, the goal was to determine any systematic differences between the methods and to evaluate the influencing factors.

3.2.2. Materials and methods

3.2.2.1. Test material and sample preparation

Two standard fuel samples were produced from wood chips and hog fuel as distinguished in the new Technical Specification for solid biofuels [11]. The chip sample (SF1) consisted of 6036 particles (volume 9.11) and the hog fuel sample (SF2) consisted of 7534 particles (volume 7.21) (Fig. 3.2.2). For each particle, all three dimensions (length, width and thickness) were measured using a digital calliper (Mahr 16ES) with an accuracy of 0.01 mm (Fig. 3.2.3). In order to reduce human errors in these measurements, the readings were electronically transmitted to an Excel data sheet. Additionally, the weight of each particle was determined with an accuracy of 0.001 g at constant temperature and humidity. The length was measured as the maximum expansion of the particles. The width was recorded as the second longest expansion perpendicular to the length, while the thickness was the third longest expansion perpendicular to both, length and width.

The particles were hand sorted by length into five size classes. They were then coloured by different stain colour (in water bath) and dried. Each colour represents one of five size classes following the same size sequence as applied for the screening devices: 3.15-8 mm (1, orange), 8-16 mm (2, green), 16-45 mm (3, yellow), 45-63 mm (4, red) and over 63 mm (5, black). The colouring was intended to enable an easy selecting and weighing of all wrongly allocated particles from any sieving tray.

In order to enable conclusions on the influence of the particle shape, a second standard sample (SF2) was designed following the same length distribution as the first standard sample (SF1) but using hog fuel. The conformity of length properties was achieved by a constant monitoring of all changes in the size distribution curve which were caused by each particle added. Particles for SF2 were thus selected to achieve a best possible fit to the cumulative length distribution curves of SF1 (Fig. 3.2.4). The hog fuel sample (SF2) had more longish and thin shaped particles (mean length-width

Fraction (length in mm)	> 3,15 – 8	> 8 – 16	> 16 – 45	> 45 – 63	> 63	*Total*
Total mass (g)	6,0	105,8	1 318,1	194,4	79,3	*1 703,6*
Share of mass (%)	0,4	6,2	77,4	11,4	4,7	*100,0*
Number of particles	468	1 965	3 502	85	16	*6 036*

Fraction (length in mm)	> 3,15 – 8	> 8 – 16	> 16 – 45	> 45 – 63	> 63	*Total*
Total mass (g)	7,1	79,6	1 004,9	133,4	47,2	*1 272,2*
Share of mass (%)	0,6	6,3	79,0	10,5	3,7	*100,0*
Number of particles	583	1 541	5 187	179	44	*7 534*

Fig. 3.2.2. Example of particles in the manually prepared and coloured standard wood chip sample (SF1, left) and hog fuel sample (SF2, right).

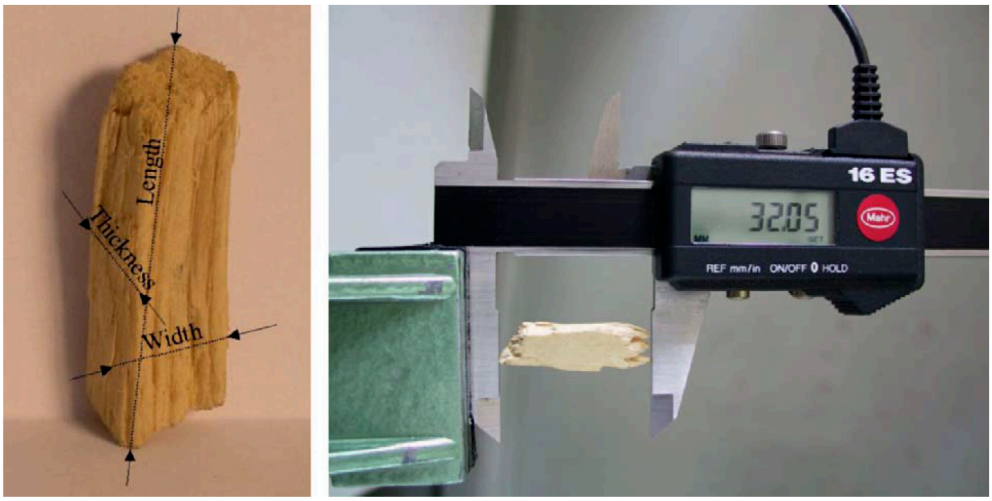

Fig. 3.2.3. Recording size data of a standard wood chip sample by manual size determination with a digital calliper gauge. Left: three measured dimensions of a particle; right: length determination with a digital calliper gauge.

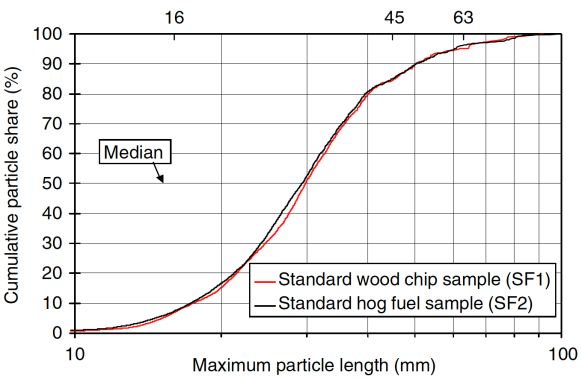

Fig. 3.2.4. Cumulative particle length distribution of the two standard samples (SF1 and SF2) according to stereometric length determinations.

ratio: 3.6) while the wood chip sample (SF1) consisted of more square-like particle shapes with a mean length-width ratio of 1.9.

Additionally to the two standard samples, 13 wood fuel samples of approximately 81 volume were produced using four different chipper types. Eight samples were from debarked round wood of spruce (one disc, one drum, one spiral chipper, one shredder) and beech (one disc, one drum, one spiral chipper, one shredder). Furthermore, one sample of chipped short rotation coppice (poplar, disc chipper) and three samples from logging residues (two spruce, one beech, all by disc chipper).

3.2.2.2. Size determination equipment

Gravimetric methods

Five different horizontal sieves and one type of rotating sieve were used in the round robin (Table 3.2.1). The tested horizontal sieves applied different shaking movements either in a one-dimensional to and fro movement (device 1, 2, 3) or in circular, two-dimensional operation (device 4) or in a three-dimensional shaking mode (device 5), which means that additionally to the circular movement a horizontal motion is introduced, too.

The rotary screen classifiers consists of five joined cylindrical screening cylinders, each of 400 mm height having a 360 mm effective screening length (Fig. 3.2.1). The cylinders form a rotating drum of 500 mm diameter and 2230 mm total length. In opposition to horizontal screenings, fine particles are separated at the beginning of the screening process and are collected in the pan underneath. All particles larger than the widest screen holes of the last ring are discharged into a final collecting pan. The inclination of the drum towards the horizontal ground (α) was 3.0° and the drum rotated at a speed of 20 min^{-1}. Due to the inclination of the drum, the remaining particles travel onward to the subsequent screening rings. The sample material is continuously fed over a slide tangen-tially onto the bottom of the first screening ring.

Table 3.2.1. Properties and screening parameters of devices tested in round robin trials.

Tested at/ device	Type	Shaking movement	Screen dimensions (mm)	Screen area (cm^2)	Amplitude (mm)	Frequency (min^{-1})
Horizontal screening						
Lab 1 (1)	Build according to SCAN-CM 49:94 [7]	One-dimensional	Rectangular 400 × 650	2600	120	160±10
Lab 2 (2)	Prototype by HFA	One-dimensional	Rectangular 500 × 500	2500	60	300
Lab 3 (3)	Stiletto Vibrating table	One-dimensional	Rectangular 500 × 500	2500	0.09-1.3	3000
Lab 4 (4)	Retsch AS 400 control	Two-dimensional	Round Ø 400	1257	30	50-300
Lab 4 (5)	Haver & Boecker EML 450	Three-dimensional	Round Ø 400	1590	0.1-2.0	3000
Rotary screening						
Lab 1, 3, 4	Rotating screen	Rotating	Cylindrical Ø 500 Length 400[a]	5655[a]		

[a] 360 mm effective screening length when excluding the blind part at the stiffened ring connection.

For all sievings (horizontal and rotary), identical sieve hole diameters were applied. They were round holes of 3.15, 8, 16, 45 and 63 mm, all screens followed the requirements of ISO 3310-2 [12].

Image analysis

For the photo-optical analysis, a commercially available classifier was used (Haver-CPA 4 RT Band, by Haver & Boecker, Germany). In this set-up, the sample is fed into a container, which serves as a feeder hopper discharging the particles to a vibrating feed canal. At the end of the canal the particles are dropped onto a conveyer belt which singularises the particles by transporting them at an approximately 22 times higher speed than on the vibrating feed canal (belt speed: 0.9 m s^{-1}). Before the particles are falling off the end of the conveyer belt, they pass a linear light source whose light is continuously registered by a digital CCD horizontal line camera on the opposite side of the light source (see Fig. 3.2.1). This camera records 4096 pixels over a width of 400 mm, thus the resolution per pixel is 98 µm. The camera processes 40 million pixels per second, whereby the matrix-equivalent resolution conforms to 24 megapixels. When a particle passes the projection plane of the camera, the incoming light is disturbed at a width, which is proportional to the particle's momentary horizontal expansion. From the retention time within the camera's scope and the recordings for the varying horizontal expansion, the size of each particle's shadow is recorded and calculated by a computer. This calculation assumes a constant particle velocity at the measuring plane. Overlapping particles are identified as one particle of respectively larger dimension. Therefore, the singularisation as well as the horizontal spreading of the particles on the conveyer belt needs to be controlled carefully.

3.2.2.3. Measuring procedure

All round robin partners followed common guidelines, which were elaborated based on the experience from pretests. Prior to measurements, the 15 samples were placed in the laboratory under constant climate conditions for minimum 24 h, thus avoiding any larger moisture changes during the processing. In all trials, the sample size was around 81; samples were homogenised by hand before the tests. To identify any particle losses during measurement, the mass of the samples was determined prior to the tests.

For the horizontal shaking screens, a maximum sample layer of approximately 5 cm was placed on the upper tray. In case more sample material had to be determined, the sample was divided in halves and the procedure was repeated, then the masses of the different fractions were unified and weighed. From preliminary tests, the duration of the screening operations was set between 11 and 16 min, depending on the specific device. In pre-tests, any further screening had led to insignificant mass changes of below 0.3% min^{-1}, determined between any two consecutive sieves, related to the total sample mass. Any particles sticking in the holes of a tray were added to the sieve oversizes of the respective tray.

In the rotary screening device, the samples were placed in eight equal 1-l heaps on a tray from where they were slowly poured by hand into the infeed slide of the drum. Thus, a more or less constant feeding rate of 11 min^{-1} was achieved.

In case that particles were found as sieve oversize on the 63-mm screen (in horizontal screening) or were discharged from the 63-mm sieve to the final collecting pan (in rotary screening), the maximum expansion of the largest particle was measured by hand using a calliper gauge. This information was required for the calculation of the mean particle size and the size distribution curve.

For horizontal screenings all samples were tested at four laboratories (five different devices) in three replications. Rotary screenings were tested at three laboratories (three devices) in three replications per sample. For the image analysis, the number of replications was increased to five in order to gain enough results for repeatability calculations since this equipment was available at only one partner. The feeding rate was set individually for each fuel, whereby it was attempted to minimise overlapping particles or particles tangent to each other as much as possible by visual inspection. These overlappings were higher for fine than for coarse fuels, thus the processing duration for each 8-1 sample varied between 5 and 20 min. In all trials, the online classification was made according to the maximum particle expansion applying the "maximum length mode" of the device. This length is calculated from the identified longest distance between two pixels of a computed cohesive particle area, it is then defined as the length axis. The maximum width of a particle is calculated from the longest distance between two pixels on the axis perpendicular to the length axis. This is done simultaneously with the length determination.

3.2.2.4. Calculation procedures

For data evaluation and statistical processing, the measured size allocations were reduced to a single parameter. Here the median value of the particle collective was chosen because it is less susceptible towards extreme values or outliers than the mean particle size. This applies for example when few bigger particles do not fall through a designated screen hole. The median value is defined as the calculated particle size where 50% of the particle amount is below and 50% is above. Therefore, the distribution is separated into two halves. Graphically, the median value is established by the intersection of the cumulative distribution curve with the 50%-line (see also Fig. 3.2.4 and Fig. 3.2.7). For screening and stereometric separation, the particle amount is given by mass, for image analysis it is given by the particle's projection area, which is continuously calculated during the recording. Consequently, the calculated fractions are either the mass of all particles in the respective size class related to the total mass (for the screening and stereometric method) or the aggregated area of all particles in a size class related to the total aggregated particle area (for image analysis) which are set as 100% sample amount.

The image analysis of particles also allows a calculation of the sphericity which is a parameter to describe the shape of a particle. Its calculation follows Eq. (1):

$$\psi = \frac{C}{2\sqrt{\pi A}} \qquad (1)$$

where ψ is the sphericity, $\psi \geq 1$; C is the circumference of the projection area of the regarded particle; and A is the projection area of the regarded particle ("shade") in image analysis.

Here, the sphericity is the measured circumference of the projection area of a particle divided by the circumference of a circle equal in size. In the case of a perfect sphere shape (round projection area), the sphericity of the particle is $\psi = 1.0$. The more a particle deviates from a round shape, the higher is the sphericity. Sphericity was determined for all 13 conventional round robin fuels. Due to an accidental contamination of the standard fuels in the course of the round robin—most likely by human hairs—the sphericities of the standard fuels (SF1, SF2) could not be considered here. Other than for size distribution, sphericity is very sensitive to such contamination because small particles are treated equal to large particles in the computation of mean values.

Repeatability and reproducibility of the results were tested by comparison of the relative repeatability (r) and reproducibility

limits (*R*) according to ISO 5725-1 [13]. Both parameters were calculated with the pre-setting that individual test results shall differ less or are equal to a probability *P* of 95% under repeatability or reproducibility conditions.

3.2.3. Results and discussion

3.2.3.1. Tests with standard fuels

The results from trials with the standard fuels show that the methods tested for particle size determination (image analysis, horizontal and rotary screening) are largely incompatible. Compared to all other methods, a horizontal screening operation generally tends to overestimate the share of small particles when a classification by length is desired. If the particles are to be classified by width, there is a higher compliance of the screening results for wood chips (SF1) with the results from the image analysis. This, however, is not true for the hog fuel sample (SF2), where the reference median value (width) is overestimated by screening. This is indicated in Figs. 3.2.5 and 3.2.6 where the 100%-line represents the reference value for the median of either the particle length or the particle width distribution, both determined by stereometric measurement.

For the length, which is commonly regarded as the useful fractionating criterion, the highest conformity with the reference median value is given by the measurement with the image analysis system. This applies particularly for the wood chip sample, while there is a 13% difference to the reference method for hog fuels (Fig. 3.2.5). This is probably due to a less effective singularisation for such fuel types, which may be more susceptible to overlapping positions on the conveyer due to the rougher surface of hog fuels which thus may inhibit the particles to slide off from each other while being transported through the measuring plane of the camera. The higher roughness of the hog fuel particles is caused by the comminution with blunt tools which is rather a smashing than a cutting process.

For the rotary classifying system, a relatively high conformity with the reference median particle length is given when wood chips are measured (sample SF1). However, this technique has some drawbacks when using hog fuels (SF2 in Fig. 3.2.5). Nevertheless, for the chip sample (SF1), the results from rotary screening come much closer to the reference values than those of any of the horizontal screening devices.

The underestimation of particle lengths by horizontal screening can also be read from Fig. 3.2.7, where the position of the respective size distribution curve from the screening methods was offset to the left-hand side compared to the reference length distribution curve.

For horizontal screening systems, the particle width seems to be a more decisive dimension than the particle length; this is indicated by the higher compliance with the reference value in Fig. 3.2.6. It can be explained by the fact that long and thin particles can pass the screen holes vertically. Such conclusions were also made for particles of less than 5 mm sieved by metal wire cloth screens [14].

The phenomenon of vertically passing particles is reduced by using rotary screening equipment, where the passage through the screen holes is limited by the retention time of a hole below a particle during each rotation. Consequently, long and thin particles are partly detained from slipping through the aperture. This had been intended by the designers of the equipment and therefore the rotary screen can be characterised as a measuring system for a deliberate incomplete screening. For comparable results, it is however important that the screening time, which is determined by a combination of angle and rotation speed, is always kept constant.

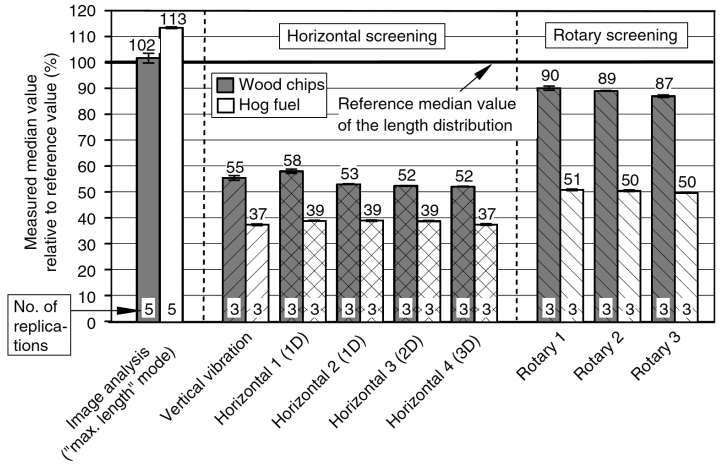

Fig. 3.2.5. Measured mean median values and standard deviations for the standard wood chips (SF1) and hog fuel (SF2) sample relative to the respective reference median value of the length distribution as determined by stereo-metric method (1D, 2D and 3D = one-, two- and three-dimensional shaking operation).

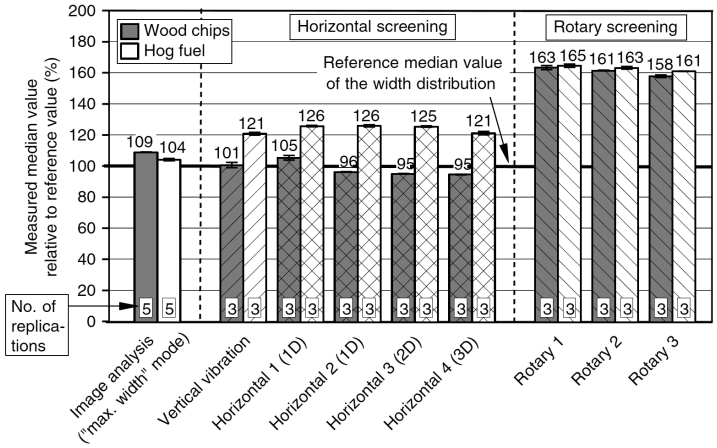

Fig. 3.2.6. Measured mean median values and standard deviations for the standard wood chip (SF1) and hog fuel (SF2) sample relative to the respective reference median value of the width distribution as determined by stereo-metric method.

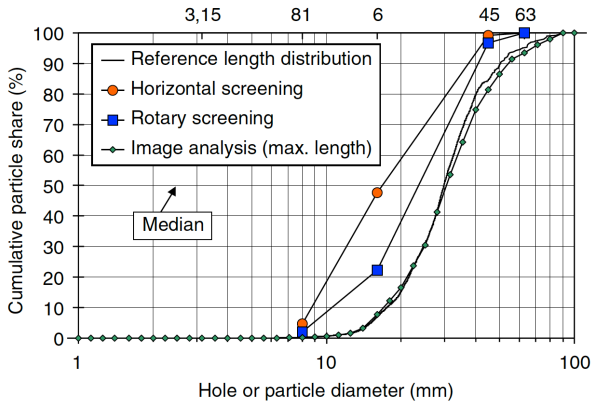

Fig. 3.2.7. Cumulative particle size distribution, examples for different testing methods applied on the standard wood chips sample.

The degree to which oversized particles pass the screen holes vertically can be read from Table 3.2.2, where all wrong allocations are quantified. Their recognition was made by the different colouring of the fractions which allowed a separation by hand followed by subsequent weighing of all separated size classes on a sieve (see also Fig. 3.2.2). Thus, the misplacement in Table 3.2.2 must be interpreted as the mass of particles which do not belong on the sieve relative to the total mass of the fraction (chip colour) as determined by stereometric means according to the particle length. For larger size classes, the misplacement is higher than for smaller ones. Above 45 mm almost all particles are longer than the hole diameter which they pass. For image analysis, the trend is rather the opposite, as "misplacement" is in this case a function of the number of overlapping particles, but the degree of misplacement is generally much lower. As expected, the total level of particle misplacement is largely higher for hog fuel than for wood chips (see Table 3.2.2). This is true for all devices tested.

3.2.3.2. Tests with conventional fuels

The conventional round robin fuels (mainly wood chips and hog fuels) performed similar to the standard fuels (Table 3.2.3). Screenings of hog fuel samples, such as shredded spruce or the standard hog fuel sample (SF2), had a high relative deviation of median values to image analysis. Generally, deviations were larger when comminution was made with blunt tools. This can be read from the fact that all shredded material and the standard hog fuel sample, which are known to have been produced by a more crushing

Table 3.2.2. Share of misplaced particles from the two standard samples (for horizontal and rotary screens, the mean values of the participating laboratories are given).

Fraction (length in mm)	Share of mass (%)	Shares of misplaced materiala (%)		
		Image analysis (max. length)	Horizontal screens	Rotary screens
Wood chip standard sample (SF1)				
> 3.15-8	0.4	39.4	3.4	2.2
> 8-16	6.2	11.4	62.2	24.6
> 16-45	77.4	3.7	55.5	21.9
> 45-63	11.4	19.7	99.7	96.5
> 63	4.7	6.1	100.0	100.0
Average misplacement[b] (%)	26.7	62.9	34.2	
Hog fuel standard sample (SF2)				
> 3.15-8	0.6	43.5	8.7	3.3
> 8-16	6.3	16.7	57.1	26.8
> 16-45	79.0	5.0	89.4	64.6
> 45-63	10.5	10.8	100.0	99.8
> 63	3.7	8.0	100.0	100.0
Average misplacement[b] (%)	56.0	88.4	66.9	
Overall average misplacement[c] (%)	41.4	75.7	50.6	

[a] Total of shares above and below cut size referring to weight of sample fraction (reference measurement).
[b] Total of shares referring to total sample weight.
[c] Total of shares referring to total sample weight for both standard samples (SF1, SF2).

Table 3.2.3. Conventional and standard round robin fuels: deviations of median values from size distribution by screening devices compared to the median value as determined by image analysis (particle length).

Tested fuel	Median length (by image analysis) (mm)	Relative deviation to median value by image analysis (left column)		
		Horizontal screens (%)[a]	Vertical vibration (%)[b]	Rotary screens (%)[c]
Finer fuels, P16				
Spruce, shredder[d]	29.73	-80.7	-77.4	-66.8
Spruce, drum chipper	27.84	-72.9	-72.5	-60.1
Beech, shredder[d]	29.30	-66.7	-65.1	-55.8
Spruce, standard hog fuel (SF2)[d]	33.22	-66.1	-67.1	-56.3
Beech, drum chipper	26.06	-58.8	-59.0	-50.3
Poplar (SRC), disc chipper	20.83	-53.5	-51.3	-42.7
Spruce, disc chipper	18.30	-49.8	-48.6	-37.1
Beech, disc chipper	16.36	-49.6	-51.1	-35.6
Average	-	-62.3	-61.5	-50.6
Medium fuels, P45				
Spruce, logging residues, disc chipper	38.87	-51.7	-49.3	-32.1
Beech thinnings, disc chipper	25.83	-50.1	-49.7	-41.7
Spruce, logging residues, high needle content, disc chipper	40.06	-47.3	-46.7	-32.0
Spruce, broad range blend, disc chip	38.80	-44.8	-43.3	-27.9
Spruce, standard wood chips (SF1)	30.28	-40.7	-45.5	-14.0
Beech, spiral chipper	44.27	-35.9	-32.8	-29.5
Average	-	-45.1	-44.6	-29.5
Coarse fuels, P63				
Spruce, spiral chipper	67.44	-46.1	-44.5	-33.6
Overall average		-54.3	-53.6	-41.0

[a] Mean values for four horizontal screening devices.
[b] Using flat screens in horizontal orientation.
[c] Mean values for three rotary screening devices.
[d] Hog fuel sample.

than cutting action, appear among the largest deviations in Table 3.2.3. Deviations were also higher for spruce than for beech wood which could be explained by the fact that spruce wood is mostly longer fibred and therefore any crushing action after the cutting by the chipper (e.g. in the calibration screen) will easier lead to breaks over the length axis rather than across the fibre line. This would result in more longish than round particles.

From the results, it can be seen that samples with a high mean particle sphericity—which for example means a large share of long and thin particles—have a high tendency for deviations from the image analysis results (here chosen as reference values). This is indicated by the regression analysis in Fig. 3.2.8. Although the coefficient of determination (R^2 = 0.526) is relatively low, the correlation is statistically significant at the 5% error level.

For the finer fuels, which would be classified as wood chips "P16" according to the recently published European technical specification for solid biofuels [11], the deviations of the screening methods to the reference

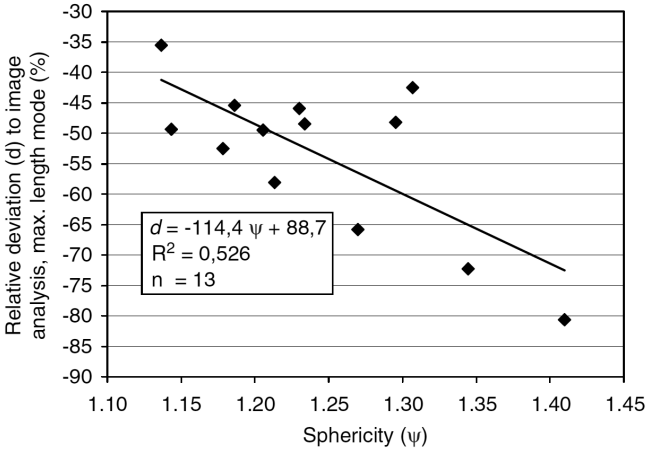

Fig. 3.2.8. Linear regression analysis for the relative deviations (d) of the median values by horizontal screening ("Horizontal 1") from image analysis results as a function of the mean particle's sphericity (ψ). Image analysis results gained in "max. length" mode measured with the conventional fuel samples.

image analysis results are higher than for medium or coarse wood chips (P45 or P63). This can be read from the average results in Table 3.2.3, it reflects the different moments of inertia which the particles are exposed to before they pass a screen hole.

Smaller particles are easier to be brought into a favourable vertical orientation towards the respective screen holes because the required torques are lower. Further explanation for the observed deviations can be seen in the fact that the gravimetric methods (screening) are always susceptible towards an inhomogeneous moisture distribution in the sample, while this influence can be excluded for the image analysis. If smaller particles in a sample are drying more rapidly than larger ones, they would contribute a lower mass share to the total sample mass and thus cause a displacement of the median value.

3.2.3.3. Tests on repeatability and reproducibility

The average relative repeatability limits *r* for horizontal (1.2%) and rotary screenings (1.8%) are exceedingly low. This is shown in Table 3.2.4. For the image analysis, the repeatability was slightly worse compared to screening (particularly compared to horizontal screening devices).

The relative reproducibility limits *R,* which characterise the variation between the laboratories, cannot be compared here consistently because the image analysis was available at only one laboratory. Moreover, interpretations are difficult due to the low number of devices and laboratories involved in the round robin and the samples are subject to mechanical wear which could have contributed to the observed variation. Generally, however, the relative reproducibility limits based on the median values for horizontal and rotary screening results seem acceptable. On average, reproducibility was better for horizontal screenings than for rotary screening (Table 3.2.4).

3.2.4. Conclusions

Particle size analysis of biofuels is a difficult task which is associated with high measuring uncertainties if different measuring principles are applied. In horizontal screening, a severe underestimation of the particle length is given, while some improvements are found for the rotary screening method. Results from both screening methods are highly repeatable

Table 3.2.4. Mean relative repeatability and reproducibility limits according to ISO 5725-1 [13] for the median values of conventional and standard fuel samples as determined by the tested measuring principles (significance level α = 0.05, probability P = 95%).

Measuring principle	P16 (fine)	P45 (medium)	P63 (coarse)	Average[a]	*n*[b]
Relative repeatability limits *r* (%)					
Horizontal screening	0.9	1.7	0.9	1.2	180
Rotary screening	1.2	1.8	6.3	1.8	135
Image analysis («max. length» mode)	1.9	3.1	5.4	2.6	75
Relative reproducibility limits *R* (%)					
Horizontal screening	4.5	10.2	6.5	6.9	4
Rotary screening	12.0	7.7	14.2	10.4	3

[a] Mean value for the total number of measurements.
[b] Number of measurements for repeatability limits *r;* number of devices/laboratories for reproducibility limits *R.*

but a direct comparability is not given. The highest conformity with the reference values is given for an image analysis system. But this technology is still relatively costly, therefore its major focus of application is presumably given when large sample volumes are processed or when frequent sampling is required. As long as the widespread horizontal screening methods are still common in practice, the image analysis can preferably be applied when conformity to such measurements is not required. This applies for example in an internal quality assurance system for large biofuel suppliers or purchasers. Here the additional benefits of this technology, such as combinations with automated sampling processes or the additional measuring features (e.g., the mean particles' sphericity or the mean length-width ratio), could also be utilised. Nevertheless, it would be useful to launch a standardisation process in order to include the image analysis method to the scope of applicable standard laboratory principles for biofuels, too. This would be a first step to overcome the disadvantages of the screening methods.

Acknowledgements

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3.3. Particle density determination of pellets and briquettes

Several methods and procedures for the determination of particle density of pellets and briquettes were tested and evaluated. Round robin trials were organized involving five European laboratories, which measured the particle densities of 15 pellet and five briquette types. The test included stereometric methods, methods based on liquid displacement (hydrostatic and buoyancy) applying different procedures and one method based on solid displacement.

From the results for both pellets and briquettes, it became clear that the application of a method based on either liquid or solid displacement (only tested on pellet samples) leads to an improved reproducibility compared to a stereometric method. For both, pellets and briquettes, the variability of measurements strongly depends on the fuel type itself.

For briquettes, the three methods tested based on liquid displacement lead to similar results. A coating of the samples with paraffin did not improve the repeatability and the reproducibility.

Determinations with pellets proved to be most reliable when the buoyancy method was applied using a wetting agent to reduce surface tensions without sample coating. This method gave the best values for repeatability and reproducibility, thus less replications are required to reach a given accuracy level. For wood pellets, the method based on solid displacement gave better values of repeatability, however, this instrument was tested at only one laboratory.
Keywords: Particle density; Pellets; Briquettes; Liquid displacement; Solid displacement; Stereometry.

Détermination de la masse volumique nette des pellets et des briquettes. Plusieurs méthodes et procédures ont été testées et évaluées afin de déterminer la masse volumique nette de pellets et de briquettes. Des tests inter-laboratoires auxquels ont participé cinq laboratoires européens ont été organisés. Au cours de ceux-ci, les masses volumiques nettes des types de pellets et de cinq types de briquettes ont été mesurées. Ces tests comprenaient des méthodes stéréométriques, des méthodes basées sur le déplacement des liquides (hydrostatique et flottabilité) auxquelles ont été appliquées différentes procédures. Une méthode était basée sur le déplacement des solides.

À partir des résultats obtenus à la fois pour les pellets et les briquettes, il est devenu clair que les méthodes basées sur les déplacements de liquides, ou de solides (testées uniquement sur des échantillons de pellets) conduisent à une amélioration de la reproductibilité, lorsqu'elles sont comparées aux méthodes stéréométriques. Pour les pellets, comme pour les briquettes, la variabilité des mesures dépend fortement du combustible lui-même.

Pour les briquettes, les trois méthodes testées, basées sur le déplacement des liquides, conduisent à des résultats similaires. Une imprégnation des échantillons à la paraffine, n'améliore pas la répétabilité et la reproductibilité des mesures.

Les mesures les plus fiables sur des pellets ont été obtenues avec la méthode flottabilité en utilisant un agent mouillant, pour réduire la tension de surface et un échantillon de pellets sans imprégnation de paraffine. Cette méthode a donné les meilleures valeurs de répétabilité et de reproductibilité, un plus faible nombre de répétions est donc requis pour atteindre un niveau de précision donné. Pour les pellets de bois, la méthode basée sur le déplacement des

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solides a donné de meilleures valeurs de répétabilité, cependant cet équipement n'a pu être testé que par un seul laboratoire.

3.3.1. Introduction

Several national standards [1-3] describe the particle density of pellets and briquettes as a quality indicator of densified fuels. It is also defined as quality parameter for briquettes in the technical specifications prCEN/TC 14961 [9].

Moreover, this property has an influence on bulk density and on combustion properties of pellets and briquettes (heat conductivity, burning time and rate of degasification) [4,5,11]. The determination of this parameter, which is defined as ratio of the mass and the volume of a sample including pore volume, faces several difficulties. While mass measurements are easy to realise, the volume determination is connected with several uncertainties. Irregular particle shapes and surface roughness increase the result variability, particularly for stereometric methods. Moreover, the hygroscopic properties of compressed wood fuels, which seem to depend on the type of raw material [6] introduce incertainties for the volume estimation by liquid displacement methods. These are mostly due to the liquid penetration into the compressed fuel, the bonding forces and the dissolving of the fuel's shape. Finally, the hygroscopic properties of pellets and briquettes not only act on the mass of the sample but also influence its volume [5].

This paper aims at comparing different methods for the estimation of the particle density of pellets and briquettes. It focuses on stereometric methods and determinations applying liquid and solid displacement. These methods have been tested in an international round robin by five different European laboratories using 15 pellet and five briquette types.

3.3.2. Materials and methods

3.3.2.1. Pellets sample selection

The 15 selected pellet types (Table 3.3.1) were produced in five different European countries and were all commercially available. The selection includes wood pellets with different diameter and pellets made from agricultural residues. The moisture contents of the pellets were between 6% and 10% (w.b.).

Table 3.3.1. Description of the different pellet types selected for testing.

Pellets code	Diameter (mm)	Raw material
P1	6	Mixed wood
P2	6	Softwood
P3	6	Hardwood
P4	6	Hardwood
P5	8	Mixed wood
P6	8	Mixed wood
P7	8	Mixed wood
P8	6	Mixed wood
P9	6	Mixed wood
P10	9	Straw
P11	6	Miscanthus
P12	6	Softwood
P13	6	Softwood
P14	8	Hay
P15	9	Straw

3.3.2.2. Briquettes sample selection

The five briquettes selected were produced in four different European countries and were all commercially available. Their origin, production process, shape and raw material are listed in Table 3.3.2. As for pellets, the moisture content was measured

before testing. The moisture contents of all briquettes were below 10% (w.b.).

3.3.2.3. Tested methods

Particle density is calculated as the ratio of the mass to the sample volume including pore volume. Even though the moisture content seems to have an influence on the particle density [7], in this study, the increase of the volume, depending on the moisture content, is neglected. The particle density is thus expressed for moisture contents as "as received". All participating laboratories followed the same guidelines, which had jointly been elaborated in order to exclude effects caused by inconsistent handling procedures.

Table 3.3.3 shows the tested methods and the number of laboratories involved in the round robin trials. Each participating laboratory performed 15 replications for each tested fuel and each tested method.

Liquid displacement methods

Liquid displacement methods are based on the Archimedes principle. The volume of a sample is estimated by the mass of the volume that is displaced while the sample is submerged in liquid. However, those methods have two major disadvantages. First, due to hygroscopic fuel properties, liquid may be absorbed. Second, air bubbles may build up on the sample surface while submerged due to surface tension. In order to avoid

Table 3.3.2. Description of the different briquette types selected for testing.

Briquette code (Origin)	Production press	Cross section	Shape	Raw material
B1 (France)	Extruder	Hexagonal		Mixed wood
B2 (Germany)	Extruder	Square		Hardwood
B3 (Germany)	Chamber	Rectangular		Mixed wood
B4 (Spain)	Piston	Circular		Mixed wood
B5 (Austria)	Piston	Circular		Softwood

Table 3.3.3. Particle density: tested methods and number of laboratories involved for briquettes and pellets.

Tested methods	Number of laboratories involved in briquette testing	Number of laboratories involved in pellet testing
Stereometry 1[a]	5	4
Stereometry 2[b]	3	Not tested
Hydrostatic and paraffin coating	4	4
Hydrostatic and wetting agent	Not tested	3
Buoyancy without paraffin coating	2	Not tested
Buoyancy and paraffin coating	4	4
Buoyancy and wetting agent	Not tested	3
Solid displacement method	Not tested	1

[a] Stereometrical cross-section calculation.
[b] Cross-section calculation by cut out paper.

the impact of these disadvantages, samples may be coated, e.g. with paraffin, while the addition of wetting agents is used to reduce surface tension.

The tests on liquid displacement methods were performed in pure water, (on paraffin-coated or non-coated samples) and in a mix of water and wetting agent (for non-coated samples). The applied paraffin had a melting point between 52 and 54 °C. The added wetting agent was t-Octylphenoxypolyethoxyethanol; polyethylene glycol tert-octylphenyl ether (CAS number: 9002-93-1; trade mark Triton X-100), at a concentration in water of 1.5 g/l.

The pellet sample, between 4 and 8 g, was weighed to the nearest 0.0001 g while the briquette sample (each single briquette) was weighed to the nearest 0.1 g.

For the volume calculation, the density changes of the liquid caused by different temperatures, was taken into account.

The hydrostatic method. In the hydrostatic method, the mass of the liquid which is displaced by the sample was determined. A glass beaker filled with liquid (water or mix of water and wetting agent) was placed on the weighing platform of a balance. The sample material was placed in a cage that was fixed at a vertically moving arm of a tripod.

The liftable arm realised the submergence of the sample filled cage, which should have no physical contact with the beaker glass when under water (Fig. 3.3.1). The mass difference between the empty system and the system with a sample placed in the cage allows the volume determination of the sample and the calculation of the particle density (ρ_u) according to Eq (1)

$$\rho_u = \frac{m_u}{m_{w.dis}} \rho_w \qquad (1)$$

where ρ_w is the density of liquid at a given temperature, m_u is the weight of the test sample in air and $m_{w.dis}$ is the weight of liquid displaced by the test sample.

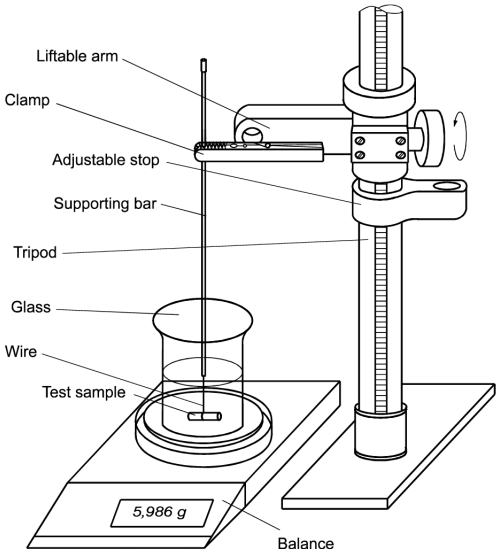

Fig. 3.3.1. Volume determination with the hydrostatic method [4].

The buoyancy method. In the buoyancy method, the apparent weight of the sample during submergence is determined, the buoyancy is the difference between the sample's weight in air and its apparent weight in liquid. In contrast to the hydrostatic method, the weighing platform is not loaded with a liquid container. For briquette volume determination, the liquid container was positioned underneath a balance, which was equipped with a below-balance weighing hanger (Fig. 3.3.2). Pellet testing was conducted by the use of a particle density determination kit provided for the balance by the manufacturer (Fig. 3.3.3). The kit provides a bridge for the weighing plate where the beaker glass was placed on the bridge in order to prevent any direct load on the weighing plate. The sample volume was determined by the difference between the mass of a sample in air and the mass of the same sample submerged in the liquid. The particle density (ρ_u) of the sample is calculated according to Eq. (2).

$$\rho_u = \frac{m_u}{m_u - m_s} \rho_w \qquad (2)$$

where ρ_w is the density of liquid at a given temperature, m_u is the weight of the test sample in air and m_s is the weight of the sample in liquid.

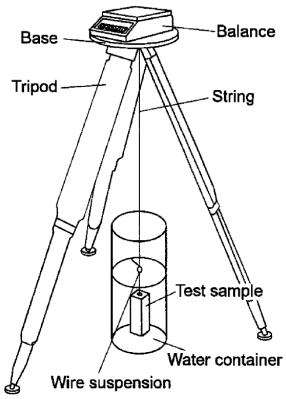

Fig. 3.3.2. Volume determination with the buoyancy method (measurement by the bottom of a balance).

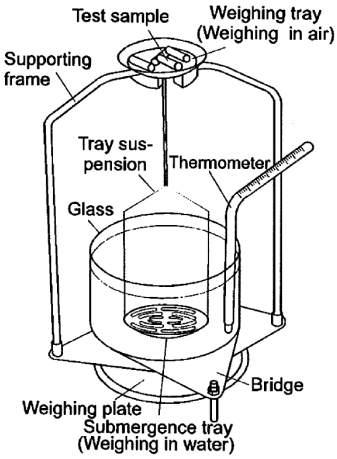

Fig. 3.3.3. Volume determination with the buoyancy method (kit [4]).

Paraffin coating. The paraffin coatings were been performed by immersing the sample in a bath of liquid paraffin (temperature: 100 °C). Samples were weighed before and after the paraffin coating, in order to calculate the mass of the applied paraffin. The paraffin density was 900 g/l.

Stereometric methods

Stereometric methods are based on the measurement of the dimensions (e.g. diameter, length, width, height) of a single regularly shaped particle using length-measuring instruments (e.g. calliper gauge, rule). The volume of the sample is then determined by calculating the volume of the nearest regular geometrical shape (cylinder, cuboid, cube).

Stereometric measurements on pellets. A single pellet sample was weighed to the nearest 0.0001 g. All size measurements were made at a precision of 0.1 mm. In the calculation the pellet was considered to have the shape of a cylinder.

The convex or concave endings of each pellet were removed perpendicularly to the axis of the pellet. The extremities were cut and sanded in order to obtain smooth sides. The length of the pellet was then measured twice (measures were taken at the right angle of each other). The diameter was measured six times, twice at each end and twice in the middle of the pellet; between two successive measurements the pellet was always rotated by 90°.

Stereometric measurements on briquettes. A single briquette sample was weighted to the nearest 0.1 g. All the measures were made at a precision of 0.1 mm. The irregular sides of each single briquette were removed, the extremities were cut by a blade or a band saw at a right angle to the axis of the briquette.

The length of each briquette was determined twice by a calliper gauge (precision 0.1 mm). Where the briquette had a central hole, the diameter if this hole was measured (twice, perpendicular, at each ending) and the hole volume was subtracted from the total volume.

For the calculation of the briquette's cross-section two different approaches were applied. As done for pellets, the first approach was based on measuring the three dimensions of the briquette and to estimate its volume by using geometric formulaes. The second approach aimed at estimating the surface of the briquette's cross-section by the use of a paper sheet of known dimensions. The sheet was weighed (to the nearest 0.0001 g), afterwards the briquette was placed on the paper in such a way that the contour of the briquette could be drawn on the paper. Finally, the briquette's cross-section was precisely cut by a pair of scissors and weighed. The mass relation between the cut piece of paper and the original sheet mass enabled the calculation of the cross-section area of the briquette. This cross-section was then multiplied by the length of the sample in order to estimate its volume.

Solid displacement method

A commercially available powder pycnometer (GeoPyc 1360 by Micromeritics, USA, Fig. 3.3.4) was used. In this method the apparent volume is determined by the displacement of a powder ("DryFlo"). At first an empty measurement is made without any sample in order to measure the initial volume of the powder. A plunger is moved step by step until a predefined consolidation force is reached; here the position of the plunger in the cylinder is recorded. Then the actual measurement was performed by adding the sample into the cylinder and repeating the same procedure as for the blank test using the same consolidation force. The volume was calculated from the difference of the two plunger positions, before and after sample addition. Only one partner tested this equipment and as it is a non-destructive method it was also used to determine the penetration coefficient of paraffin for pellets (see Section 3.3.2.4).

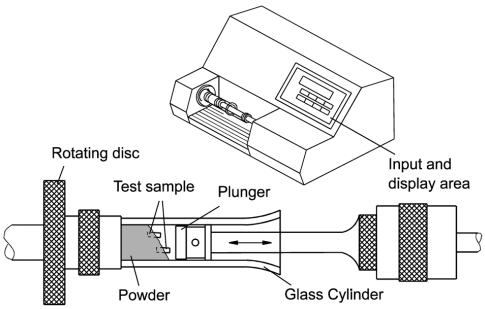

Fig. 3.3.4. Volume determination of pellet samples with the displacement of solid method (pycnometer [4]).

3.3.2.4. Analysed parameters

In order to evaluate the influence of each tested method and each selected fuel on the particle density results, individual and global mean values and standard deviations were calculated for the 15 replications at each laboratory and for all participating laboratories. Moreover, the relative and absolute repeatability and reproducibility limits were calculated following the ISO 5725.2 [8]. For pellets, repeatability and reproducibility were calculated for three distinct groups of pellets. A first group consisted of all 15 pellet types tested in the project, a second group consisted of agricultural residue pellets only and a third group consisted of wood pellets only.

The number of replications which are required to ensure several accuracy level of 0.5%; 1%; 2% or 5% was determined following a common statistical calculation procedure according to Dagnelie [10].*

Concerning the paraffin coating different calculations were made in order to evaluate different correction modes for the added

* Le principe de ce recueil de publications est de présenter les contributions scientifiques telles qu'elles ont été publiées par les revues scientifiques auxquelles elles ont été proposées. Cependant, en dépit du soin apporté par les auteurs et par les différents relecteurs, quelques légères erreurs subsistaient dans les versions publiées par les revues. Ce paragraphe a été corrigé par rapport à la version publiée.

volume of the coating. Knowing the mass of applied paraffin and the density of paraffin, the applied volume of paraffin on the sample was determined by calculation.

In the first approach, it was assumed that the total quantity of paraffin remains outside the sample. Therefore, a 4 corrected density (Corr1 density) was calculated by subtracting the calculated volume of paraffin from the measured volume. This calculation was done for briquettes and pellets.

In a second approach (for pellets only), the use of the pycnometer makes it possible to determine the average amount of paraffin which penetrates the sample during preparation (coating). Three of the 15 pellet types were chosen for the measurements (a 6 mm diameter wood pellet, a 8 mm diameter wood pellet and a pellet made of agricultural residues). The three samples were measured twice with and without paraffin coating (10 replications each). The comparison between both volumes and the quantity of applied paraffin allowed the share of paraffin penetration for each of the three pellet types to be determined.

These paraffin penetration shares were applied on all tested pellets (according to the raw material and the diameter) to calculate a second corrected density (Corr2 density) by regarding only the paraffin volume, which remains on the pellet's surface. The corrected densities (Corr1 and Corr2) were compared to the non-corrected density (Nc density).

3.3.3. Results and discussion for pellet trials

3.3.3.1. Mean values and standard deviation

Fig. 3.3.5 shows the results and standard deviations of the tested particle density determination methods for 15 pellet types (mean values for all laboratories). Concerning the variability of the results, a tendency depending on the single pellet types is observed. This was confirmed by an ANOVA (two-way ANOVA method and pellet types, fixed model) which shows that the single pellet type has a significant influence and the method has a highly significant influence on the variances.

In fact, the variability is influenced by the value of the particle density itself. Variations are highest for low-density pellets, while they

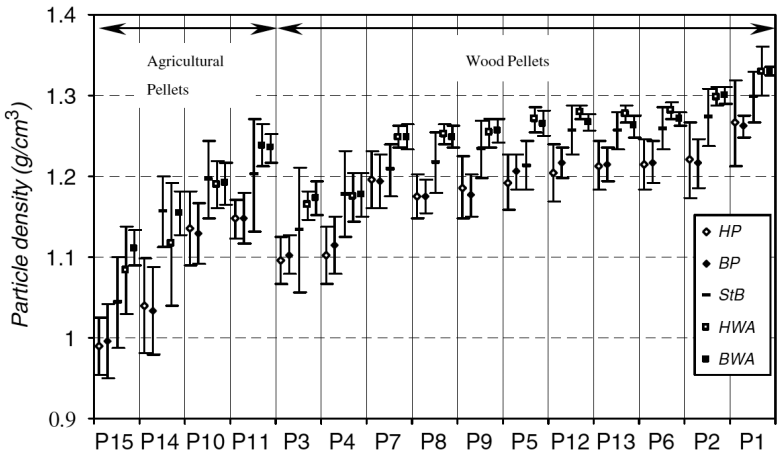

Fig. 3.3.5. Particle density of 15 pellet types estimated by 5 different methods (mean value and standard deviation of involved laboratories). HP, hydrostatic with paraffin coating; BP, buoyancy with paraffin coating; StB, stereometric measurements; HWA, buoyancy with wetting agents; BWA, buoyancy with wetting agents.

are lowest for high-density pellets. This was confirmed by a ANOVA analysis (two-way analysis: diameter and raw material) on the variances with the density as a co-variable.

For agricultural residues pellets and low-densities wood pellets (density < 1.2 g/cm³), stereometric determinations lead to a higher variability (variance of 0.062 (g/cm³)²) compared to the liquid displacement methods, particularly when the buoyancy method with addition of wetting agent is used (variance of 0.019 (g/cm³)²).

Concerning wood pellets with higher particle densities, as test of variance equality has showed that methods using wetting agent give equivalent variances (0.028 (g/cm³)²) which are significantly lower to the ones obtained with the three other methods.

Due to the paraffin coating, densities obtained with methods using paraffin (hydrostatic and buoyancy) are lower than for the other methods.

The comparison between hydrostatic and buoyancy methods with paraffin and wetting agent was made with ANOVA (a two-way variance analysis, methods and partners, fixed model). This analysis has shown that buoyancy and hydrostatic methods—no matter whether paraffin coatings or wetting agents are applied—seem to produce similar results and can therefore be regarded as compatible. It was also found that the influence of the laboratory on the result is highly significant. Additionally an interaction between the laboratory and the method factors was observed which means that for some laboratories the mean values of particle density obtained with both methods can be different.

As the solid displacement method was only tested by one laboratory the results shown in Fig. 3.3.6 are those obtained for this laboratory only. For low-density pellets, the variability of the results obtained with the method based on solid displacement is comparable to the one obtained with the liquid displacement methods. For the wood pellets with higher densities, the variations are smaller.

3.3.3.2. Repeatability, reproducibility limits and number of replications

Table 3.3.4 shows the relative and absolute repeatability and reproducibility limits of the tested particle density determination methods.

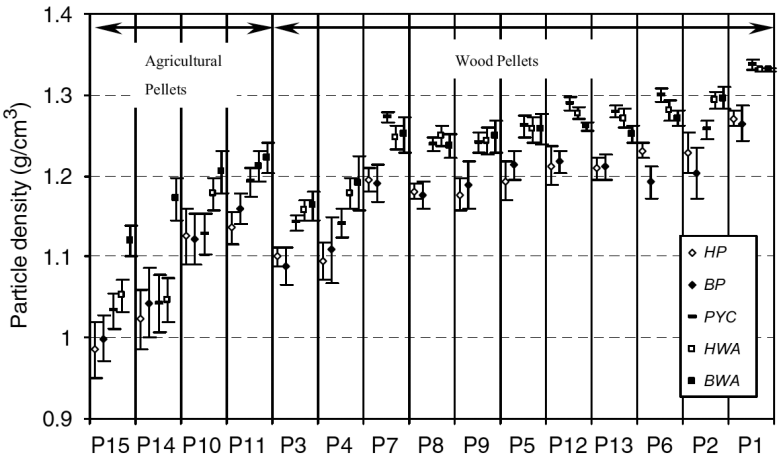

Fig. 3.3.6. Particle density of 15 pellet types estimated by 5 different methods (mean value and standard deviation of one laboratory). HP, hydrostatic with paraffin coating; BP, buoyancy with paraffin coating; PYC, solid displacement method; HWA, buoyancy with wetting agents; BWA, buoyancy with wetting agents.

Additionally, the required minimum number of replications which are necessary to obtain a given accuracy level (0.5%, 1%, 2% and 5%) are given.

The parameters are given for three different groups of pellets: wood pellets, agricultural pellets and all pellets together.

Regarding evaluations of all pellets it is obvious that repeatability limits are higher for stereometric determinations (8.7%) and displacement methods with paraffin coatings (7.1% for hydrostatic and 6.1% for buoyancy) compared to immersion methods where wetting agents are added (4.9% for hydrostatic and 3.5% for buoyancy).

The advantages of the wetting agents method was also confirmed when the laboratory influence is considered (reproducibility limits R), at least this applied for wood pellets. However, for agricultural pellets only the buoyancy method applying wetting agents seems also to result in better reproducibility.

It has to be added that the pellet raw material influences the repeatability and the reproducibility. Pellets from agricultural residues show worse results than wood pellets. The group made of agricultural pellets showed *r*% ranging from 4.56 to 11.55% depending on the method, while repeatabilities were between 2.19% and 7.66% for wood pellets.

The buoyancy method applying wetting agents requires the least replications to achieve a given accuracy level. For example, to secure an accuracy level of 2%, only 8 replications are needed for wood pellets, while 71 replications are necessary for the stereometric method.

Table 3.3.4. Particle density determination of pellets: relative (*r*%) and absolute (*r*) repeatability (mean value through laboratories), relative (*R*%) and absolute (*R*) reproducibility limits and the required number of replications (*n*) to reach a certain accuracy level.

Pellets	Method	*r* (g/cm³)	*r* (%)	*R* (g/cm³)	*R* (%)	*n*			
						0.5%	1%	2%	5%
Wood pellets	St	0.09	7.66	0.12	9.36	1137	284	71	11
	HP	0.08	6.52	0.11	9.59	315	179*	20	3
	HWA	0.04	3.52	0.05	4.13	240	60	15	2
	BP	0.07	5.60	0.08	6.75	124	31	8	1
	BWA	0.04	2.91	0.04	3.47	127	32	8	1
Agricultural residues pellets	St	0.13	11.55	0.16	13.85	644	161	40	6
	HP	0.10	8.89	0.12	11.44	417	104	26	4
	HWA	0.09	7.51	0.16	13.59	391	98	24	4
	BP	0.09	9.00	0.12	11.64	367	92	23	4
	BWA	0.05	4.56	0.07	6.33	79	20	5	1
All pellets	St	0.11	8.73	0.13	10.63	1137	284	71	11
	HP	0.08	7.12	0.12	10.04	417	104	26	4
	HWA	0.06	4.90	0.10	7.80	391	98	24	4
	BP	0.07	6.10	0.09	7.75	367	92	23	4
	BWA	0.04	3.46	0.05	4.40	127	32	8	1

HP hydrostatic with paraffin coating, BP buoyancy with paraffin coating, StB stereometric measurements, HWA buoyancy with wetting agents, BWA buoyancy with wetting agents.

* Le principe de ce recueil de publications est de présenter les contributions scientifiques telles qu'elles ont été publiées par les revues scientifiques auxquelles elles ont été proposées. Cependant, en dépit du soin apporté par les auteurs et par les différents relecteurs, quelques légères erreurs subsistaient dans les versions publiées par les revues. Ce tableau a été corrigé par rapport à la version publiée.

It also appears that it is difficult to achieve accuracy below 1% for the whole range of materials. In fact, even when applying the buoyancy method with wetting agent, 32 replications would be required to meet the 1% accuracy demand considering all the pellet types.

However the data presented here are calculated for the most variable pellet types, when variability is lower, e.g. for pellet type P1, the minimum number of replications can be much lower. Then, for example, only two replications would be enough to obtain a precision of 0.5% when using buoyancy with wetting agent method on pellet type P1.

Concerning the method based on solid displacement, Table 3.3.5 shows the relative (*r*%) and absolute (*r*) repeatability limits obtained by the single laboratory where the tests were made. These results are compared to the buoyancy with wetting agent method.

The method using the powder pycnometer shows a better repeatability for the wood pellets than the buoyancy with wetting agent method (2.34% and 3.88%, respectively), which results in a lower required number of replication. To reach 1% accuracy only nine replications are needed instead of 32 using the buoyancy method with addition of wetting agent.

3.3.3.3. Correction of coating volume

The global mean value of the applied paraffin share (in relation to the total sample weight) is 8.9% in average (through laboratories and pellet types). The share of the applied paraffin on the different pellet types vary from 4% to 22%. The variability between the participating laboratories is important with calculated coefficients of variation ranging between 14% and 37%.

The calculated shares of paraffin penetration for the 3 pellets type was: 68% for pellets made from agricultural residues, 36% for 6 mm wood pellets, and 26% for 8 mm wood pellets. Those penetration shares, which are particularly high for agricultural pellets, show that it is not acceptable to assume that the applied paraffin does not penetrate the sample. Penetration probably depends on the particle density of the fuel itself but it is difficult to extrapolate as the calculation was only done for three pellet types. As explained in Section 3.3.2.4 those penetration shares were used to calculate the Corr2 density.

Fig. 3.3.7 shows the three densities for the hydrostatic method with paraffin coating, similar tendencies were observed for the buoyancy method. Particle density measurements where the volume of the paraffin coating is subtracted from the measured volume (Corr1 and Corr2) are higher than Nc densities. The reason is obvious as the measured volume is reduced, consequently particle density increases. Corr1 density is always superior to Corr2 density because, for Corr2, a lower paraffin volume is subtracted from the total volume since the quantity of paraffin that penetrates the pellet is not considered.

Table 3.3.5. Particle density determination of pellets: relative (*r*%) and absolute (*r*) repeatability (mean value through laboratories) and the required number of replications (*n*) to reach a certain accuracy level.

Pellets	Method	*r* (g/cm³)	*r* (%)	*n*			
				0.5%	1%	2%	5%
Wood pellets	PYC	0.03	2.34	38	9	2	1
	BWA	0.05	3.88	127	32	8	1
Agricultural residues pellets	PYC	0.07	6.70	186	47	12	2
	BWA	0.06	5.38	73	18	5	1
All pellets	PYC	0.05	3.66	186	47	12	2
	BWA	0.05	4.29	127	32	8	1

PYC, solid displacement; BWA, buoyancy with wetting agents.

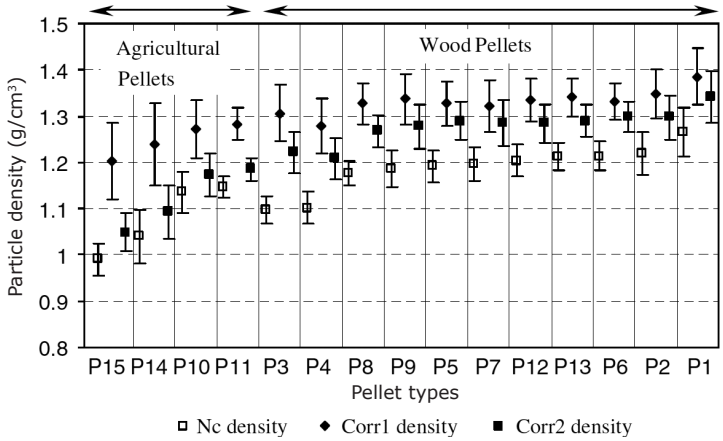

Fig. 3.3.7. Non-corrected density (Nc density) and corrected densities (Corr1 and Corr2 densities), mean value and standard deviation, depending on the pellet types obtained with the hydrostatic with paraffin coating method.

The statistical analysis (one-way analysis of variance, factor: correction) confirms that the three densities differ highly significantly from each other. Additionally, the coefficient of variations of the Corr1 density are also higher than the ones calculated for the two other densities (*t*-test, α = 0.05%).

The introduction of a correction for the volume of applied paraffin does not have a significant influence on repeatability and on reproducibility when the tested wood pellets and the full range of pellets are regarded. In contrast, pellets made from agricultural residues show significantly higher values of reproducibility for Corr1 density compare to the Nc (Table 3.3.6).

The results show that the correction has an important influence either on the value of particle density and, for Corr1, also on the variability of the results obtained amongst laboratories. This is due to the fact that the volume of applied paraffin can be important compare to the initial sample volume and varies from one pellet type to another and also between laboratories.

3.3.4. Results and discussion for briquette trials

3.3.4.1. Mean values and standard deviation

The results of the particle density determination for the five briquettes tested during the round robin test are shown in Fig. 3.3.8 (mean values for all laboratories and standard deviations). It seems that the single-briquette type influences the variability of the results.

Table 3.3.6. Absolute (*R*) and relative (*R* %) reproducibility limits of non-corrected density (Nc) and corrected density (Corr1) for hydrostatic and buoyancy measured on pellets made from agricultural residues.

Methods	*R* (g/cm³)				*R* (%)			
	Hydrostatic		Buoyancy		Hydrostatic		Buoyancy	
	Nc	Corr1	Nc	Corr1	Nc	Corr1	Nc	Corr1
Agricultural residues pellets	0.123	0.221	0.130	0.210	11.44	17.66	11.64	16.95

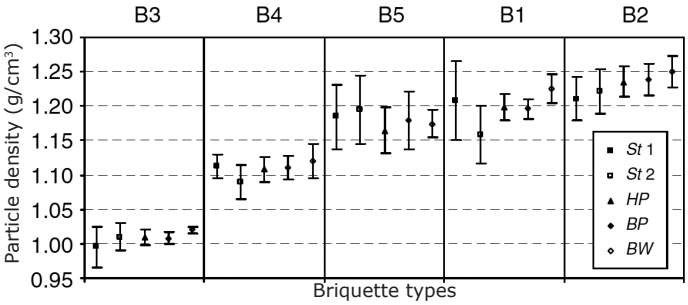

Fig. 3.3.8. Particle density of five briquette types estimated by five different methods (mean value and standard deviation of participating laboratories). St1, St2, stereometry 1 and 2, HP, hydrostatic with paraffin coating, BP, buoyancy with paraffin coating, BW, buoyancy without paraffin coating.

For example, briquettes B5 show higher variability compare to B3 or B4. Briquettes B1 show a higher variability especially for the stereometric methods. This could be due to intrinsic physical properties of the fuel such as the surface roughness and the presence of a central hole. It can also be noticed that stereometric methods lead to more variable results compared to immersion methods.

A two-way ANOVA (methods and laboratories), which was done for each briquette types separately, shows that the two stereometric methods give results which are statistically different for briquettes B1, B3 and B4 and that the influence of the laboratory leads to difference for briquettes B2, B4, B5. No interaction was observed between both parameters. The two-stereometric methods cannot be regarded as equivalent.

The ANOVA also reveals that except for one briquette (B5), methods using samples with paraffin coatings (buoyancy and hydrostatic) do not differ significantly. The influence of the laboratory is significant for four of the tested briquettes (except briquette B3). As for pellets an interaction between both parameters was observed, oneway ANOVA were made (factor: method, fixed model) for each laboratory and the results have shown that for the briquette B5 all labs have given results which differ significantly between HP and BP methods.

For briquettes B1, B3 and B4 only one lab of the four involved in the tests gave different results. Concerning the briquette type B2, no influence of the method was found. Except for briquette type B5, for practical application it is therefore not relevant which of the methods based on liquid displacement is used.

3.3.4.2. Repeatability, reproducibility and number of replications

Table 3.3.7 shows the relative (r%) and absolute (r) repeatability (mean value through all laboratories), the relative and absolute reproducibility (R% and R) of the tested particle density determination methods. Additionally the required minimum number of replications which are necessary to achieve a given accuracy level (0.5%, 1%, 2% and 5%) is given. It is obvious that determinations by immersion methods lead to better repeatabilities and reproducibilities than stereometric measurement. The values of repeatability and reproducibility limits are almost twice as high as for hydrostatic or buoyancy determinations.

It should be mentioned that the reproducibility and repeatability limits calculated for each method clearly depend on the briquette type itself. A test of variance equality (α = 0.05) showed that repeatability and reproducibility differ significantly from one briquette to another.

According to Table 3.3.7, an accuracy demand of 1% can only be met by a large number of replications (between 26 and 41 for immersion methods). Compared to other methods the buoyancy method without

Table 3.3.7. Particle density determinations of briquettes: relative (*r*%) and absolute (*r*) repeatability (mean value through laboratories) and relative (*R*%) and absolute (*R*) reproducibility.

Methods	*R* (g/cm³)	*r* (%)	*R* (g/cm³)	*R* (%)	*n*			
					0.5%	1%	2%	5%
Stereometric measurements 1	0.08	6.75	0.11	10.17	257	64	16	3
Stereometric measurements 2	0.08	7.46	0.11	9.56	300	75	19	3
Hydrostatic with paraffin coating	0.05	3.92	0.063	5.39	165	41	10	1
Buoyancy with paraffin coating	0.05	3.99	0.069	5.91	141	35	9	1
Buoyancy without paraffin coating	0.05	4.37	0.061	5.24	105	26	7	1

The required numbers of replications (n) are given for certain accuracy levels.

paraffin coating (BW) is associated with the lowest required number of replications. For example, to guarantee an accuracy level of 2%, seven replications are needed, while 19 are necessary for the stereometry 2 method. However, when variability is lower, e.g. for briquette type B3, the required number of replications can be much lower.

Then, for example, only four replications would be enough to obtain a precision of 0.5%, when using buoyancy pure water method on briquette type B3.

3.3.4.3. Correction of coating volume

In average the mass share of the paraffin coating was only 1.8% of the total sample weight. Compared to pellets, the variations between briquette types and laboratories are relatively small with values ranging from 1.2% to 2.7%.

As already explained for pellets, the corrected densities are always higher than Nc densities (Table 3.3.8). For each type of briquette, the mean values of corrected and Nc densities were statistically different (ANOVA one-way analysis, factor: correction). A *t*-test (α = 0.05%) showed that the measuring deviations are equal for both methods of calculation (similar coefficients of variation).

Concerning the values of repeatability and reproducibility calculated for Nc and corrected densities, it appears that the introduction of a correction for the volume of applied paraffin does not have a significant influence. This is due to the relatively small volume of the paraffin compared to the total briquette volume.

3.3.5. Conclusions

3.3.5.1. Particle density determination of pellets

For all methods tested, it appears that the pellet type, linked to the level of particle density, influences the variability of the results.

Generally, the stereometric method is less favourable due to higher variability compared to the liquid displacement methods.

Buoyancy and hydrostatic methods (no matter whether paraffin coatings or wetting agents are applied) produce similar results. For practical application, both methods can be regarded as compatible. Within the group of the liquid displacement methods, considering all pellet types, the buoyancy method using non-coated samples and wetting agent mixed with water performed better in terms of repeatability and reproducibility. This

Table 3.3.8. Mean values, standard deviation (Std) and coefficient of variation (CV%) for the non-corrected (Nc) density and corrected density (Corr1) for hydrostatic and buoyancy methods for the particle density of briquettes.

Briquette code		Hydrostatic method (g/cm³)		Buoyancy method (g/cm³)	
		Nc	Corr1	Nc	Corr1
B5	Mean	1.165	1.193	1.180	1.205
	Std	0.033	0.032	0.042	0.040
	CV%	2.8	2.7	3.5	3.3
B4	Mean	1.108	1.129	1.110	1.128
	Std	0.018	0.017	0.017	0.018
	CV%	1.6	1.5	1.5	1.6
B1	Mean	1.198	1.237	1.196	1.235
	Std	0.019	0.021	0.015	0.014
	CV%	1.6	1.7	1.3	1.1
B2	Mean	1.235	1.263	1.239	1.267
	Std	0.022	0.020	0.023	0.021
	CV%	1.8	1.6	1.8	1.6
B3	Mean	1.009	1.027	1.008	1.026
	Std	0.011	0.012	0.009	0.011
	CV%	1.1	1.2	0.9	1.1

method also needs fewer replications to fulfil a given accuracy level.

The tested solid displacement method also performed well in terms of repeatability particularly for wood pellets, but as this method was applied by only one of the laboratories, further confirmation is required.

If coating of pellets by paraffin is applied before submersion in liquid, a correction for paraffin penetrating the sample is required. A simple assumption of all paraffin remaining outside of the pellet is unacceptable. The use of differentiated correction factors derived from measurements with the solid displacement method (powder pycnometer) is possible. However, due to the increased efforts for the paraffin treatment without any improved precision compared to the uncoated samples, this procedure seems less favourable.

3.3.5.2. Particle density determination of briquettes

Similar to the observations with pellets, the accuracy and precision of the measurements for briquettes depend on the briquette type too, and the stereomettric method is again less favourable compared to the liquid displacement methods. Both stereometric methods lead to higher variability, higher values of repeatability and reproducibility and need more replications to reach a given accuracy.

For both, accuracy and practical reasons, the liquid displacement methods seem suitable to become a standard laboratory. For the buoyancy method a balance is required which allows weighing by the use of a bottom hitch to connect the hanging sample material. If no paraffin coating is applied the sample preparation time is low but the water has to be changed often due to briquette disintegration (sometimes after each replication). However, as the coating of the sample surface does not reduce the variability of the results, the efforts of paraffin coating can be abandoned.

For both, pellets and briquettes, it can be stated that an accuracy level better than 1% can hardly be achieved, for the methods tested. Indeed, over this limit, the number

of repetitions needed is far too high for field measurement.

Acknowledgements

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3.4. Comparative study of durability test methods for pellets and briquettes

Different methods for the determination of the mechanical durability (DU) of pellets and briquettes were compared by international round robin tests including different laboratories. The DUs of five briquette and 26 pellet types were determined. For briquettes, different rotation numbers of a prototype tumbler and a calculated DU index are compared. For pellets testing, the study compares two standard methods, a tumbling device according to ASAE S269.4, the Lignotester according to ÖNORM M 7135 and a second tumbling method with a prototype tumbler. For the tested methods, the repeatability, the reproducibility and the required minimum number of replications to achieve given accuracy levels were calculated. Additionally, this study evaluates the relation between DU and particle density.

The results show for both pellets and briquettes, that the measured DU values and their variability are influenced by the applied method. Moreover, the variability of the results depend on the biofuel itself. For briquettes of DU above 90%, five replications lead to an accuracy of 2%, while 39 replications are needed to achieve an accuracy of 10%, when briquettes of DU below 90% are tested. For pellets, the tumbling device described by the ASAE standard allows to reach acceptable accuracy levels (1%) with a limited number of replications. Finally, for the tested pellets and briquettes no relation between DU and particle density was found.
Keywords: Pellets; Briquettes; Durability; Biofuel quality; Lignotester; Particle density.

Étude comparative des méthodes de test de la durabilité des pellets et des briquettes. Différentes méthodes de détermination de la durabilité mécanique (DU) des pellets et des briquettes ont été comparées lors d'un test inter-laboratoire auquel ont participé plusieurs laboratoires. Les durabilités de 5 types de briquettes et de 26 types de pellets ont été déterminées. Pour les briquettes, la comparaison porte sur différents nombres de rotations à effectuer dans un tambour prototype et le calcul d'un index de Durabilité. Concernant les pellets, deux méthodes normalisées, un appareil tournant décrit par la norme ASAE S269.4 et le Lignotester décrit par la norme ÖNORM 7135, sont comparées à une autre méthode basée sur l'utilisation d'un prototype de tambour tournant. Pour les méthodes testées, la répétabilité, la reproductibilité et le nombre minimum de répétitions pour atteindre des niveaux de précision donnés ont été calculés. De plus, cette étude évalue la relation entre durabilité et masse volumique nette.

Les résultats montrent, tant pour les pellets que pour les briquettes, que les valeurs de durabilité mesurées et leur variabilité sont influencées par la méthode appliquée. De plus, la variabilité du résultat dépend du biocombustible lui-même. Pour les briquettes d'une durabilité supérieure à 90 %, 5 répétitions conduisent à une précision de 2 %, alors que 39 répétitions sont nécessaires pour atteindre une précision de 10 % lorsque des briquettes d'une durabilité inférieure à 90 % sont testées. Pour les pellets, l'appareil tournant décrit par la norme ASAE permet d'atteindre des niveaux de précision acceptables (1 %) avec un nombre limité de répétitions. Enfin, pour les types de pellets et de briquettes testées, aucune relation entre durabilité et masse volumique nette n'a pu être mise en évidence.

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3.4.1. Introduction

Durability (DU) and particle density are the main parameters describing the physical quality of densified solid biofuels like pellets and briquettes. Both fuel types are susceptible to mechanical wear, which leads to production of fine particles or dust during transport, transhipment and storage. Dust emissions are not only an inconvenience for the consumer, they are also a health hazard [1]. Additionally, fine particles and dust can disturb feeding systems of boilers and may lead to inhomogeneous combustion processes. Finally, dust may contribute to fire and explosion risks during handling, storage and transhipment [2].

Mechanical DU is a quality parameter that is defined as the ability of densified biofuels to remain intact when handled [3]. It is measured by the resistance of densified fuels towards shock or/and friction. Therefore, DU is an important quality parameter with regard to handling and transportation processes of briquettes and pellets. Particle density is another parameter, which is commonly taken as a measure of DU, e.g. high particle density leads to a high DU. However, as shown by Obenberger and Thek [4], this assumption is not valid.

The presented research, realised within the European project BioNorm, aims at providing a knowledge basis to the Technical Committee TC335 at CEN ("Standards for solid biofuels"). Its main goal is therefore to identify and to evaluate the best appropriate methods for the DU determination of pellets and briquettes. Already described methods and existing national standards serve as basis.

3.4.2. Materials and methods

3.4.2.1. Sample material

The briquettes selected for the round robin trials are made from wood and are commercially available in their country of origin. The briquette selection includes two briquette types produced by extruder press (B1, B2), one was produced by a chamber press (B3) and two by piston presses (B4, B5) (Table 3.4.1). As high moisture contents (MC) may influence the DU result [5], this parameter was measured prior to the determinations in order to avoid fuel moisture contents of more than 10%.

The briquettes used in the samples were prepared by cutting at both ends to a length equivalent to two times the diameter. Depending on the laboratory, the cutting was performed by blade or band saw.

The DUs of two pellet selections (Table 3.4.2), were determined during these presented trials. The first selection included wood pellets (6 and 8 mm diameter) and agricultural residues pellets (straw, hay and Miscanthus) produced in Austria, Denmark,

Table 3.4.1. Description of the selected briquettes for the round robin trials.

Briquette code	Press type	Shape	Raw material	Country of origin
B1	Extruder		Mixed wood	Belgium
B2	Extruder		Hardwood	Germany
B3	Chamber		Mixed wood	Germany
B4	Piston		Mixed wood	Spain
B5	Piston		Softwood	Austria

Table 3.4.2. Description of the selected pellets.

Pellets selection 1				Pellets selection 2			
Pellets code	Diameter (mm)	Raw material	Country of origin	Pellets code	Diameter (mm)	Raw material	Country of origin
P1	6	Mixed wood	Belgium	P16	6	Mixed wood	Belgium
P2	6	Softwood	Belgium	P17	6	Softwood	Belgium
P3	6	Hardwood	Spain	P18	8	Mixed wood	Denmark
P4	6	Hardwood	Spain	P19	8	Mixed wood	Denmark
P5	8	Mixed wood	Denmark	P20	6	Mixed wood	Germany
P6	8	Mixed wood	Denmark	P21	10	Straw	Germany
P7	8	Mixed wood	Denmark	P22	6	Mixed wood	Austria
P8	6	Mixed wood	Germany	P23	6	Mixed wood	Austria
P9	6	Mixed wood	Germany	P24	6	Mixed wood	Austria
P10	9	Straw	Germany	P25	6	Mixed wood	Austria
P11	6	Miscanthus	Germany	P26	8	Spruce	Finland
P12	6	Softwood	Austria				
P13	6	Softwood	Austria				
P14	8	Hay	Austria				
P15	9	Straw	Austria				

Germany and Spain. The second selection included 10 wood and one straw pellets, all commercially available in their country of origin (Austria, Belgium, Denmark, Finland and Germany). The first pellets selection was used for method and fuel comparisons, while the second one was applied for a round robin test designed for comparing the results of the method described by ASAE S269.4 (Tumbler) and ÖNORM M 7135 (Pneumatic).

The moisture contents of the pellet samples were determined before testing. As shown by Obernberger and Thek [4], MC and DU are not correlated at moisture content levels below 10%. Only pellets with moisture content below 10% were included in the trials, in order to avoid the MC influencing the DU results.

3.4.2.2. Briquettes DU test

The briquette DU is estimated by using a dustproof rotating drum prototype (Fig. 3.4.1). The drum has an internal diameter and a 598 mm depth (volume 1681). It is equipped with a baffle (200 × 598) mm, perpendicular to the wall surface of the cylinder. In the tests, presented here the rotation speed was fixed at 21 rpm.

A test portion of 21 sample material, from which fine particles had been removed, was weighed to the nearest 0.1 g and placed in the drum for 105, 210, 315, 410 and 630 rotations. After each tested rotation number, the sample material was removed from the drum and screened mechanically

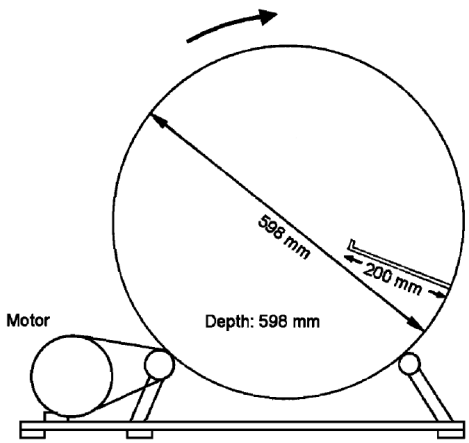

Fig. 3.4.1. Principle of the briquette durability tester.

or manually for 30 s using a 40 mm metal wire cloth according to ISO 3310-1 [6]. The particles remaining on the sieve (sieve oversizes) were weighed to the nearest 0.1 g. Both, sieve over- and undersizes were returned into the drum and the tumbling was continued until the subsequent tested rotation number was achieved. Tumbling, sieving and weighing procedures were continued until each sample was exposed to 630 rotations. The DU was calculated from the mass share of the sieve oversizes to the total initial mass. The results are given in percentage as the mean value of five replications.

Based on the DU result obtained for each rotating number, a DU curve was plotted with the number of rotations on the abscissa. From this curve a DU index was defined as the ratio, in percentage, between the area under the DU curve and the area related to a non-abraded material [5].

An international round robin was conducted with five briquette types (Table 3.4.1), which were shipped to five European laboratories. For the data analysis, each rotating number and the DU index were considered as separate methods.

3.4.2.3. Pellet DU test

Principles

The ASAE drum. The ASAE S269.4 standard [7] describes a tumbling device (Fig. 3.4.2) made of a rectangular container in aluminium or stainless steel with inner dimensions of (300 × 300 × 125) mm. In order to enforce the tumbling effect the box is equipped with a 230 mm long baffle, which extends 50 mm into the container. The baffle is affixed symmetrically to a diagonal of one side of the box. Rivets and screws are kept to a minimum and they are well rounded. The container rotates on an axis, which is centered perpendicular to the sides of the box. The rotation speed is fixed to 50 rpm.

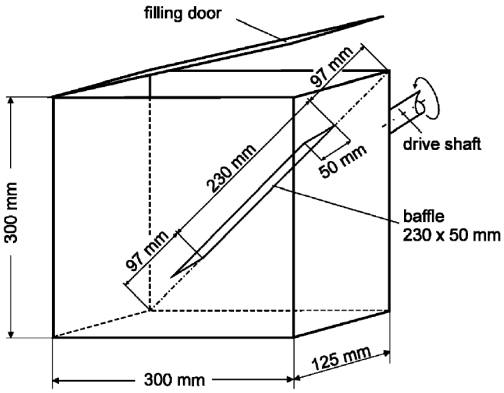

Fig. 3.4.2. ASAE S269.4 apparatus for durability testing of pellets.

In the trials described here, a 500 g sample was tumbled for 500 rotations before being sieved manually with a 3.15 mm round hole sieve according to ISO 3310.2 [8]. The DU is expressed as the percentage in mass of the pellets remaining on the sieve to the total sample weight. It is calculated as the mean value of three replications.

The lignotester. The ÖNORM M 7135 standard [9] refers to a commercial device (Ligno-Tester LT II of Borregaard Lignotech), which exposes the pellets sample to shocks inside a test chamber. The sample material is swirled by a defined air stream that induces the particles to collide against each other and the perforated walls of the test chamber. The test chamber has a four side pyramid form (walls consist of a 2 mm round hole sieve), which is orientated with the tip downwards (Fig. 3.4.3). The inside dimensions of the pyramid are (230 ± 5) mm at the base and (126 ± 10) mm in height. For the tests described here, the fines were removed, before testing, by sieving the sample manually with a 3.15 mm round holes sieve [8]. A (100 ± 0.1) g sample is placed in the test chamber before an air stream of 70 mbar was blown for 60 s into the test chamber. During the treatment the abraded fines were collected

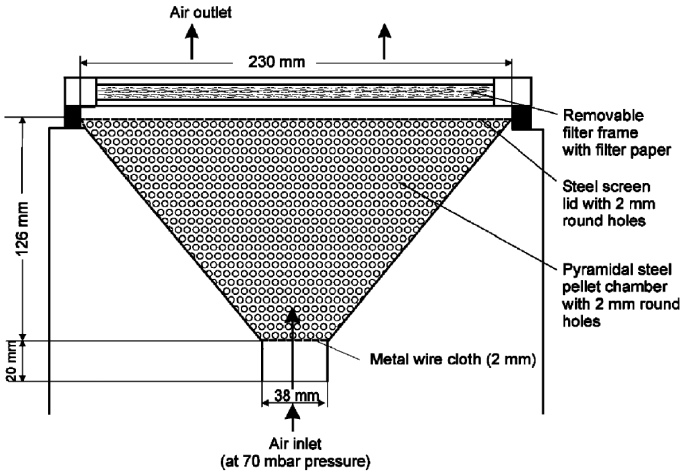

Fig. 3.4.3. ÖNORM M 7135 apparatus for durability testing of pellets.

as sieve undersize below the perforated test chamber. After the device has automatically switched off, the remaining pellets in the test chamber are removed and weighed. The DU is expressed as the percentage in mass of the pellets remaining in the chamber to the initial sample mass. It is calculated as the mean value of five replications.

The briquette drum. The procedure and the briquette DU tester are described in Section 2.2. For pellet testing, the 105 rotations period was chosen. Fines were separated by using a 3.15 mm round hole sieve [8]. The number of replications was set to 5.

Method testing

The tests performed on pellets were divided in two tests series related to two different pellet selections (Table 3.4.2).

First test series. The aim of the first test series was to compare results gained by the three selected methods. Trials were performed on the pellets of the first selection (Selection 1, Table 3.4.2). Two laboratories tested the tumbler described in ASAE S269.4 (in three replications); the Lignotester according to ÖNORM M7135 (in 20 replications) was tested by five laboratories and one laboratory used the briquette DU drum (five replications) for determinations on pellets. Besides the differences among the tested devices, the analysis also focussed on differences between pellet subgroups (agricultural residues pellets, 6 and 8 mm diameter wood pellets, high-DU pellets). The high-DU pellets correspond to the highest DU class (DU over 97.5%) according to CEN/TS 14961 [10]. Additionally, the measured DU values were compared to the pellet particle densities.

Second test series. The second test series was based on the results of the first series; an international round robin test was organised with four participating laboratories measuring 11 pellet types (Selection 2, Table 3.4.2). The numbers of replications were fixed to 5 and 10, respectively, for the ASAE S269.4 and the ÖNORM M7135 standards. This round robin focussed on the DU repeatability and reproducibility limits, and on the relation between the results of these two standard methods.

3.4.2.4. Repeatability, reproducibility and number of replications

The absolute and relative repeatability and reproducibility limits (when applicable) of the tested methods were calculated following ISO 5725.1 and 2 [11]. The required minimum number of replications to achieve a given accuracy level is calculated following a common statistical calculation procedure

according to Dagnelie, 1975, vol. 2, p. 30 [12].*

3.4.2.5. Particle density

Particle density is the ratio of the sample mass and its volume including pore volume. The volume of the selected pellets and briquettes was estimated using the buoyancy method in liquid. This method has been shown to have a low variability [13,14]. The method is based on the Archimedes principle; the pellet sample is weighed in air and in a liquid. By knowing the liquid density the volume of the sample can be calculated. For the determination of pellets, a commercially available density determination kit was applied while for briquette measurements a setup with a below-balance weighing hanger was used.

The pellet samples had a mass of 5-8 g, the briquettes were prepared to have a length equal to two times the diameter.

For pellet testing a wetting agent was added to the water in order to avoid bubbles formation and to allow the liquid to fill voids and pores that communicate with the surface of the pellets (*t*-Octylphenoxypolyethoxyethanol; polyethylene glycol tert-octylphenyl ether—CAS number: 9002-93-1—trade mark Triton X-100) at a concentration of 1.5 g/l, which leads to a liquid density of 0.996 kg/l. In laboratory conditions, the effect of the temperature on the liquid density was neglected.

Due to the fast disintegration of the briquette samples in the liquid when using wetting agent, the particle density of the selected briquettes was measured in pure water.

* Le principe de ce recueil de publications est de présenter les contributions scientifiques telles qu'elles ont été publiées par les revues scientifiques auxquelles elles ont été proposées. Cependant, en dépit du soin apporté par les auteurs et par les différents relecteurs, quelques légères erreurs subsistaient dans les versions publiées par les revues. Ce paragraphe a été corrigé par rapport à la version publiée.

3.4.3. Results and discussion

Fig. 3.4.4 shows the results (mean values of all laboratories and standard deviations) of the DU measurements, for the five briquette types tested during the round robin test. The standard deviation includes the variation within individual laboratories and between laboratories. As expected, the DU values decreased with an increasing number of rotations. The figure also shows that the individual briquette type influenced the variability (standard deviation) of the DU measurements. The standard deviation of the measurements increased with a larger number of drum rotations (except B4, for which destruction of the sample was observed).

The DU index gives a value close to the mean value calculated for all tested rotation numbers. Nevertheless, the variability of the DU index is higher than for the 105 rotation method, except for the briquette with the lowest DU (B4).

Table 3.4.3 shows, for each tested method, the absolute (r) and the relative (r%) values of the repeatability limits (mean value of the laboratories involved in the round robin), as well as the values of the reproducibility limits (absolute, R, and relative, R%). The required minimum numbers of replications in order to achieve a given accuracy level (2%, 5% and 10%) are also indicated.

For all tested methods the repeatability and reproducibility limits are high, when all briquette types are considered. Nevertheless, the 105 rotations period leads to the lowest relative repeatability value (15.0%) and the lowest relative reproducibility limit (40.9%). The comparatively low repeatability limit of the 105 rotations test period leads to a lower required number of replications to achieve a given accuracy level. For example, to secure an accuracy level of 10%, 39 replications shall be conducted, while a number of 966 replications should be necessary for the 630 rotations method.

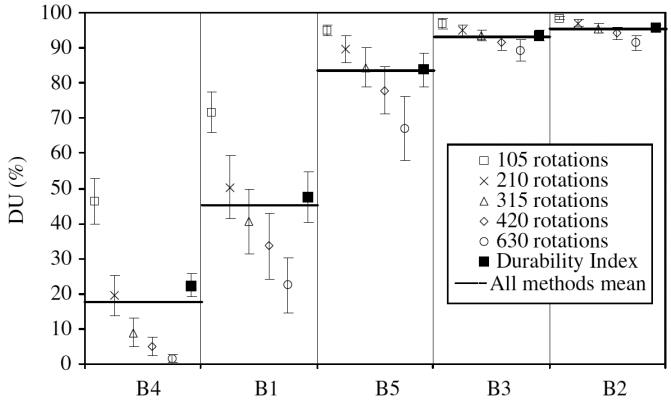

Fig. 3.4.4. Durability (DU) of five briquette types determined by five periods of drum rotations and by the durability index (mean values plus/minus standard deviations of five laboratories).

Table 3.4.3. Briquettes durability testing: relative and absolute repeatability (*r* and *r*%, mean of five laboratories) and reproducibility limits (*R* and *R*%); minimum required number of replications to achieve a given accuracy level (*N*) for the given numbers of rotations and the durability index.

Method	*r*[a]	*r*%[b]	*R*[a]	*R*%[b]	*N*		
					2%	5%	10%
105 rotations	12.3	15.0	33.4	40.9	974	156	39
210 rotations	15.4	21.9	43.9	62.6	6611	1058	264
315 rotations	15.9	24.6	40.5	62.7	14548	2328	582
420 rotations	16.8	27.9	38.4	63.5	18494	2959	740
630 rotations	18.3	33.6	37.1	68.2	24148	3864	966
Durability Index	12.9	18.8	33.8	49.3	2114	338	85

[a] Absolute values (%).
[b] Relative values (% of DU).

From a practical point of view, it seems that an accuracy level better than 10% can hardly be achieved for the DU estimation of briquettes by the tested trial setup. Indeed, even with the 105 rotations method, the time necessary to perform one single determination is relatively long (a 105 rotations test needs 5 min). Improvement of the method should be tested; in particular, higher rotation speeds than the 21 rpm applied here could shorten the required rotation times. Moreover, the high variability might be enhanced by larger individual sample volumes.

Nevertheless, for briquettes of DU above 90%, the accuracy of the method is improved. Table 3.4.4 shows, for the 105 rotations period, that absolute and relative repeatability limits of briquettes having a DU of 90% and above, are both below 5%, while they are higher for DUs below 90%. The same observation was made for the absolute and relative reproducibility limits, which are also improved for briquettes of a DU of 90% and above. Table 3.4.4 also shows that, for DUs of 90% and above, only one replication is needed to achieve 5% accuracy, while five replications secure an accuracy of 2%.

For the selected pellets (Selection 1, Table 3.4.2) the first test series had a DU range from 93.6% to 99.4%, when the tumbler according to ASAE S269.4 was applied. The same pellets determined by the Lignotester following ÖNORM M7135 showed a DU range from 91.2% to 99.3%. Tests performed using the briquette drum lead to smaller result range, from 97.6% to 99.8%.

Fig. 3.4.5 shows the mean values and the standard deviations for all laboratories of

Table 3.4.4. Briquettes durability testing: relative and absolute repeatability (*r* and *r*%, mean of five laboratories) and reproducibility limits (*R* and *R*%); minimum required number of replications to achieve a given accuracy level (*N*) for 105 rotations and briquettes above and below durability of 90%.

Briquette durability	105 rotations method						
	r[a]	*r*%[b]	*R*[a]	*R*%[b]	*N*		
					2%	5%	10%
DU < 90%*	18.8	31.8	52.6	89.2	974	156	39
DU ≥ 90%	3.9	4.1	4.6	4.7	5	1	1

[a] Absolute values (%).
[b] Relative values (% of DU).

the DU results on 15 pellet types and for the three tested methods. The standard deviation includes the variation within the individual laboratories and between the laboratories.

For both wood and agricultural residues pellets, the differences between results from ASAE and ÖNORM standards are larger for pellets below DU 97.5%. Furthermore, for this pellet group the Lignotester of the ÖNORM standard leads to lower DU values compared to the tumbler according to ASAE standard. For high-DU pellets (DU 97.5% or more), values gained by ASAE and ÖNORM are more similar, while the tumbler according to ASAE standard seems to measure slightly lower DU values. A test of variance equality performed on the three tested methods (Bartlett test, significance level $a = 0.05$) confirms that the individual pellet type highly influences the variability of the measurements. Moreover, the standard deviation observation shows that the DU factor level influences the variability: the lower the pellets DU is, the higher the variability is (Fig. 3.4.5). However, the comparison of the coefficient of variation (*T*-test, significance level $a = 0.05$) indicates that ASAE standard systematically leads to lower variability, compared to ÖNORM.

The variability of the briquette tester (only tested at one laboratory) is generally low compared to the two other methods. Nevertheless, the DU values of the different pellet types are relatively close together, thus a pellets differentiation or a classification according to this method can hardly be realised.

Table 3.4.5 shows, for ASAE and ÖNORM methods, the mean values of absolute (*r*) and relative (*r*%) repeatability limits (for laboratories involved in the method testing) and the mean values of absolute (*R*) and relative (*R*%) reproducibility limits. The table also indicates the number of replication needed to achieve a given accuracy level (0.5%, 1%, 2%). These parameters are calculated for 6 and 8 mm wood pellets, agricultural residues pellets, and for all pellets together. Additionally, these values are given for pellets having a DU of more than 97.5% (according to ASAE S269.4).

The absolute and relative repeatability values are, regarding all pellets, 0.9 for the ASAE standard and 1.8 for the briquette tester. For the ÖNORM standard "*r*" is 2.1 and "*r*%" is 2.2. Again the pellets DU influences the repeatability values. The lowest repeatability limits (both absolute and relative) were observed for high-DU pellets.

For the whole pellets selection, the reproducibility values (absolute and relative) are: 1.4 and 1.5 (*R* and *R*%, respectively) for the ASAE standard. The ÖNORM leads to higher

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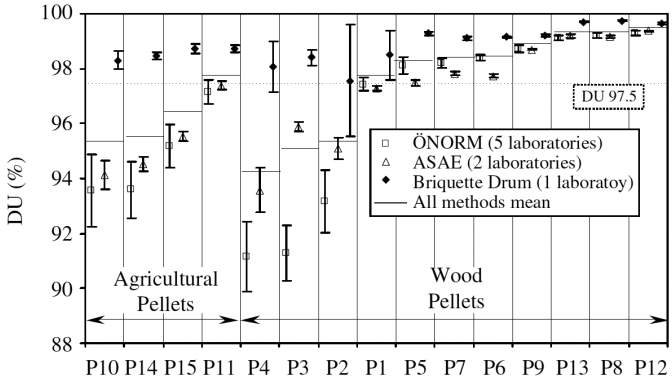

Fig. 3.4.5. Durability (DU) of 15 pellets determined by three different methods (mean values plus/minus standard deviations of involved laboratories).

variability between laboratories; the *R* value is 3.8 and the *R* % value is 4.0. As for repeatability, the tested fuels influence this parameter.

For all investigated subgroups of pellets, the ÖNORM standard leads to higher repeatability and reproducibility values (higher variability) than the ones obtained by following ASAE standard.

This is also reflected by the required minimum number of replications to achieve a given accuracy level (Table 3.4.5). The low result variability of the ASAE standard leads

Table 3.4.5. Pellets durability testing: relative and absolute repeatability and reproducibility limits for the tested methods.

Method	Involved labs	r^a	r %^{b}	R^a	R %^{b}	N		
						0.5%	1%	2%
All pellets								
ASAE	2	0.9	0.9	1.4	1.5	24	6	1
ÖNORM	5	2.1	2.2	3.8	4.0	83	22	5
Briquette drum	1	1.8	1.8			67	17	4
6 mm diameter								
ASAE	2	1.0	1.0	2.4	2.4	24	6	1
ÖNORM	5	2.1	2.2	3.8	4.0	83	22	5
Briquette drum	1	2.4	2.4			67	17	4
8 mm diameter								
ASAE	2	0.2	0.3	1.4	1.4	1	1	1
ÖNORM	5	0.6	0.6	2.9	3.0	3	1	1
Briquette drum	1	0.2	0.2			1	1	1
Agricultural residues pellets								
ASAE	2	0.9	1.0	2.1	2.2	7	3	1
ÖNORM	5	2.8	2.9	7.1	7.5	49	12	3
Briquette drum	1	0.6	0.6			2	1	1
Wood pellets DU> 97.5								
ASAE	2	0.1	0.1	0.4	0.4	1	1	1
ÖNORM	5	0.3	0.3	1.0	1.0	3	1	1
Briquette drum	1	0.1	0.1			1	1	1

The minimum required number of replications for a given accuracy level are also given.
[a] Absolute values (%).
[b] Relative values (% of DU).

to a smaller amount of required replications. For example, to secure an accuracy level of 1%, only six replications are needed when all selected pellets are regarded, while 22 are required for the ÖNORM standard. Table 3.4.5 also reveals that the demanded five replications of the ÖNORM standard lead to an accuracy level of 2%.

It also appears that accuracy levels below 0.5% can only be achieved for high-density pellets, at least with practicable numbers of replications. Indeed, even using the ASAE method 24 replications are needed considering all pellet types.

Nevertheless, the data presented here were calculated for a great variation of pellet types. For other pellets the required number of replications can be far smaller, e.g. only six replications are required to obtain a precision of 0.1%, when ASAE method is used on pellets having a DU above 97.5%.

The test series on the second pellets selection (Selection 2, Table 3.4.2) leads to DU ranges from 95.6% to 99.4%, estimated by the tumbler following ASAE S269.4. The same pellets measured by the Lignotester according to ÖNORM lead to DU values between 92.7% and 99.4%. Fig. 3.4.6 shows the mean values for DU and the standard deviation for 11 pellet types tested by all laboratories participating in the round robin. It appears that nine of the selected pellets have a DU (estimated by ASAE S269.4) above 97.5% and thus belong to the highest pellets quality class according to [12].

As expected from the first test series, an ANOVA (significance level a = 0.05) confirms that results gained by ASAE and ÖNORM are significantly different. A T-test (significance level a = 0.05) comparing coefficient of variation of the two methods indicates the higher variability of the ÖNORM measurements.

A linear regression analysis (significance level a = 0.05) was conducted with results given by the tumbler according to ASAE and the Lignotester according to ÖNORM (Fig. 3.4.7). When all selected pellets are considered, the coefficient of determination R^2 for the regression line is 0.69. When only pellets with a DU of more than 96% are regarded, the coefficient of determination is lower (R^2 = 0.47). The low coefficients of determination indicate that it is hazardous to extrapolate results from one method to the other.

The calculation of the repeatability and the reproducibility limits of the ASAE and ÖNORM methods confirm the results obtained in the first test series (Table 3.4.6). Also, here both parameters are lower for the ASAE S269.4 than for the ÖNORM M 7135.

Fig. 3.4.8 shows DU values of 15 tested pellets (Selection 1, Table 3.4.2) which were determined by the ASAE tumbler, they are compared to their respective particle densities. It appears that no clear relation can

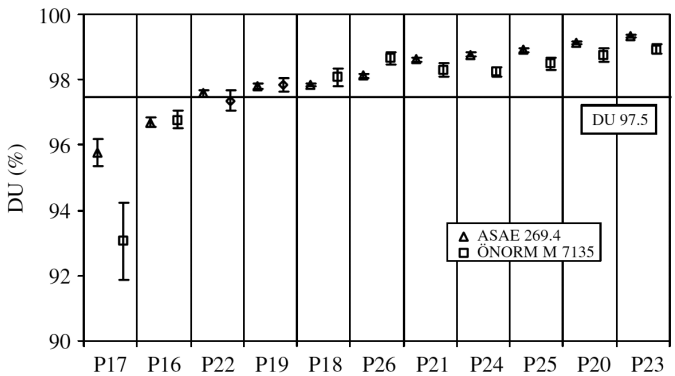

Fig. 3.4.6. Durability (DU) of 11 pellets estimated by ASAE S269.4 and ÖNORM M 7135 (mean value and standard deviation of four laboratories).

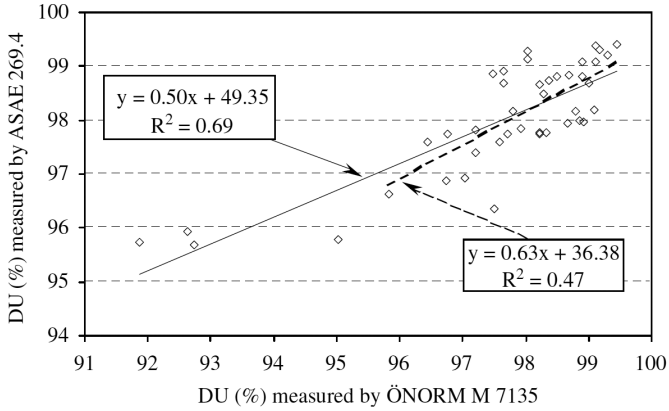

Fig. 3.4.7. Relation between ÖNORM M 7135 and ASAE S269.4 results for durability (DU) of pellets.

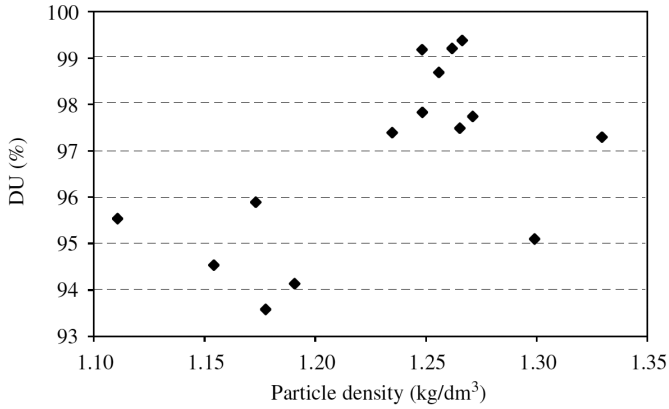

Fig. 3.4.8. Relation between durability (ASAE S269.4) and particle density for the 15 tested pellets (R^2 = 0.33).

Table 3.4.6. Pellets durability testing absolute and relative repeatability and reproducibility.

Method	r^a	$r\%^b$	R^a	$R\%^b$
ASAE	0.6	0.6	0.7	0.7
ÖNORM	1.0	1.1	3.0	3.0

[a]Absolute values.
[b]Relative values.

be drawn between these two parameters. A linear regression analysis (significance level α = 0.05) revealed a coefficient of determination, R^2, of 0.33. In contradiction to the statement of several pellet producers, this confirms observations made by Obernberger and Thek [4]. For pellets made from different raw material, produced by different equipment and under variable conditions, there is no relation between DU and particle density of pellets. The correlation between briquette DU and particle density has also been investigated, for the five selected briquette types. But, also for briquettes, no relation between these two properties was found.

3.4.4. Conclusions

The results from the briquette testing support the conclusion that briquette DU is best determined by tumbling in a drum for 105 rotations. For this treatment, the lowest repeatability and reproducibility limits were given. Nevertheless, the briquette DU testing is associated with a relatively high variation of the results, particularly when considering the full range of possible briquette types. With the equipment applied here, accura-

cies below 10% can hardly be achieved in practice. However, for briquettes having a DU of 90% and higher, an accuracy level of 2% can be achieved by five replications. The applicability for proving any conformity with relatively high briquette quality is given by the method investigated here.

For pellet testing, the tumbling device described by the ASAE S 269.4 gives the most repeatable and reproducible results. Moreover, this method requires the least number of replications to achieve a given accuracy level, while measurements with the pneumatic device following ÖNORM M 7135 imply a higher number of replications. In practice, an accuracy level of 0.5% can be reached when the tumbling device according to ASAE is applied, this is particularly true for high DU pellets.

The variation of DU measurements for briquettes and pellets is highly influenced by the fuel type and the fuel properties. For most individual fuel types the required minimum number of replications to achieve a given accuracy level is far smaller than for a collection of pellets or briquettes.

There is no clear correlation between the DU results of pellets measured by the tumbling device and those given by the Lignotester. Generally comparisons between the two methods are thus not advisable. In the same way, useful correlations between DU and particle density could not be observed, neither for briquettes nor for pellets.

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3.5. Internal particle size distribution of biofuel pellets

Several methods for disintegration of biofuel pellets were tested and compared for their ability to break up the pellets into the original particles of the raw material. Analyses performed on softwood pellets and straw pellets concluded that wet disintegration in water at ambient temperature is insufficient for a determination of the internal particle size distribution of wood- and straw pellets. When the wet disintegration was performed with water heated to the boiling point and coupled with mechanical disintegration in terms of stirring a more complete disintegration of the pellets was obtained. Based on the results obtained in the initial study a round robin was set up including six European laboratories where the selected method was tested. In the round robin test the method combining heated water and stirring of the slurry was tested on solid biofuel pellets produced of comminute straw, deciduous wood and coniferous wood respectively. With the method a satisfactory disintegration was obtained of all three types of pellets.

Further wet disintegration of coniferous pellets was compared to a dry disintegration using a hammer mill. The dry disintegration of the coniferous pellets resulted in smaller particle sizes compared to the wet disintegration using heated water and stirring of the slurry indicating a further disintegration of the original particles in the hammer mill process.

Overall the wet disintegration combined with mechanical impact was found to be the most suitable method for disintegration of solid biofuel pellets. Combined with sieving analysis the method gives realistic image of the internal particle size distribution of solid biofuel pellets.
Keywords: Wood pellets; Straw pellets; Particle size distribution; Solid biofuel; Quality characterisation.

Distribution granulométrique des particules constituant les pellets. Plusieurs méthodes ont été testées et comparées pour désagréger des pellets de biocombustibles, afin de déterminer leur capacité à séparer les particules de matière première qui les constituent. Des analyses réalisées sur des pellets de bois résineux et de paille permettent de conclure que la désagrégation dans de l'eau à température ambiante est insuffisante pour déterminer la distribution granulométrique des particules constituant les pellets de bois résineux et de paille. Lorsque cette désagrégation est réalisée à l'aide d'eau chauffée à une température proche du point d'ébullition et qu'elle est couplée à une désagrégation mécanique par agitation, une désagrégation plus complète des pellets est obtenue. Sur base des résultats obtenus lors de l'étude initiale, un test inter-laboratoire a été mis sur pied au cours duquel six laboratoires ont testé la méthode sélectionnée. Durant le test inter-laboratoire, la méthode combinant eau chauffée et agitation de la suspension a été testée sur des pellets de biocombustibles solides produits à partir de paille broyée, de bois de résineux et de bois feuillus. Avec cette méthode, une désagrégation satisfaisante a été obtenue pour les trois types de pellets.

Ensuite, la désagrégation par voie humide de pellets de résineux a été comparée à la désagrégation à sec en utilisant un broyeur à marteaux. La désagrégation à sec des pellets de conifère conduit à des particules plus petites comparées à celles obtenues par la désagrégation utilisant de l'eau chauffée et une agitation de la suspension, ce qui indique une désagrégation plus poussée des particules d'origine dans le traitement par broyeur à marteaux.

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Globalement, la désagrégation par voie humide, combinée à une agitation mécanique s'est révélée la méthode la plus adaptée pour désagréger des pellets de biocombustibles solides. Combinée à une analyse par tamisage, la méthode donne une image réaliste de la distribution granulométrique des particules constituant les pellets de biocombustibles solides.

3.5.1. Introduction

The growing focus on reduction of greenhouse gases emissions, especially carbon dioxide has led an increasing part of electricity producers to convert coal pulverized systems into co-combustion using solid biofuel in the form of comminuted wood- or straw pellets [1]. Co-combustion of biomass and coal are identified as a short term, low risk and sustainable way for reducing emissions of CO_2, SO_x and NO_x [3]. Advantages of co-combustion are several; environmental in terms of substitution of fossil fuels with solid biofuel, technical as boiler efficiency is improved and economical as the fuel costs are reduced [1]. By that, substitution of a part of the pulverized coal by comminute pellets is an excellent possibility for long term sustainable CO_2 reduction [8].

The growing consumption of pellets, both for direct combustion and for suspension combustion have increased the import of pellets from outside Europe. Considering Europe with the formal 15 countries, the use of imported biomass for electricity production could reduce by about 20% the costs related to the achievement of renewable energy use goals [5-7]. In consequence, the transport of solid biofuel pellets appears profitable both on an economical and environmental point of view, even if milling is necessary before combustion in pulverized systems. The milling shall bring the pellets into particles having the sufficient aerodynamic properties. For pulverized systems the particle size distribution of the fuel is a characteristic of main importance as it influences the fuel feeding, the ignition, the amount of unburned particles and the furnace temperature [4,9,10].

The recommended particle size distribution of the fuel for co-combustion varies, Baxter recommend that all particles shall be below 6.34 mm with most of the particles having a size of 3 mm [3]. Even that the specifications of some consumers are more strict [2] the influence of the maximum biomass particle size in co-combustion is not well defined [11,12]. The shape and size distribution of the particles are depending on the milling parameters (hammer or knives, rotation speed, type and diameter of the grids) and on the raw material characteristics. Moreover, the hammer mill design depends on the quality of the pellets as the internal particle size distribution and the desired particle size after milling [18].

A method based on a wet disintegration of the pellets is commonly used for describing the internal particle size distribution of the pellets. Assuming that the internal particle size distribution of the pellets reflects the particle size distribution of the comminute pellets the method is used to estimate the behaviour of the comminute product in a suspension boiler.

The present study investigates a number of methods to describe the internal particle size distribution of solid biofuel pellets. The methods are based on either wet-disintegrations of the pellets in water and subsequent drying and sieving of the particles or on a dry disintegration by crushing the pellets in a laboratory scale hammer mill and subsequent sieving of the particles. The obtained particle size distributions from the wet-disintegration methods are compared to the particle size distribution obtained with the dry disintegration. For the present used wet disintegration

method an initial study was carried out to investigate whether comparable results were obtainable between laboratories and moreover to see whether the method could be improved. The initial study gave rise to changes in the method and a round robin test with six participating laboratories was subsequently conducted for this revised method. The aim of the round robin test was to verify the revised method and to determine the variation in terms of repeatability and reproducibility in the obtained results.

3.5.2. Materials and methods

3.5.2.1. Test material

All test material origin from commercially produced biofuel pellets acquired from the Danish energy company 'DONG energy'. The pellets had a diameter of 8 mm and a maximum length of approximately 40 mm, the straw pellets being slightly shorter than the wood pellets. For the initial study of the wet disintegration method two types of solid biofuel pellet was chosen. One type was produced of coniferous wood and one type produced of comminuted straw. The dry disintegration method was tested on coniferous wood pellets from the same lot used for the wet disintegration study. For the round robin test three types of solid biofuel was chosen. Beside coniferous wood and straw pellets similar to the types used in the initial study a second type of wood pellets produced of deciduous wood was included in the round robin test. For all tests, samples of 50 kg was acquired and subsequently divided into test portions of 300 g with a riffle divider (18 splits with split size of 28.2 mm).

3.5.2.2. Initial wet disintegration study

Three laboratories participated in the initial study of the existing method which consists of a wet disintegration of the pellets and subsequently drying and determination of the particle size distribution of the obtained particles. The disintegration was performed by placing a test portion of 300 ± 10 g pellets and 1200 ± 10 ml demineralised water at ambient temperature in a watertight container with a volume of approximately 2000 ml for 24 h for disintegration. After the disintegration the test portion was transferred to a drying container. To secure that all material was transferred the disintegration container was rinsed with demineralised water into the drying container. The material was dried at 35 ± 5 °C in 24 h and subsequently left for 24 h to equilibrate with the ambient temperature and humidity in the laboratory.

Beside the disintegration with demineralised water at ambient temperature additional tests was performed, using demineralised water heated to boiling point and using mechanical impact as stirring with a spoon respectively to facilitate the disintegration. All types of disintegrations were performed in duplicates.

After the equilibration the test portion was divided into two portions each of 150 ± 10 g by means of a riffle divider. One portion was used for moisture determination [13] and one portion was further divided into two test portions of 75 ± 5 g for particle size determination.

Moisture determination was performed by drying the moisture test portion in a drying cabinet at 105 °C for 24 h [13]. The moisture content of the equilibrated material shall be between 5% and 15% in order to avoid that the particles become brittle or agglomerates.

Duplicate particle size analysis were performed with a Retsch AS 200 control 'g' sieving machine using eight Ø200 mm sieves with an aperture size of (mm) 3.15 (round holes [14]), 2.8, 2.0, 1.4, 1.0, 0.5, 0.25 and 0.125 (mesh wire [15]). For wood pellets the sieving analysis was performed on the full

portion of 75 ± 5 g whereas the straw pellets was divided into two approximately equal portions and sieved in two rounds to avoid clogging of the wire gauze. The sieving time was set to 30 min using amplitude of 3 mm which secure that the sieving is complete i.e. the mass change in the sieves between two consecutive sievings is below 0.2% [16].

3.5.2.3. Dry disintegration study

The dry disintegration was performed in duplicates using a laboratory hammer mill fitted with six equidistantly pivoting hammers. The rotation axis are mounted on a 10 mm diameter steel disc with the pivoting axe centre located 12.5 mm from the edge of the disc, on the diameter line. The hammers are T shaped, with a square section of 5 mm side; the pivoting part is 37.5 mm long from the centre of the axis to the perpendicular part. The milling chamber has a internal diameter of 190 mm and a 55 mm width. A 160 mm internal diameter and 55 mm width grid perforated by 3 mm round holes is centred in the milling chamber. The rotation speed of the engine axe is 2800 rpm, and the engine has a power of 1.1 kW.

The particle size distribution of the dry disintegrated pellets was determined as described in Section 3.5.2.2 but using seven Ø200 mm sieves with an aperture size in mm of 3.15 (round holes [13]), 2.8, 2.0, 1.4, 1.0, 0.5 and 0.25 (mesh wire [14]). The choice of sieves reflects the recommended selection from CEN/TS 15149-2 [17].

3.5.2.4. Wet disintegration round robin test

Six laboratories participated in the round robin test. The method used was based on the experience from the initial study. The disintegration was performed by placing a test portion of 300 ± 10 g pellets and 2000 ±10 ml demineralised water heated to a temperature of 95 ± 3 °C in a container with a volume of approximately 5000 ml and carefully mixing the pellets and water to a slurry by stirring with a spoon. The slurry was left for 24 h where after it was transferred to a drying container. To secure that all material was transferred the disintegration container was rinsed with demineralised water into the drying container. The material was dried for 24 h at 60 ± 5 °C in 24 h and subsequently left in the laboratory for 48 h to equilibrate with the ambient temperature and humidity in the laboratory. All disintegrations were performed in triplicates.

After the equilibration the test portion was divided into two portions each of 150 ± 10 g by means of a riffle divider. One portion was used for moisture determination as described in Section 3.5.2.2 and one portion was further divided into two test portions of 75 ± 5 g for particle size determination.

Particle size analyses were performed using the sieving equipment and procedure described in Section 3.5.2.3. For the two types of wood pellets the sieving analysis was performed on the full portion of 75 ± 5 g whereas the straw pellets was divided into two approximately equal portions and sieved in two rounds to avoid clogging of the wire gauze.

3.5.2.5. Statistics

For comparisons of the results from the round robin test (3.5.2.4) are the 25%-, 50%- and 75% quantiles calculated. The variation in the particle size distribution obtained within and between the laboratories is calculated as the standard deviation:

$$SD = \sqrt{\frac{(x - \bar{x})^2}{(n - 1)}} \qquad (1)$$

where x is the value, $\bar{x}$ the mean value, n is the number of values.

All data, which cannot be rejected due to procedure errors, are applied in the calculation of the standard deviations. Thereby no

a priori identification of outliers is employed since any variation between the distributions is assumed to be within the range that is achievable with the method.

Differences between the calculated quantiles from each laboratory are identified using a one-way ANOVA with following model:

$$Quantile = \alpha_0 + \beta_1\ value + \varepsilon \qquad (2)$$

where α_0 and β_1 are the parameters for fixed effects, ε the $\sim N(0, \sigma_2)$ and *value* the mean of the three replicate measurements of the particle size for the specific quantile.

Standard deviation of repeatability (S_r) and standards deviation of reproducibility (S_R) of the quantiles are calculated with the Mean Square from the one-way ANOVA where:

$$S_r = \sqrt{MS_{withingroups}} \qquad (3)$$

The repeatability is calculated under test conditions where independent analysis is obtained using the same method, equipment and operator in the same laboratory on identical samples. The repeatability limit or repeatability (r) is the maximum value under which is situated, with a 95% probability, the absolute value of the difference between two results obtained in conditions of repeatability.

$$S_r = \sqrt{MS_{betweengroups}} \qquad (4)$$

The reproducibility is calculated under test conditions where independent analyses are obtained using the similar method and equipment in different laboratories on identical samples. The reproducibility limit or reproducibility (R) is the maximum value under which is situated, with a 95% probability, the absolute value of the difference between two results obtained in conditions of reproducibility.

3.5.3. Results and discussion

3.5.3.1. Initial wet disintegration study

Solid biofuel pellets produced of coniferous wood and straw were tested at three different laboratories using the existing method and modifications hereof. The obtained cumulative particle size distributions indicate that there is a considerable influence from the applied disintegration method on the distribution (Figs. 3.5.1 and 3.5.2). For the wood pellets it is evident that using demineralised water at room temperature result in an insufficient disintegration of the pellets. Water at room temperature is prescribed in the existing method. Applying heated water and mechanical impact through stirring of the slurry improve in various degrees the disintegration of wood pellets. However, there is some overlap in the particle size distribution curves between the three methods i.e. water at room temperature, heated water and water at room temperature combined with stirring of the slurry (large circle in Fig. 3.5.1). Combining the two effects, heated water and stirring of the slurry, resulted in a more complete disintegration which is seen as the thick solid distribution curves which are separated in the upper part of Fig. 3.5.1 (HW + STG, the small circle).

The disintegration of the straw pellets was much more complicated and some of the experiments were abandoned. In some of the experiments the material absorbed all of the 1200 ml water and in several of the experiments was found agglomerated particles in the material for the sieve analysis. Compared to the tests using water at ambient temperature (Fig. 3.5.2, W), the tests applying heated water (HW) improved the disintegration. Opposite the tests using water at ambient temperature and stirring (W + Stg) only improved the disintegration in one of the five performed tests. Finally the one test applying both heated water and stirring (HW + Stg)

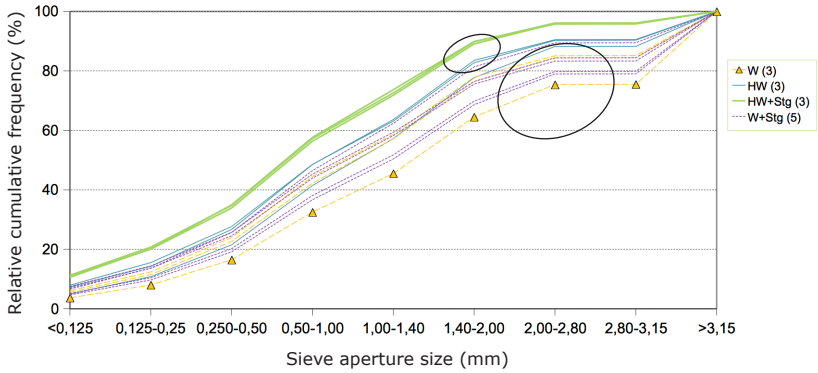

Fig. 3.5.1. Particle size distribution of wood pellets disintegrated with water at ambient temperature (W), heated water (HW), heated water combined with stirring (HW + Stg) and water at ambient temperature combined with stirring (W + Stg). Values in brackets in inserts indicate the number of replicate of the specific combination of treatment.

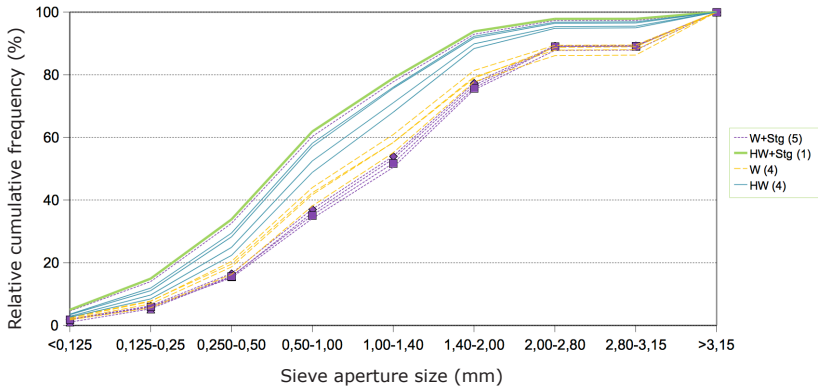

Fig. 3.5.2. Particle size distribution of straw pellets disintegrated with water at ambient temperature (W), heated water (HW), heated water combined with stirring (HW + Stg) and water at ambient temperature combined with stirring (W + Stg). Values in brackets in inserts indicate the numer of replicate of the specific combination of treatment.

showed the best disintegration compared to all other combinations (Fig. 3.5.2). By that it was decided to proceed with a round robin test using the method where the two effects, heated water and stirring, are combined.

3.5.3.2. Dry disintegration study

Solid biofuel pellets from coniferous wood was disintegrated in a hammer mill as described in Section 3.5.2.3 at one laboratory. The test was performed on three samples in triplicates and the obtained particle size distributions show smaller particle sizes for the samples disintegrated with a hammer mill compared to the particle size distributions for wet disintegrated pellets (Fig. 3.5.3). The difference is a consequence of both the aperture size of the sieve applied to the hammer mill and the hammer mill's further disintegration of the particles into even smaller particles. Applying other aperture sizes would result in particle size distribution that would differ from the present. As a mean for estimating the particle size distribution of the pellet powder after the hammer mill at a specific

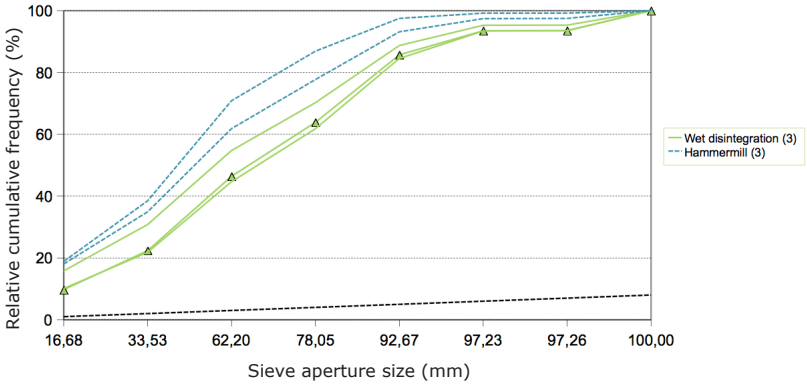

Fig. 3.5.3. Particle size distribution of wood pellets disintegrated in heated water combined with stirring and wood pellets disintegrated. In a hammermill. Values in brackets in inserts indicate the numer of replicate of the specific combination of treatment.

production facility a method using dry disintegration in laboratory scale hammer mills could be considered. The laboratory hammer mill should then be fitted with sieves having an aperture size corresponding to the full scale hammer mill. However, the obtained results will then only be valid for the specific laboratory and full scale hammer mill and therefore not applicable as a standard test method for inter-laboratory comparisons.

3.5.3.3. Wet disintegration round robin test

Solid biofuel pellets produced of coniferous and deciduous wood and comminuted straw were tested in a round robin test at six different laboratories. Except for one laboratory the obtained particle size distributions of the disintegrated deciduous wood pellets showed very little variation (Fig. 3.5.4). The reason to the large deviation for the one laboratory is assumed to be use of too small amplitude in the sieving operation compared to prescribed amplitude of 3 mm. The amplitude is identified to have a significant influence on the result of a sieving operation [16] and the deviating result was excluded in the calculation of repeatability and reproducibility. As with the coniferous wood pellets, the particle size distribution of the deciduous wood pellets was very similar between five of the six laboratories (Fig. 3.5.4). The exception is the one laboratory which also obtained deviating results for the coniferous wood pellets and the deviating result was here also excluded in the calculation of the repeatability and reproducibility (Table 3.5.2).

The sieving of the straw pellet material caused problem in three of the laboratories where mass losses of the disintegrated material during the sieving operations caused rejection of the results. However, for the three laboratories where the disintegration of straw pellets was completed without problems the resulting particle size distributions was very similar (Fig. 3.5.4). In the following evaluation of the method concerning straw pellets only the results from three of the six laboratories are used.

The mean value, the standard deviation (SD) and the coefficient of variance (CV) at the 25%-, 50%- and 75% quantile for the cumulative particle size distributions of straw-, coniferous- and deciduous pellets is presented in Table 3.5.1. The coefficient of variation expresses the standard deviation as a percentage of the sample mean making the size of variation relative of the size of observation. In all but three tests (Lab 2, straw and Labs 3 and 4 deciduous) the CV decrease

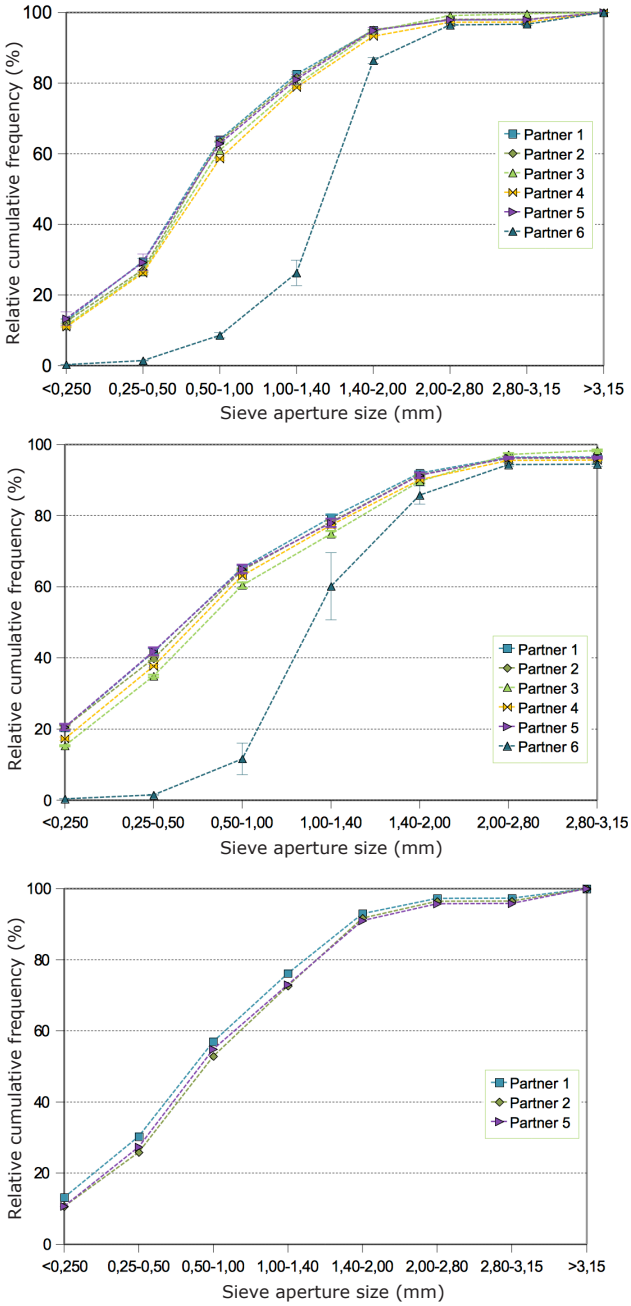

Fig. 3.5.4. Particle size distribution tested in a round robin with six laboratories. Coniferous pellets (upper figure), deciduous pellets (middle figure) and straw pellets (lower figure) are disintegrated using heated water combined with stirring of the slurry. The sieving of the straw pellet material caused problem in three of the laboratories, hence only the results from three of the six laboratories are presented.

Table 3.5.1. Mean value, standard deviation and coefficient of variance for the 25%-, 50%- and 75% quantiles from the particle size distribution of the tested solid biofuel pellets.

Lab	Quantiles	Straw pellets			Deciduous wood pellets			Coniferous wood pellets		
		25%	50%	75%	25%	50%	75%	25%	50%	75%
1	Mean (mm<)	0.42	0.87	1.38	0.30	0.68	1.27	0.43	0.80	1.24
	SD (mm<)	0.025	0.041	0.054	0.011	0.020	0.023	0.011	0.013	0.015
	CV	5.81	4.71	3.91	3.75	2.92	1.81	2.63	1.57	1.25
2	Mean (mm<)	0.49	0.95	1.47	0.31	0.71	1.31	0.46	0.81	1.25
	SD (mm<)	0.006	0.011	0.026	0.007	0.010	0.017	0.014	0.005	0.003
	CV	1.32	1.19	1.75	2.21	1.48	1.30	3.05	0.63	0.23
3	Mean (mm<)	-	-	-	0.37	0.80	1.41	0.48	0.84	1.30
	SD (mm<)	-	-	-	0.002	0.012	0.032	0.005	0.003	0.002
	CV	-	-	-	0.57	1.46	2.30	0.98	0.30	0.13
4	Mean (mm<)	-	-	-	0.34	0.74	1.33	0.48	0.87	1.33
	SD (mm<)	-	-	-	0.006	0.014	0.028	0.006	0.007	0.008
	CV	-	-	-	1.64	1.92	2.11	1.29	0.84	0.60
5	Mean (mm<)	0.47	0.91	1.46	0.30	0.68	1.31	0.43	0.81	1.27
	SD (mm<)	0.036	0.052	0.079	0.011	0.023	0.037	0.035	0.031	0.035
	CV	7.71	5.71	5.38	3.61	3.35	2.83	8.13	3.84	2.78
6	Mean (mm<)	-	-	-	1.11	1.32	1.72	1.38	1.63	1.88
	SD (mm<)	-	-	-	0.045	0.065	0.157	0.075	0.026	0.014
	CV	-	-	-	4.07	4.94	9.12	5.45	1.57	0.74

Table 3.5.2. Performance characteristic for the method calculated as repeatability and reproducibility.

Material	Quantiles (%)	N	X (mm)	S_r (mm)	S_R (mm)	S_r (%)	S_R (%)
Coniferous pellets	25	5	0.46	0.018	0.039	3.94	8.53
	50	5	0.83	0.016	0.049	1.94	5.93
	75	5	1.28	0.018	0.063	1.40	4.92
Deciduous pellets	25	5	0.33	0.0082	0.055	2.51	16.80
	50	5	0.72	0.016	0.089	2.22	12.36
	75	5	1.33	0.028	0.087	2.11	6.54
Comminuted straw	25	3	0.46	0.025	0.056	5.46	12.23
	50	3	0.91	0.039	0.067	4.30	7.37
	75	3	1.44	0.057	0.092	3.96	6.31

N: number of values, X: mean value, s_r: estimate of repeatability, S_r: estimate of relative repeatability, s_R: estimate of reproducibility, S_R: estimate of relative reproducibility.

with increasing quantiles indicating that the relative accuracy of the particle size determinations decline with decreasing particle size. This most likely reflects the increasing probability of clogging of the openings in the sieves with small apertures.

The repeatability was found to be smaller than the reproducibility indicating that the disintegration and sieving procedure varies between the laboratories and thereby influence the result of the method.

3.5.4. Conclusions

The result of the initial wet disintegration study illustrated that wet disintegration in water at ambient temperature is an unsuitable method for determining the internal particle size distribution of wood- and straw pellets. The method does not adequately separate the particles in the pellets from each other and thereby result in an overestimation of larger particles. When the wet disintegration was performed with water heated to the boiling point and coupled with mechanical disintegration in terms of stirring a more complete disintegration of the pellets was obtained. In the round robin test the method combining the use of heated water and stirring of the slurry was tested on solid biofuel pellets produced of comminuted straw and deciduous- and coniferous wood. With this revised method a satisfactory disintegration was obtained of all three types of pellets.

The dry disintegration of coniferous pellets resulted in smaller particle sizes compared to the wet disintegration using heated water and stirring of the slurry. The particle size distribution of the dry disintegrated pellets to a large extend reflects aperture size of the applied sieves in the hammer mill and the further comminution by the hammer mill of the disintegrated particles.

It is not possible from the present study to identify whether wet or dry disintegration as applied here, describes the resulting particles size distribution of a large scale hammer milling operation in the best manner. However, as shown in the round robin test the revised wet disintegration method gives highly repeatable and reproducible results and the method may provide both a relative measure of the particle size distribution of the produced fuel powder in a large scale milling operation and the particle size distribution of the raw material used for the pellet production respectively. Further, the wet disintegration method can be conducted by any laboratory having access to sieving equipment, whereas the dry disintegration demands a hammer mill and a procedure which secure that the dry disintegration results in comparable results between laboratories. By comparing results obtained with the wet disintegration method to the particle size distribution for the actual produced fuel powder in large scale hammer mills a functional relationship might be constructed. The relationship would be explicit for the specific hammer mill and the operation hereof.

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Chapitre 4

Von Rittinger theory adapted to wood chip and pellet milling, in a laboratory scale hammermill

The study draws upon the milling theories developed for the ore processing industry (Von Rittinger, Kick and Bond theories) in order to define a method for characterising wood chip and pellet energy consumption during milling.

Energy consumption during wood milling depends on three main factors: the material moisture content, the particle size difference between the feed and the milled product, and the material itself. The latter may be characterised by a single parameter based on an adaptation of Von Rittinger's constant.

A relation characterising wood pellet energy consumption as a function of the particle size distribution of the pellet ingredients and the milled pellets is proposed. This is characteristic of each type of pellet for each moisture content value considered.
Keywords: Milling; Wood chips; Pellets; Grindability; Von Rittinger; Kick.

La théorie de Von Rittinger adaptée aux plaquettes et aux pellets de bois, dans un broyeur de laboratoire. L'étude s'inspire des théories sur le broyage issues de l'industrie traitant les minerais (théories de Von Rittinger, Kick et Bond) pour définir une méthode de caractérisation de la consommation énergétique des plaquettes et des pellets de bois.

La consommation énergétique des plaquettes de bois dépend de trois facteurs principaux : l'humidité de la matière, la différence de granulométrie entre alimentation et produit broyé et la matière elle-même. Cette dernière peut être caractérisée par un paramètre unique, basé sur une adaptation de la constante de Von Rittinger.

Une relation caractérisant la consommation énergétique des pellets de bois en fonction des distributions granulométriques des constituants des pellets et des pellets broyés est proposée. Elle est caractéristique de chaque type de pellets pour chaque humidité considérée.

4.1. Introduction

The international drive to cut greenhouse gas emissions, carbon dioxide in particular, has prompted many electricity producers to convert some of their pulverized coal or gas plants to biomass co-combustion plants. There is even an example of a plant being converted to run on wood pellets exclusively [1]. Adding biomass to fossil fuels has a number of advantages. From an environmental point of view using biomass reduces fossil CO_2, SO_x and NO_x emissions rapidly. Moreover, from a technical point of view boiler efficiency increases and lower fuel cost makes the plants more cost-effective [2-4].

Some power plants use biomass (generally wood-derived) in pulverized form, which requires milling before use to produce wood particles that have the requisite aerodynamic and combustion properties to be used in the process. In such systems the characteristics of the biomass particles are vitally important as they impact upon feeding, combustion kinetics, the combustion residue volume and the hearth temperature [5-8].

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For most such co-combustion units the specification requires all of the particles to pass through a 6.34 mm mesh and the majority to pass through a 3 mm mesh [4]. However, some consumers sometimes apply stricter specifications [1,9].

Grinding chips and pellets can pose problems, however. There is a lack of data for dimensioning the mill when designing power plant supply circuits. Conversely, in existing circuits, acceptance of new materials requires true scale pilot projects. More generally, little research appears to have been done overall into fine milling of biomass.

This paper therefore attempts to identify ways of characterising the energy requirements for wood chip and pellet milling. To that end, existing knowledge of biomass milling is assessed in the light of practice in other sectors such as the food and ore processing industries.

4.1.1. Milling theories

Milling theory is based on the relation between energy consumption and the product particle size obtained from a given feed size. In Eqs. (1) to (5), $E_{1\text{-}2}$ (specific energy) is the milling energy needed for a particle size reduction from x_1 to x_2, per unit of mass; x is a particle size distribution characteristic (for example, Q80 or Q50 - the passing size of 80% or 50% of the measured material, respectively - the 80 or 50 quantile of the distribution), the indices 1 and 2 of x (x_1, x_2) indicate that x relates to the feed and the milled product, respectively, n is an exponent expressing the process magnitude, and C is a constant characterising the material to be milled, the units of which balance the equation.

Historically, a number of milling theories have been proposed [10]. Of these, three deserve closer attention as they have come down through the generations and are still cited in many publications today.

Von Rittinger theory (1867) is the oldest. This states that the new surface area generated is directly proportional to the energy required for size reduction. As the surface area of a quantity of particles of uniform diameter x is proportional to $1/x$, the energy required for size reduction is therefore also proportional. It is expressed by the general formula (Eq. (1))

$$E_{1\text{-}2} = C_{VR}\left(\frac{1}{x_2} - \frac{1}{x_1}\right) \qquad (1)$$

where C_{VR} is a constant characteristic of the material.

Kick (1885), meanwhile, postulated that the work required is proportional to the volume reduction of the particles concerned. Kick's equation is shown below:

$$E_{1\text{-}2} = C_K\,(\ln(x_1) - \ln(x_2)) = C_K \ln\frac{x_1}{x_2} \qquad (2)$$

where C_K is a constant characteristic of the material. The ratio x_1/x_2 is sometimes called the reduction ratio.

From a physical point of view, Bond's theory (1952) assumes that the energy transmitted to a body by a compressive force is initially distributed in the mass and is proportional to x_1^3, but as soon as surface cracking starts, that energy is concentrated in the cracks and is then proportional to x_1^2. Consequently, it is assumed that grinding proper is halfway between x_1^2 and x_1^3.

According to this theory, for particles of similar shape, the crack length is equivalent to the square root of the surface. The milling energy being proportional to the crack length, it is expressed according to Eq. (3) as:

$$E_{1\text{-}2} = C_B\left(\frac{1}{\sqrt{x_2}} - \frac{1}{\sqrt{x_1}}\right) \qquad (3)$$

where C_B is a constant characteristic of the material.

Von Rittinger's, Kick's and Bond's laws were collated by Charles [11], who stated that the relations between energy E and par-

ticle size characteristic x established by these three authors are special forms of the same differential equation (Eq. (4)).

$$dE = -C\frac{dx}{x^n} \quad (4)$$

The exponent n of x takes the values of 2, 1 and 1.5 for the Von Rittinger, Kick and Bond theories, respectively.

Hukki [12] then showed for the mineral industry that each of these values corresponds to a relatively restricted particle size distribution range and that the exponent is not constant but depends on the particle size distribution level x itself, as expressed by Eq. (5).

$$dE = -C\frac{dx}{x^{f(x)}} \quad (5)$$

That relation would subsequently be developed and refined. Morell [13] postulated that the constant C is itself dependent on the particle size distribution and is not the same for all rocks. Stamboliadis [14] proposes a relation where the effects of particle size distribution are expressed in the form of a theoretical frequency distribution.

With a view to their application to wood size reduction, the above studies have the advantage of estimating the energy consumption of a grinding circuit from a small number of parameters. These are applicable to a wide range of different materials, from a hardness point of view at least. Even though they were developed for the kind of mill used in the mineral industry, they indicate that a relationship may be established between the energy consumed by milling a material and the particle size distribution of the product and of the raw material.

4.1.2. Biomass grinding

Relatively little information is available in the literature on energy consumption for biomass grinding. Mani et al. [15] quote a few authors who studied this in the 1980s. More recently, Vigneault et al. [16,17] published energy consumption data for corn grinding. Since then, the publications on this topic have been mainly the work of few authors, including Mani [9,18-23]. Also, the development of the wood torrefaction technique and its impact on the grindability of biomass [24-26] provides some measurements of energy consumption in wood size reduction [27]. Lastly, the comminution of Miscanthus, switchgrass, energy cane and poplar was recently studied by Miao [28]. The data published by these different authors are summarised in Table 4.1.

The work of Vigneault et al. [16,17] involves a comparison of corn grinding energy consumption according to the hammer design and type of screen fitted to the grinder in a commercial mill. The research shows the significant effects of hammer design and rate on energy consumption. All other things being equal, measured consumption varied from 10 to 12 kWh/tonne for thin and conventional hammers, respectively. The type of screen used also affected grinding energy consumption.

Laskowski et al. [19-21] established a relationship between the compression behaviour of seeds and energy consumption in a laboratory-scale mill. This work clearly shows the effects of moisture content and comminuted material on energy consumption. For a given material, the higher the grain moisture content, the higher the grinding energy consumption. Measured consumption levels ranged from 15 to 144 kWh/tonne.

The milling characteristics of three forms of *Cynara cardunculus* L., were described by Gil et al. [22] in a study on the use of this raw material as solid biofuel. The moisture content of the considered materials is between 9.5% and 13.0%. Trials were made in a hammermill equipped with interchangeable screens perforated by 5, 2, 1 and 0.5 mm mesh. The work shows the influence of the screen

Table 4.1. Energy required to grind different biomasses (E_{cn}): some published data (index 1, feed; index 2, product; PSD, particle size distribution; H, moisture content; H Pc and H Pb, considered material moisture content; Gd, screen; GM, geometric mean; MS, average; Q50, quantile 50; Q95, quantile 95; P, relative energy measurement; N, netbiomass; G, overall grinding circuit; NS, not specified).

Material	PSD in (x_1-mm)	PSD out (x_2-mm)	H1 (%)	H2 (%)	P	E_{cn} (kWh/t)
Source: [15]						
Wheat straw	GM: 7.67	Gd 0.8; 1.6; 3.2	NS	8.3	N	11.4-51.6
	GM: 7.67	Gd 0.8; 1.6; 3.2	NS	12.1	N	24.7-45.3
Barley straw	GM: 20.52	Gd 0.8; 1.6; 3.2	NS	6.9	N	13.8-53.0
	GM: 20.52	Gd 0.8; 1.6	NS	12.0	N	27.1-99.5
Corn stover	GM: 12.48	Gd 0.8; 1.6; 3.2	NS	6.2	N	7.0-22.1
	GM: 12.48	Gd 0.8; 1.6; 3.2	NS	12.0	N	11.0-34.3
Switchgrass	GM: 7.15	Gd 0.8; 1.6; 3.2	NS	8.0	N	23.8-62.6
	GM: 7.15	Gd 0.8; 1.6; 3.2	NS	12.0	N	27.6-58.5
Source: [18]						
Poplar	Q_{50}: 8.0	Q_{95}: 1.0	11.8	8.1	G	82-89
Pine	Q_{50}: 9.2	Q_{95}: 1.0	HPc	HPc - 34%	G	113.2-119.1
Pine bark	Q_{50}: 8.25	Q_{95}: 1.0	HPb	HPb - 9%	G	18.1-23.6
Source: [27]						
Poplar	NS	MS: 0.2-1.2	13	NS	N	100-375
Poplar	NS	MS: 0.2-1.2	10	NS	N	50-150
Source: [19-21]						
Beans	NS	Grid 1.0	10-18	NS	NS	27-64
Peas	NS	Gd: 1.0	10-18	NS	NS	23-50
Lupins	NS	Gd: 1.0	10-18	NS	NS	70-144
Vetch	NS	Gd: 1.0	10-18	NS	NS	15-36
Source: [16]						
Corn	NS	GM: 0.49-0.51	NS	NS	NS	10.0-12.4
Source: [9]						
Pine chips	8	Gd: 3; 4; 6	10-15	8.6-9.3	G	36-53
Source: [22]						
Cynara pellets	NS	Q_{50}: 0.5-0.25	12.0 ± 0.3	11.0 ± 05	G	6.1-35.0
Cynara stem	NS	Q_{50}: 0.77-0.27	10.4 ± 0.2	9.5 ± 0.6	G	15.0-79.9
Cynara plant	NS	Q_{50}: 0.71-0.24	13.0 ± 0.8	11.0 ± 0.7	G	14.2-60.8
Source [23]						
Switchgrass	GM: 8.3	GM: 0.43-0.65	9.0 ± 0.5	NS	G	31.8-46.0
	GM: 8.3	GM: 0.43-0.65	9.0 ± 0.5	NS	N	14.7-18.5
Wheat straw	GM: 7.1	GM: 0.66-0.83	9.0 ± 0.5	NS	G	34.8-46.8
	GM: 7.1	GM: 0.66-0.83	9.0 ± 0.5	NS	N	14.6-19.1
Corn stover	GM: 8.3	GM: 0.53-0.63	9.0 ± 0.5	NS	G	28.8-47.7
	GM: 8.3	GM: 0.53-0.63	9.0 ± 0.5	NS	N	13.0-19.8

on the global electrical consumption of the milling circuit. It is as well observed that *C. cardunculus* L. pellets need less energy to be milled (6.1-35 Wh/kg depending on the used screen) if compared to whole plants and stems (15.0-79.9 Wh/kg and 14.2-60.8 Wh/kg, respectively).

The milling of switchgrass, wheat straw and corn stover in a hammermill equipped with a 3.2 mm mesh screen has been studied by Bitra et al. [23]. Two hammer types and five rotation speeds of the rotor are compared on an electrical consumption basis, considering total specific energy and effective specific energy (total energy from which the empty consumption of the hammermill has been taken away). The global specific energy consumption (empty and in charge) is influenced by the rotor speed and ranges from 31.8 to 46.0 Wh/kg, from 34.8 to 46.8 Wh/kg and from 28.8 to 47.7 Wh/kg for switchgrass, wheat straw and corn stover, respectively. In the same test conditions, effective specific energy is less influenced by the hammermill settings. In this case, the recorded consumption range from 14.7 to 18.5 Wh/kg, from 14.6 to 19.1 Wh/kg and from 13.0 to 19.8 Wh/kg for switch-grass, wheat straw and corn stover, respectively.

In a study designed to assess the pros and cons of combining wood torrefaction and densification (pelletisation), Bergman et al. [27] compared the energy consumption when grinding dry and moist poplar with the values for torrefied wood, using a disk chipper. According to the particle size distribution (average particle size 0.2-1.2 mm), grinding oven-dry wood consumed between 50 and 150 kWh/tonne, whereas slightly moister wood (10-13%) required a consumption range of 100-325 kWh/tonne, for the same particle size distribution range. These measurements also show a clear trend towards higher energy consumption for the finest degrees of milling.

Wood size reduction has also been studied by Esteban et al. In an initial paper [9] on the energy efficiency of a pellet production circuit, the raw material comprised Norway pine logging residue chips conditioned to a homogeneous moisture content of between 10% and 15% that had undergone initial crushing in a hammermill fitted with a perforated screen with 8 mm diameter mesh. This material then underwent further refining in the same mill with 6, 4 or 3 mm screen sizes, alternatively. The total energy consumption for each pass was measured and ranged from 29 to 53 kWh/tonne.

In a second paper Esteban et al. [18] compared the efficiency of different grinding circuits, from an energy consumption point of view in particular. Three materials with similar particle size distributions were tested: poplar chips (Q50:8 mm), pine chips (Q50:9.3 mm) and pine bark (Q50:8.25 mm). Twelve one-stage or multi-stage material preparation circuits were assayed with the aim of producing particles that could be used to feed pulverized fuel burners (Q95:1.0 mm and Q12: 0.125 mm). The overall energy consumption values for the different alternatives were measured and compared with one another. It is to be noted that few of the alternatives tested achieved the target particle size distribution. However, the energy consumption of the different material/grinding circuit combinations varied more from one material to another than from one process to another. The average overall energy consumption for all the processes tested for grinding poplar was 85.4 kWh/tonne (with a standard deviation of 4.9 kWh/tonne), the value for pine chips was 118.5 kWh/tonne (standard deviation 6.2 kWh/tonne) and that for pine bark was 19.7 kWh/tonne (with a standard deviation of 3.9 kWh/tonne). The authors also noted that grinding greatly reduced the material moisture content in the case of all three materials tested.

Mani et al. studied the energy consumption for grinding biomass [15]. The authors looked at various factors that could affect

grinding energy consumption. Material properties listed include the type of biomass itself, moisture content, particle size distribution (before and after grinding), bulk density and particle density. The equipment itself can also have an impact, for example, the type of grinder used, the settings, feed rate.

Using a low-powered grinder Mani et al. studied the net energy consumption when grinding four materials (wheat straw, barley straw, corn stover and switchgrass). The particle size distribution was expressed in terms of the geometric mean (switchgrass: 7.15 mm; wheat straw 7.67 mm; corn stover: 12.48 mm; barley straw 20.52 mm). Three screen sizes were used to grind the biomass, which was pre-conditioned at two moisture content levels (approx. 8% and 12%). Measured consumption data varied from 11.04 kWh/tonne to 62.55 kWh/tonne according to the above influencing factors. On the basis of these data the authors defined relations between the grinder screen size and energy consumption. These are linear for the driest materials and polynomial for the highest moisture content.

Mani et al. were probably the first to formalise the relationship between energy consumption and an expression of product particle size distribution (screen size) in biomass grinding. Miao confirmed the effects of moisture content on grinding energy consumption. Moreover, he proposed a power type relation to express the energy consumption according to the screen diameters used in comminution [28].

4.1.3. What milling theory for biomass?

From an analysis of the available literature it appears that the material ground and its origin affect the grinding, giving rise to a specific relationship between energy consumption and particle size distribution for a given system. This has been shown in the case of ores but little work has been done on biomass. For example, the particle size distribution of the starting material is not always measured and, when it is, the deviation from the product particle size distribution is rarely considered when stating the results. However, the particle size distribution deviation between the initial state and final state of the material is the very basis of milling theory in the mineral industry.

Moisture content is probably the primary additional influencing factor to be taken into consideration when studying the use of biological materials, i.e., biomass. Grinding is no exception. However, the scant data available on this topic are sometimes contradictory. All the same, it appears appropriate to take the view that the higher the moisture content of a material, the more energy will be consumed in grinding it.

No data are available in the literature concerning energy consumption for grinding wood pellets. The influencing factors are likely to be the same as with other types of biomass. However, some specific properties of biofuel pellets need to be considered, namely the durability and particle size distribution of the raw material, both included in the specification by the main power plant pellet users. The pellet raw material undergoes a final particle size reduction before use in the power plant. This reduction is probably a big energy consumer, compared with that which could be expressed in terms of durability. The cohesion of the material within the particles forming the pellets is in fact likely to be greater than the cohesion between the particles.

This study therefore sets out firstly, to assess the possibility of using ore milling theories to characterise biomass grinding and identify the most appropriate parameters for the purpose. Secondly, to propose a biomass grinding procedure and model that will produce comparable results for different forest species. And lastly, to assess the applicability of the proposed model to milling of pellets.

4.2. Materials and methods

4.2.1. Test bench

An outline diagram of the equipment used for the purpose of this study is provided in Fig. 4.1. The mill was equipped with six swinging metal hammers (flails) equally spaced around a 100 mm diameter steel disc. The centre of the axis around which the hammers swung was 12.5 mm from the edge of the disc. The hammers were T-shaped with a 5 mm side cross-section. The swinging part of the hammers was 37.5 mm in length from the centre of the axis of rotation to the base of the T part perpendicular to it. The milling chamber had an inside diameter of 190 mm and its width was 55 mm. An interchangeable curved screen with an inside diameter of 160 mm and 55 mm wide was centred within the milling chamber. The rate of rotation of the drive shaft was 2800 rpm. The electric motor developed a rated power of 1.1 kW. Six different screens were used on the hammermill for the purpose of the experiment. The screens were perforated with round meshes of diameter 8, 6, 5, 4, 3 and 2 mm.

The grinder was fed manually at a regular rate, taking care not to overload it. The feed rate therefore varied according to the characteristics of the test materials. The mill discharged into a box from which the grind was collected in order to determine the particle size distribution and moisture content. The grinder power supply leads were connected to a HIOKI 316920 power demand analyzer to measure and record the differential potential (V), current intensity (A), grinder instantaneous power draw (W), frequency (Hz) and time (s). The measuring interval was 1 s. The equipment also calculated the energy consumption (Wh) for grinder operation. The data were recorded by a data logger and transmitted to a computer.

4.2.2. Wood chip characteristics

The wood chips used in this research came from two deciduous species and two coniferous species: oak (*Quercus* sp.), beech (*Fagus sylvatica* L.), spruce (*Picea abies* (L.) Karst) and pine (*Pinus* sp.). Chips were prepared from wood rafters (without bark and preparatory drying) produced in Belgian sawmills, which were chipped using a disc chipper (Van Deale TS 180). Each material was prepared in order to produce five input moisture content classes. The averages for these classes (and the number of observations) are shown in Table 4.2. They range from 1.1% to 22.4%. The maximum value of the median of the feed particle size distribu-

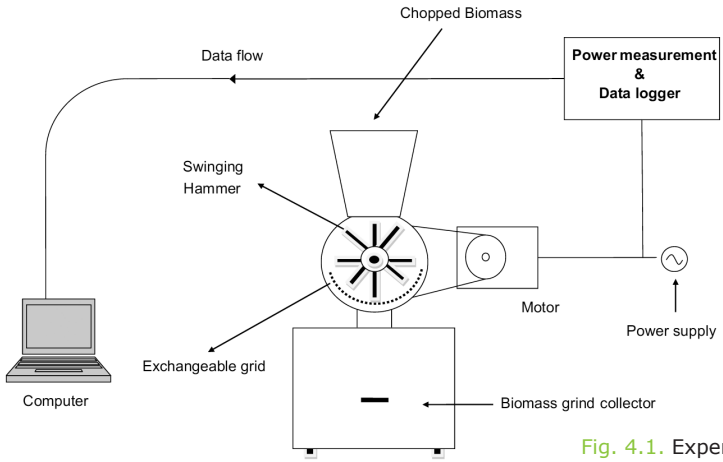

Fig. 4.1. Experimental milling equipment.

Table 4.2. Moisture content of wood chips tested in grinding trials. H1, H2, H3, H4 and H5 (in %) are the average values of the moisture content classes for each material; (n) is the number of observations in the class, Q50 Max and Min (in mm) are the maximum and minimum medians of the particle size distributions for each species (for all moisture content classes).

	H1% (n)	H2% (n)	H3% (n)	H4% (n)	H5% (n)	Q_{50} Max (mm)	Q_{50} Min (mm)
Coniferous							
Pine	4.9 (13)	9.0 (13)	13.5 (15)	17.4 (16)	20.8 (5)	4.93	0.40
Spruce	1.5 (12)	7.0 (13)	12.2 (16)	16.1 (13)	21.3 (8)	7.38	0.44
Deciduous							
Oak	1.4 (13)	7.8 (15)	12.4 (14)	17 (12)	22.4 (9)	4.98	0.45
Beech	1.1 (13)	8.1 (13)	13.4 (13)	16.5 (11)	21.7 (13)	5.83	0.46

tions (Q_{50} max) is 7.38 mm and the minimum value of the median of the product particle size distributions (Q_{50} min) is 0.40 mm.

4.2.3. Wood chip grinding method

Five moisture content classes. To achieve the different moisture content levels (H1-H5, see Table 4.2) to be grouped into five classes, the fresh chips were air-dried or oven-dried (105 °C, as long as necessary to reach the desired moisture content). To avoid clogging the meshes, in particular the smaller ones, with over-moist material the moisture content levels greater than 25% were disregarded in this experiment.

Twelve particle size distribution treatments. Initially, the chips underwent preliminary grinding in an industrial grinder before being screened on a 16 mm diameter round mesh screen. The aim of this step was to make the particle size distribution of the test chips compatible with the laboratory-scale mill used. About 8 kg of material was then divided into eight sub-samples of approximately 1 kg. Grinding was carried out using six screens with different mesh diameters. The screen mesh diameters were 8, 6, 5, 4, 3 and 2 mm.

In the course of the grinding protocol (illustrated by Fig. 4.2) the eight sub-samples (RM 1-8) underwent initial grinding using an 8 mm diameter mesh screen (G8, replicates 1-8). The grind was recovered and reground using screens with mesh of a different diameter. One grinding pass (G8 R1) was performed with a 6 mm screen (G86), 3 passes (G8 R2-4) with a 5 mm screen (G85 R 1-3), 2 passes (G8 R5 and 6) with a 4 mm screen (G84 R1 and 2), 1 (G8 R7) with a 3 mm screen (G83) and 1 (G8 R8) with a 2 mm screen (G82).

In each case, the resulting grind was recovered and reground. The grind from the 6 mm screen (G86) was reground on a 4 mm screen (G864) before being recovered again and ground on a 2 mm screen (G8642).

The grind from the 5 mm screen (G85 R1-3) was ground on different screens, one on the 4 mm screen (G854), one on the 3 mm screen (G853) and one on the 2 mm screen (G852).The grind from the 4 mm screen (G84 replicates 1 and 2) was also ground on different screens, one on the 3 mm screen (G843) and one on the 2 mm screen (G842).

This procedure generated 13 different treatments characterised by particle size distribution differences between the feed particle size distribution and the product particle size distribution. At least one repetition has been performed for each treatment, but the procedure imposed to repeat some treatments more than once: millings with 8 mm (G8), 5 mm (G85) and 4 mm (G84) mesh screen were performed in 8, 3 and 2 replications, respectively. In this case mean values of the

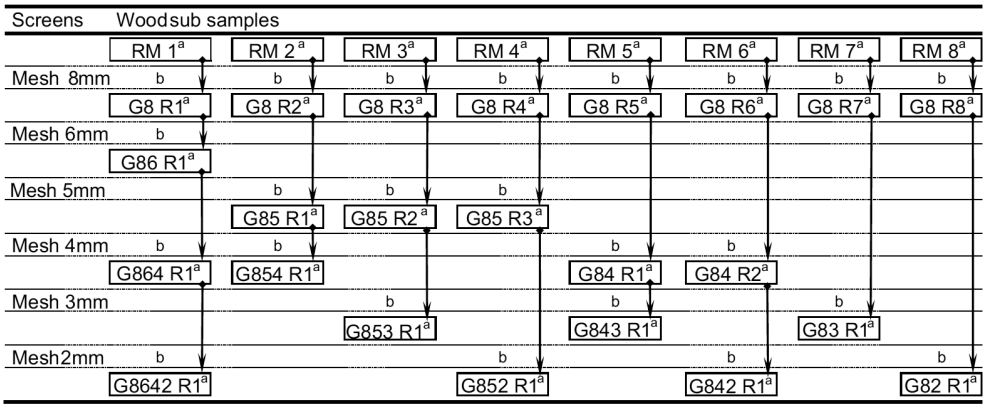

Fig. 4.2. Wood milling experimental plan applied to each moisture content class and wood species combination: (a) mass, moisture content and particle size distribution determination, (b) energy recording for milling from the feed into the product.

repetitions was considered. Considering the five moisture content classes, 62 different treatments have been applied to Pine and Spruce, while 63 have been applied to Oak and Beech. In each trial one sample was taken to measure the moisture content and the particle size distribution before and after grinding. The procedure was repeated for the four selected materials, for each moisture content class.

Measured data. When grinding each subsample the following data were collected: sample mass (g), idle consumption (Wh) and consumption over the grinding period (Wh), feed particle size distribution (mm – according to the size of the particles this characteristic was determined in accordance with the instructions given in European standards EN 15149:2010-1 or EN 15149:2010-2 [29,30]), the product particle size distribution (mm – measured according to EN 15149:2010-2 [29]), the feed and the product moisture content (% – measured according to EN 14774:2009-2 [31]).

Collected data. The following parameters were calculated with the aid of the measured data: E_{cn} = net energy consumption, per kg of dry material input, given by the consumption over the grinding period less the idle consumption (Wh/kg oven-dry); the medians of the material particle size distributions before and after grinding.

Determination of the most appropriate milling theory to characterise biomass. Based on Charles' relation (4) and knowing the particle size distribution (distribution median) of the material before and after grinding, there is a pair (constant *C* and exponent *n*) which minimises the deviation sum of squares (DSS) between the measured energy consumption values and those estimated with the aid of the model. The average of the exponents n thus determined was then compared to the values of that parameter according to the Von Rittinger, Kick and Bond laws (2, 1.5 and 1), in order to select the most appropriate one to express wood grinding energy consumption.

With the parameter n fixed, the constant *C* which minimises the deviation sum of squares was then recalculated for each material and each moisture content level.

Finally, for each species of wood a linear relation (passing through the origin) between *C* and the average moisture content of the class was determined. The coefficient of that regression line characterised the species and expressed its grindability.

4.2.4. Pellets characteristics

The five batches of commercial wood pellets used in this study were of various origins and their characteristics are shown in Table 4.3. For each batch, the trials were grouped into two moisture content classes. The averages of the values in those classes (*H*1 from 0.18% to 0.97% and *H*2 from 5.71% to 12.44%) are also shown in the table. The following properties were also considered: durability (DU, 98.5% to 99.3%), particle density (PD, 1.23 to 1.32 g/cm^3), fines (*F*, 0.12-4.9%), bulk density (BD, 632-722 kg/m^3).The median of the particle size distribution of the pellet ingredients (PIPSDQ50, 0.39-1.18 mm) completes the list of properties considered.

4.2.5. Pellets grinding method

Two moisture content levels. The nature of the pellets restricts the number of moisture content classes. The pellets were therefore tested at two moisture content levels (moisture content at delivery and after oven drying for 24 h at 105 °C).

Six particle size reduction levels for whole pellets. Approx. 16 kg of pellets were divided into eight sub-samples (P1-P8). In the course of the grinding protocol (illustrated by Fig. 4.3) these whole pellet sub-samples were milled using screens of different mesh diameters (8, 6, 5, 4, 3 and 2 mm). The resulting grind (PG8 R1 to R3, PG6, PG5, PG4, PG3, PG2) was collected and then milled again using screens with smaller mesh sizes.

Sixteen particle size distribution treatments for pre-milled pellets. The grind from the first sub-sample (PG8 R1) underwent a further three successive millings using the 6, 4 and 2 mm diameter mesh screens (PG86, PG864, PG8642, respectively). The grind from the second sub-sample (PG8 R2) underwent a further three successive millings using the 5, 3 and 2 mm diameter mesh screens (PG85, PG853, PG8532, respectively). The grind from the third sub-sample (PG8 R3) underwent a further two successive millings using the 4 and 2 mm diameter mesh screens (PG84, PG842, respectively).

The grind from the 6 mm screen (PG6) underwent a further three successive millings on the 5, 4, and 2 mm screens (PG65, PG654, PG6542, respectively), the grind from the 5 mm screen (PG5) underwent a further two successive millings on the 3 and 2 mm screens (PG532 and PG532, respectively), the grind from the 4 mm screen (PG4) underwent a further two successive millings on the 3 and 2 mm screens (PG43 and PG432, respectively) and the grind from

Table 4.3. Characteristics of wood pellets used in the grinding trials. *H*1, average of moisture content class 1; *H*2, average of moisture content class 2; *n*, number of observations in the moisture content classes; DU, durability; PD, particle density at moisture content *H*2; BD, bulk density at moisture content *H*2; PIPSD Q50, median of particle size distribution of pellet ingredients.

	*H*1 (*n*)	*H*2 (*n*)	DU (%)	*F* (%)	PD (g/cm^3)	BD (kg/m^3)	PIPSD Q50 (mm)
Pellets 1	0.3 (16)	12.0 (16)	98.2	0.5	1.26	646	0.708
Pellets 2	1.1 (16)	7.7 (16)	98.8	0.3	1.33	693	0.761
Pellets 3	0.6 (16)	9.2 (16)	98.9	0.6	1.27	656	0.755
Pellets 4	0.7 (16)	5.9 (16)	98.5	4.8	1.31	701	0.848
Pellets 5	0.2 (15)	7.1 (16)	99.3	0.3	1.31	689	0.756
Min	0.2	5.9	98.2	0.3	1.26	646	0.708
Max	1.1	12.0	99.3	4.88	1.33	701	0.848

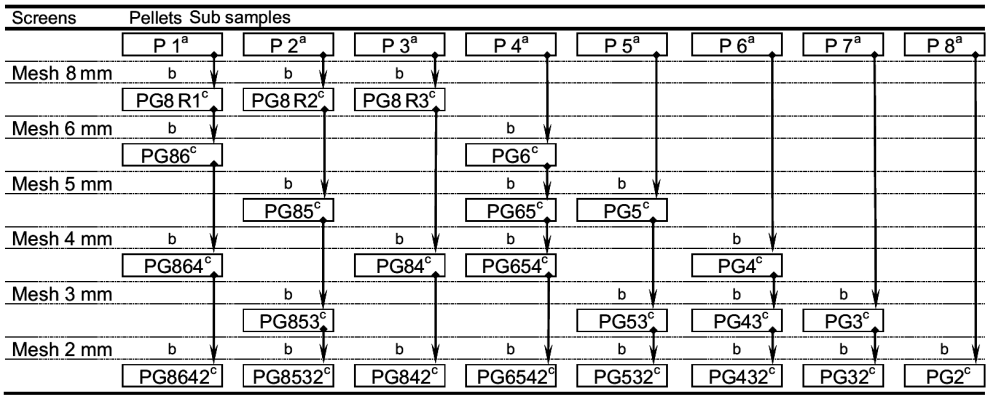

Fig. 4.3. Pellets milling experimental plan applied to both moisture content class and pellets combinations: (a) mass, moisture content and internal particle size distribution determination, (b) energy recording for milling the feed into the product, (c) mass, moisture content and particle size distribution determination.

the 3 mm screen (PG3) underwent a further 1 milling on the 2 mm screen (PG32). The last pellet sub-sample (P8) was milled on the 2 mm diametermesh screen (PG2).

Data collected. In each milling trial using whole pellets and the milled product, the data collected were identical to those obtained for the chips. Moreover, some specific properties of the pellets were also determined. These were the durability and fines rate (DU and F, measured according to EN 15210-1:2009 [32]), particle density (PD, measured according to EN 15150: 2011 [33]), bulk density (BD, measured according to EN 15103: 2009 [34]) and the median of the pellet ingredient particle size distribution (this property was determined using the disintegration and screening method recommended by Jensen et al. [35]).

Parameters calculated. The parameter used to link grinding energy consumption and particle size distribution difference between feed and products was that determined by the wood chip experiments. In this case the feed particle size distribution was characterised by the median of the pellet ingredient particle size distribution.

4.3. Results and discussion

4.3.1. Grinding energy

Table 4.4 shows the minimum and maximum energy consumption values when grinding wood chips and pellets. These data are of the same order of magnitude as those found in the literature (cf. Table 4.1), for similar wood species and moisture content levels. If all treatments are considered, it appears first of all that energy consumption varies greatly within a species, more than between species. Yet, the sample variability appears difficult to be in question as all necessary precautions have been taken in order limit to the minimum the variability between subsamples (homogeneous wood from sawmill and sample division using riffle divider). The following consumption data were recorded: for beech, 5.1-307.0 Wh/kg; for oak, 6.2-172.2 Wh/kg; for pine, 4.9-199.2 Wh/kg and for spruce 5.4-251.9 Wh/kg. The energy consumption data recorded during pellet grinding spanned a smaller range, lying between 1.4 and 18.2 Wh/kg for the five types of pellets tested. Next, the moisture content clearly affects the energy consumption when grinding chips: the higher

Table 4.4. Energy consumption for grinding wood chips and pellets: minima and maxima recorded for different material moisture content classes, for all feed and product particle size distributions (*H*, moisture content in %; *E* Min and *E* Max in Wh/kg, minimum and maximum grinding energy consumption for the material and moisture content class concerned). Bold type: minimum and maximum values for chips and pellets.

Moisture content	0 < *H* < 4.99		5 < *H* < 9.99		10 < *H* < 14.99		15 < *H* < 19.99		20 < *H* < 24.99	
Material	*E* Min	*E* Max	*E* Min	*E* Max	*E* Min	*E* Max	*E* Min	*E* Max	*E* Min	*E* Max
Beech	5.1	59.9	7.4	75.7	11.9	135.1	21.7	249.2	28.3	**307.0**
Oak	6.2	50.1	13.4	107.7	21.2	172.3	21.1	113.8	24.6	170.2
Pine	**4.9**	40.7	5.1	61.2	13.5	111.6	19.6	195.7	31.5	199.2
Spruce	5.4	54.6	7.9	162.6	14.3	244.0	15.3	240.3	17.3	251.9
Pellets 1	1.9	9.3	-	-	2.6	16.1	-	-	-	-
Pellets 2	2.2	9.4	3.0	**18.2**	-	-	-	-	-	-
Pellets 3	1.5	8.2	2.0	12.9	-	-	-	-	-	-
Pellets 4	1.6	8.3	1.9	13.0	-	-	-	-	-	-
Pellets 5	**1.4**	7.6	1.6	11.5	-	-	-	-	-	-

the moisture content, the more the energy consumption increases and the greater the differences between min and max. Pellets by their nature have a smaller moisture content range than chips. Nonetheless, this parameter also appears relevant to this product, with the highest energy consumption levels systematically being recorded for the highest moisture content classes.

However, within the moisture content classes there were considerable differences between the minimum and maximum consumption levels noted. Lastly, for similar moisture content levels, grinding pellets used less energy than grinding chips. The differences between minimum and maximum noted were also smaller.

4.3.2. Chips

For the species tested and the moisture content classes defined, the middle columns of Table 4.5 (columns *C*, *n* and DSS_{min}) show pairs *C* and *n* which minimise the DSS between the measured values and those obtained by using *C* and *n* in Charles' relation (4). It appears that the constant *C* tends to increase with the moisture content of the material, whereas the exponent *n* tends to decrease. These opposing tendencies make it hard to interpret the actual effects of moisture content and species on these materials' energy consumption during grinding. Therefore, as an initial approximation, for purposes of comparison, the materials can usefully be characterised by a single parameter (constant), at the expense of a slight loss of accuracy. The average of the exponents *n*, for all species and moisture content levels together, is 2.1. Used in Charles' relation (Eq. (4)) this value is closer to the exponent *n* leading to Von Rittinger's relation ($n = 2$) than that of Kick ($n = 1$) or Bond ($n = 1.5$). Von Rittinger's relation (Eq. (1)) is therefore suggested for the purpose of characterising grinding of the species considered. The right-hand columns of Table 4.5 (columns C_{VR}, n_{VR} and DSS_{VRmin}) show the values of the constant C_{VR} that minimise the DSS between measured values and those estimated with the aid of Von Rittinger's relation. Each species, at a particular moisture content level, is therefore characterised by a single constant.

The relation between the index expressing the particle size reduction in Von Rittinger's relation ($1/x_2 - 1/x_1$) and the grinding energy

Table 4.5. *C* and *n* pair minimising DSS, using Charles' relation and C_{VR} minimising DSS using Von Rittinger's relation. *N*, number of observations; *MC*, average moisture content of class (%); *C*, constant of Charles' relation; *n*, exponent of Charles' relation; DSSmin, sum of minimum deviations obtained with *C* and n; C_{VR}, von Rittinger's constant; n_{VR}, exponent leading to von Rittinger's relation; DSS_{VR}, least squares obtained with C_{VR} and n_{VR}.

Species	*N*	MC	*C*	*n*	DSS_{min}	C_{VR}	n_{VR}	$DSS_{VR\ min}$
Spruce	12	1.5	11.3	2.7	47.8	23.7	2.0	157.0
Spruce	13	7.0	20.8	2.9	255.9	43.8	2.0	552.8
Spruce	16	12.2	73.1	2.4	588.9	105.2	2.0	1138.3
Spruce	13	16.1	156.6	2.2	1534.9	185.5	2.0	1931.9
Spruce	8	21.3	467.5	1.5	1346.0	291.5	2.0	2920.4
Pine	13	4.9	21.9	2.3	182.9	31.8	2.0	247.5
Pine	13	9.0	36.9	2.4	354.2	55.5	2.0	527.1
Pine	15	13.5	87.4	2.4	1799.8	126.3	2.0	2540.9
Pine	16	17.4	185.7	2.0	2189.2	188.1	2.0	2191.7
Pine	5	20.8	249.6	1.8	858.1	204.5	2.0	1158.0
Beech	13	1.1	15.6	2.3	164.7	21.8	2.0	197.3
Beech	13	8.1	65.7	1.9	321.9	55.2	2.0	375.9
Beech	13	13.4	178.8	1.5	958.6	86.5	2.0	3155.3
Beech	11	16.5	141.0	1.6	565.9	77.3	2.0	1398.1
Beech	13	21.7	171.3	1.6	1056.4	99.2	2.0	2433.5
Oak	13	1.4	12.9	2.9	72.5	32.0	2.0	389.7
Oak	15	7.8	55.0	2.5	1882.3	94.7	2.0	3077.9
Oak	14	12.4	130.6	2.0	1680.7	129.3	2.0	1681.4
Oak	12	17.0	184.8	1.8	1719.7	143.6	2.0	2195.3
Oak	9	22.4	187.0	1.9	1218.3	168.2	2.0	1301.6
Total	250		Mean	2.1				

consumption is illustrated in Figs. 4.4 - 4.7 for the materials tested.

Thus expressed, the grinding energy consumption, for the four species concerned, shows two common trends. Firstly, within each moisture content level, energy consumption is proportional to Von Rittinger's particle size reduction factor. The quality of the proportionality relations between these variables is characterised by high determination factors (R^2 between 0.82 and 0.98). Von Rittinger's relation therefore largely explains the energy consumption differences within the moisture content classes for each species: the greater the difference in particle size distribution between the feed and the product, the more energy is required for grinding. In accordance with Von Rittinger, a particular species at a particular moisture content level can be considered a material, irrespective of the other moisture content levels, and characterised by a specific constant (C_{VR}).

Secondly, energy consumption rises as a function of the grinder feed moisture content level. For each species Fig. 4.8 shows the constants C_{VR}, characterising the moisture content classes, on the y-axis of the average moisture content of the class concerned. It appears that Von Rittinger's constant is proportional to the moisture content (*H*) of the material, according to a relation characteristic of the species. For each species, the relations identified have high determination factors (between 0.74 and 0.93). If *M* is defined as a constant characteristic of the species, such that $C_{VR} = M \times H$, and that rela-

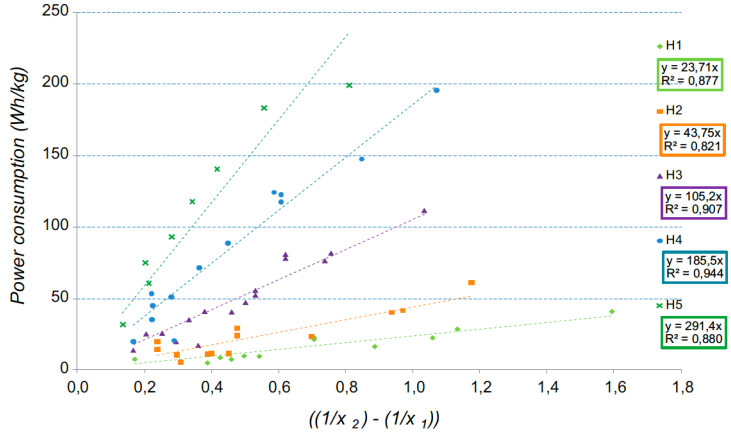

Fig. 4.4. Spruce, grinding energy consumption, for five moisture content levels increasing from H1 to H5, x_1 and x_2: feed and product particle size distribution median, respectively.

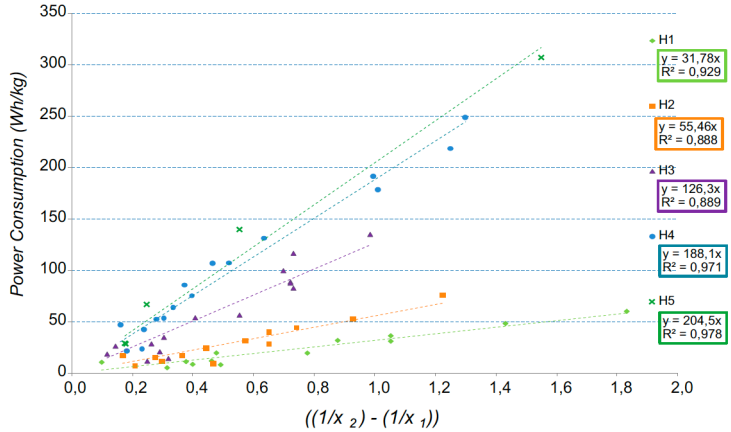

Fig. 4.5. Pine, grinding energy consumption, for five moisture content levels increasing from H1 to H5, x_1 and x_2: feed and product particle size distribution median, respectively.

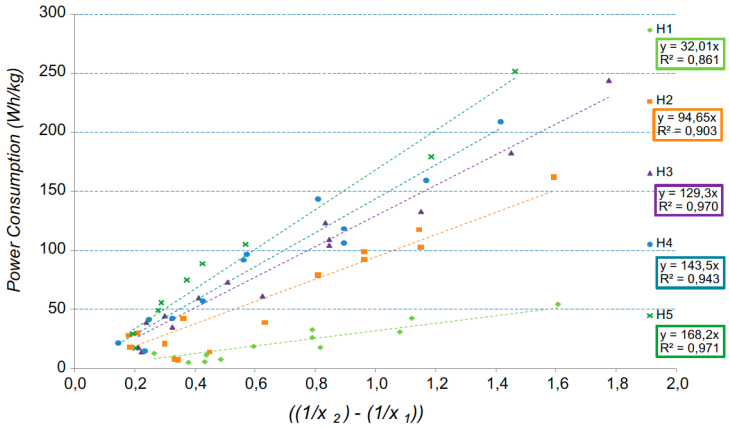

Fig. 4.6. Oak, grinding energy consumption, for five moisture content levels increasing from H1 to H5, x_1 and x_2: feed and product particle size distribution median, respectively.

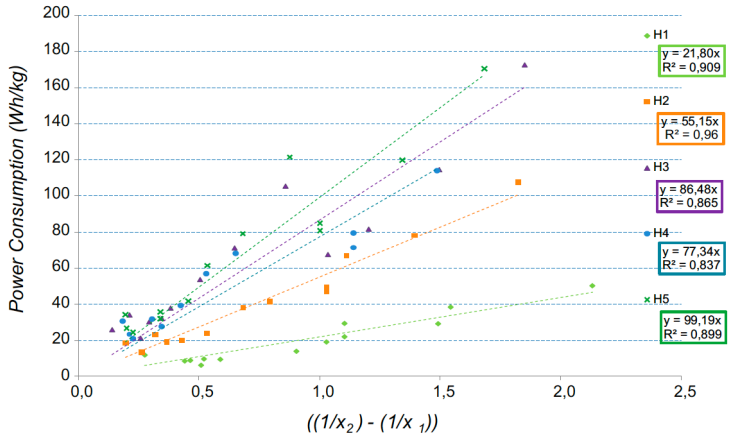

Fig. 4.7. Beech, grinding energy consumption, for five moisture content levels increasing from H1 to H5, x_1 and x_2: feed and product particle size distribution median, respectively.

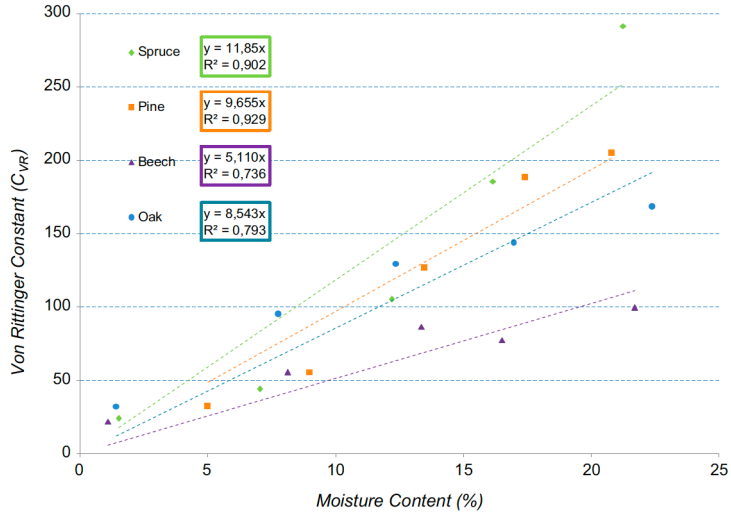

Fig. 4.8. Trend of Von Rittinger's constant as a function of moisture content for four species of wood (H: moisture content on moist base in % and C_{VR}, Von Rittinger's constant).

tion is introduced into Eq. (1), the specific grinding energy can be expressed by Eq. (6).

$$E_{1\text{-}2} = M\,H\left(\frac{1}{x_2} - \frac{1}{x_1}\right) \qquad (6)$$

Factor M characterises the grindability of a species with the aid of a single constant, the moisture content and the particle size distribution characteristics of the feed and the product. The values are beech, M = 5.11; oak, M = 8.54; pine, M = 9.65; and spruce, M = 11.85. The unit associated with the constant M balances the units of the equation when the x are expressed in mm, mass in kg, moisture content in % and energy consumption in Wh/kg dry matter. The quality of the model using the parameter M could be improved and in fact, more complex models would lead to a better fit. For example, the middle columns of Table 4.5 show that using a variable pair of parameters C and n results in a lower DSS than is the case when parameter n is fixed to obtain Von Rittinger's relation. However, these relations are less general and are characteristic of a material and its moisture content. In practice, these specific relations will be researched in order to characterise a plant's grinding energy consumption and deduce the expected impact of altering the feed or product particle size distribution.

The proposed method still has to be confirmed using mills with different principles and scales to that used in this experiment and with different species and biomasses. At this stage, Eq. (6) takes into account the influence of moisture content and particle size distribution on the milling energy requirements, as it was already observed by Mani [15] and Esteban [18]. Further, parameter *M* may perhaps be linked to other physical properties of the wood, such as the particle density or resilience, but it already has the advantage of characterising grindability by a single value. It also allows the effects of altering the feed moisture content, the particle size distribution, or both of these parameters on grinding energy consumption to be estimated. Lastly, it provides an initial guide to dimensioning a grinding plant's energy requirements.

4.3.3. Pellets

For the five types of pellets examined in this study, Fig. 4.9 shows the difference between the energy consumption when grinding whole pellets and when grinding pellets that had been pre-milled using a larger diameter mesh screen (8-3 mm according the milling position in the experimental planning). On average, grinding the pellets in question consumes 1.8 Wh/kg, 2.0 Wh/kg, 2.1 Wh/kg, 3.7 Wh/kg, 5.3 Wh/kg and 11.4 Wh/kg using screens with 8 mm, 6 mm, 5 mm, 4 mm, 3 mm and 2 mm mesh, respectively. On average, the same pellets previously ground using a screen with a larger diameter mesh than that used for the measurement had energy consumption values of 0.9 Wh/kg, 1.1 Wh/kg, 2.1 Wh/kg, 3.2 Wh/kg and 7.6 Wh/kg, using screen with 6 mm, 5 mm, 4 mm, 3 mm and 2 mm mesh sizes, respectively. The logical conclusion is therefore that, for the screen mesh diameters in question, milling whole pellets systematically consumes more energy than is the case with pre-milled pellets.

It is to be noted that, for the grinding passes using screens of mesh diameter 5 mm, 6 mm and 8 mm the recorded energy consumption was stable irrespective of the screen concerned. Moreover, the levels were low, in the order of 2 Wh/kg, compared with the values when grinding biomass that had not been pre-densified (cf. Section 4.3.1) and also compared with grinding pellets using screens of smaller mesh diameter (2 mm, 3 mm and 4 mm). It is therefore assumed

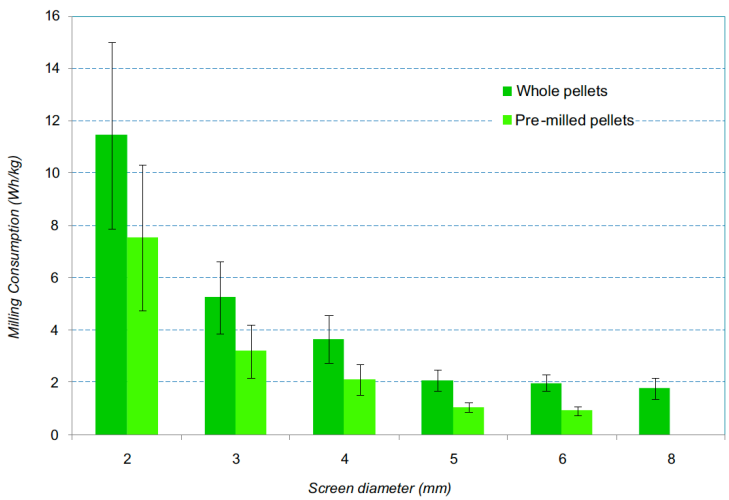

Fig. 4.9. *Energy consumption when milling pellets and their component material on 2-8 mm diameter mesh screens.*

that grinding on these screens breaks down the pellet structure but only reduces the pellet ingredient particle sizes by a negligible amount.

When milling pellets using 2 mm, 3 mm and 4 mm mesh screens the average energy consumption for whole pellets and pre-milled pellets rose as the mesh diameter decreased. Moreover, the measurement variability and the energy consumption difference between the two types of material increased similarly. It is therefore assumed that grinding pellets on 2 mm, 3 mm and 4 mm diameter mesh screens not only breaks down the pellet structure but also reduces the ingredient particle sizes. It was not possible, for the pellets in question, to establish a relation between the energy needed to break down the structure and another property of the pellets (such as durability) and the latter is therefore considered to be stable for the various materials included in this study.

A model of energy consumption when milling pellets and their component material is therefore proposed in the form of Eq. (7). This model takes account of the energy needed to break down the pellet structure. The particle size reduction is expressed by Von Rittinger's parameter calculated on the basis of the particle size distribution medians of the grind (x_2) and the pellet ingredients to characterise the input material (x_1).

The model takes account of the energy needed to break down the pellets structure. This energy is to be determined for pellets and is considered as nil for pre-milled pellets. In a first step, the three parameters *a*, *b* and D_{en} pellets have been calculated to minimise the DSS between values supplied by the model and the actual measured consumption. The mean value of D_{en} pellets has been calculated as having the value of 2.5 Wh/kg. In a second step parameters a and b have been recalculated to minimise the DSS between values supplied by the model and the actual measured consumption when the energy needed to break down the pellets is set at 2.5 Wh/kg.

$$E_{1\text{-}2} = \left[a \left(\frac{1}{x_2} - \frac{1}{x_1} \right) + b \right] + D_{En} \qquad (7)$$

Table 4.6 provides parameters *a* and *b* for the afore described model. For each type of pellets, considered individually, at two moisture content levels, the relation between the two values is characterised by high R^2 levels (0.868-0.971). However, although the effect of moisture content has been shown (cf. Section 4.3.1) it has not been incorporated into the model, as was the case with wood

Table 4.6. Parameters a and b minimising the DSS between measured energy consumption and estimated energy consumption using the model, for five different pellets, each with two moisture content values.

Pellets	H	N	D_{En} pellets	*a*	b	DSS	R^2
1	0.8	16	2.50	6.05	0.79	6.5	0.883
1	11.7	16	2.50	13.54	1.75	5.8	0.971
2	1.1	16	2.50	19.15	-8.42	5.0	0.909
2	7.7	16	2.50	17.36	-2.40	19.7	0.950
3	0.6	16	2.50	13.02	-5.93	2.8	0.957
3	9.3	16	2.50	14.05	-3.49	15.8	0.916
4	0.7	16	2.50	9.76	-1.25	5.7	0.868
4	6.0	16	2.50	9.56	-0.20	6.4	0.968
5	0.2	15	2.50	9.87	-3.86	2.8	0.958
5	7.09	16	2.50	11.22	-2.47	10.4	0.919
All		159	Selected according to pellets			81.0	0.956

chips. Moisture content appears to affect each type of pellet differently. That would need to be confirmed by increasing the number of moisture content levels of the pellets milled, something that is difficult to do in practice given this fuel's low moisture content range. A global model using parameters *a* and *b* determined individually for each type of pellet produces a relation characterised by an R^2 of 0.956.

Milling of pellets with different moisture content values can thus be modelled relatively accurately, on the basis that as well as the energy needed to mill the biomass, energy is also needed to break down the pellet structure. The milling energy for a particular type of pellets is then characterised by a pair of parameters (*a* and *b*) characteristic of the pellet moisture content.

4.4. Conclusions

The following conclusions have been drawn from this study: for beech, oak, pine and spruce chips with maximum 22% moisture content, the energy needed to mill chips of these materials varies greatly and essentially depends on three factors, i.e. the material moisture content, the particle size difference between the feed and the product and the species of wood.

The study suggests that the Von Rittinger's parameter calculated using the feed and product particle size distribution medians is the most suited to expressing the energy consumed when milling wood chips in selected moisture regime. A high Von Rittinger's parameter indicates a higher consumption of energy for the milling. Moreover, this parameter is proportional to the material moisture content, i.e. increased moisture content increases the energy consumption for milling linear.

The methodology proposed in this paper enables wood chip grindability to be characterised by a single parameter, *M*. In the context of this study, $M = 5.11$ for beech, $M = 8.54$ for oak, $M = 9.65$ for pine and $M = 11.85$ for spruce. The unit associated with the constant *M* balances the units of the equation when the *x* are expressed in mm, mass in kg, moisture content in % and energy consumption in Wh/kg dry matter.

In practical wood milling operations, the here proposed model will be used to design milling facilities. Alternatively it may be used to estimate the possibility to process a new feed material (with different particle size distribution or moisture content) in an existing facility and anticipate the product output. Finally it offers the possibility to optimise the energy costs of solid biofuels supply chains, e.g. the interaction between milling and drying operations.

Concerning wood pellets, the energy needed to mill pellets is affected by the same factors as wood chip milling: moisture content, particle size difference between feed and product, and type of pellets milled. However, for comparable moisture content levels, and screens with equivalent mesh diameter, less energy is consumed when grinding pellets than chips.

When milling pellets using 5, 6 and 8 mm mesh diameter screens the energy consumption is stable around 2.0 Wh/kg, and is therefore of minor importance when planning and performing a milling operation.

For finer milling, using 2, 3 and 4 mm mesh diameter screens, the energy consumption and the energy consumption ranges increase. In this case, for each type of pellet a model can be proposed that expresses energy consumption for pellets and their component material on the basis of the pellet breakdown energy (estimated at 2.5 Wh/kg) and a Von Rittinger parameter. In practical pellets milling operations, the results suggest that the energy consumption increase dramatically when using screens with mesh diameter below 5.0 mm and this

economical parameter should be considered when planning the milling operation.

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Chapitre 5
Conclusion générale

Cette étude débute par le constat d'un écart flagrant entre les lois exprimant l'énergie nécessaire au broyage de minerais et l'état des connaissances à ce propos lorsque la biomasse est considérée. La possibilité de définir pour la biomasse, le bois en particulier, des expressions similaires à celles utilisées pour les minerais, est le point de départ de la réflexion menée dans cette thèse. En effet, les lois empiriques de Von Rittinger, de Kick et de Bond, ainsi que leurs évolutions ultérieures dues à Charles et Hukki, bien qu'anciennes sont toujours utilisées quotidiennement dans l'industrie traitant les minerais. Elles sont basées sur une relation simple expliquant la consommation énergétique du broyage d'une matière à partir de son état granulométrique de départ (le « feed » – l'alimentation) et après broyage (le « product » – le produit). Ces lois permettent de caractériser la broyabilité de chaque minerai par une valeur unique.

Les données concernant la consommation énergétique du broyage de la biomasse sont beaucoup plus rares et plus difficiles à unifier, à exploiter, à globaliser. La diversité des propriétés considérées pour caractériser la biomasse et les méthodes utilisées pour les mesurer peuvent expliquer, au moins partiellement, cette hétérogénéité. La première partie de ce travail consiste donc, grâce aux enseignements et aux pratiques de l'industrie traitant des minerais, à déterminer quelles sont les propriétés à prendre en compte pour expliquer la consommation énergétique du broyage de bois et de pellets de bois. Les facteurs d'influence communs à ces combustibles s'avèrent être la matière elle-même, ainsi que la granulométrie et l'humidité de l'alimentation et du produit. Concernant les pellets, des propriétés supplémentaires, spécifiques, semblent devoir être considérées également : la durabilité, la masse volumique nette et la granulométrie des constituants des pellets.

Ensuite, les méthodes nécessaires à la mesure des propriétés de la biomasse identifiées comme pouvant avoir une influence sur la consommation énergétique du broyage ont été sélectionnées. C'est l'objet de la seconde partie de cette thèse, elle décrit la sélection des méthodes envisageables pour mesurer les propriétés listées ci-dessus. Des tests comparatifs ont été entrepris au travers d'essais inter-laboratoires, menés en collaboration avec des équipes de recherche européennes. Ces expérimentations ont permis de sélectionner les méthodes les plus adaptées aux essais à réaliser.

La détermination rapide de l'humidité promet un gain de temps dans la caractérisation de cette propriété. Les essais pratiqués dans le cadre de cette étude ont mis en évidence que parmi les équipements testés, ceux basés sur la caractérisation du spectre proche infrarouge et les appareils de type « conteneur » basés sur la mesure de la constante diélectrique sont plus avantageux que les autres équipements testés. Ils sont plus précis et moins sensibles aux variations du combustible et aux procédures de calibration. Il apparait également que les mesures réalisées par les appareils de type « diélectriques » sont influencés par la masse volumique en vrac des combustibles. Cette propriété doit donc être prise en compte lors des calibrations. Il est par ailleurs conseillé de réaliser une calibration pour chaque combustible considéré. Ces équipements trouvent leur place en production, lorsque l'humidité est la principale source de variation de l'approvisionnement, les autres propriétés variant peu (essence, granulométrie, masse volumique en vrac…). Ces équipements, NIR en particulier, présentent alors l'avantage

de permettre la multiplication des mesures, le suivi en ligne de l'approvisionnement et donc une maîtrise plus exacte de cette caractéristique de la matière. Par contre, ces appareils de mesure restent difficilement envisageables pour de faibles quantités de matières volontairement (très) différentes, caractéristiques de l'expérimentation.

En pratique, la mesure de la granulométrie s'effectue principalement à l'aide de tamis horizontaux. Il a été montré que ces mesures sont très répétables et reproductibles, quel que soit l'équipement utilisé pour générer les vibrations des tamis. Dans ce cas, les particules sont séparées selon une seule de leur dimension : la largeur. Depuis quelques années, la granulométrie se détermine aussi par analyse d'image, ces appareils permettent de déterminer précisément et simultanément plusieurs paramètres caractéristiques de chaque particule constituant l'échantillon. Des paramètres calculés, comme les coefficients de forme, peuvent donc être envisagés, ce qui ouvre des possibilités supplémentaires pour la caractérisation granulométrique. Malgré les multiples avantages du principe, les équipements de mesure de la granulométrie par analyse d'image restent relativement peu disponibles. Probablement à cause de leur prix largement supérieur à celui des équipements nécessaires au tamisage. De plus, la détermination de la granulométrie par analyse d'image ne fait encore l'objet d'aucune norme et la relation entre les mesures par tamisage et celles par analyse d'image n'ont encore été que très peu étudiées. Il semble donc que le tamisage ait encore de beaux jours devant lui, malgré les limites de ce système de classification.

La mesure de la masse volumique nette se heurte généralement aux imprécisions dues à l'estimation du volume de particules de forme irrégulière. La mesure manuelle des dimensions s'avère imprécise et conduit à préférer l'estimation du volume par déplacement de liquides, ou de solides. Les équipements relatifs à cette dernière alternative sont rares et peu disponibles. La mesure du volume d'un matériau hygroscopique par déplacement de liquide semble problématique et tend à préconiser l'utilisation d'un imperméabilisant (la paraffine) pour éviter l'absorption d'eau par l'échantillon et sa désagrégation. Le faible volume des pellets impose alors une correction du volume mesuré, pour éliminer l'influence de l'accroissement de volume dû à la paraffine. Les essais réalisés montrent que l'ajout d'un agent mouillant dans l'eau utilisée pour réaliser les mesures améliore leur précision. De plus, il apparait qu'il est tout aussi précis de mesurer le volume d'un pellet en le plongeant dans de l'eau additionnée d'un agent mouillant, qu'il soit paraffiné ou pas. La variabilité des résultats est influencée par le niveau de masse volumique nette.

La mesure de la durabilité des pellets nécessite de les soumettre à des chocs répétés et contrôlés, dans le but de les endommager et d'évaluer leur résistance. Les chocs peuvent être générés de 2 manières : dans un tambour tournant ou dans une enceinte fixe où est généré un fort flux d'air turbulent. Après comparaison il apparait qu'un tambour tournant permet d'atteindre une meilleure répétabilité et reproductibilité. La précision des méthodes est influencée par le type de pellets testé et par ses propriétés. Il a également été montré qu'il n'existe pas de relation claire entre les résultats obtenus par les deux méthodes et que durabilité et masse volumique nette ne sont pas liées.

La granulométrie des particules constituant les pellets s'obtient en les désagrégeant dans l'eau, avant de les sécher et de les tamiser en suivant la méthode classique pour les biocombustibles solides. Pour éviter une surestimation de la taille de ces particules (suite à une désagrégation incomplète) il est nécessaire d'utiliser une eau liquide à une température proche du point d'ébullition et d'agiter le mélange ainsi obtenu. Par ailleurs,

un broyage en laboratoire utilisant une grille similaire à celle utilisée en industrie conduit à une granulométrie plus fine que celle obtenue par désagrégation, ce qui indique que les particules subissent une ultime réduction granulométrique lors de leur utilisation en centrale électrique.

La troisième partie de cette thèse est consacrée à l'évaluation de l'opportunité d'appliquer les lois issues de l'industrie traitant des minerais au broyage du bois et des pellets de bois. Les propriétés des matières considérées sont mesurées à l'aide des méthodes sélectionnées en amont.

Les essais sur le bois ont été réalisés avec des plaquettes de Hêtre, de Chêne, de Pin et d'Epicéa, d'une humidité maximale de 22 %. Ils confirment que l'énergie nécessaire au broyage des plaquettes de ces matières est très variable et dépend principalement de trois facteurs : l'humidité de la matière, la différence de granulométrie entre l'alimentation et le produit et, enfin, l'essence de bois broyée. Il apparaît également que le paramètre de Von Rittinger calculé en utilisant les médianes des distributions granulométriques de l'alimentation et du broyat s'avère le mieux adapté à l'expression de l'énergie nécessaire au broyage de bois. Plus ce paramètre est élevé, plus le broyage est énergivore. De plus, ce paramètre est proportionnel à l'humidité de la matière.

La méthodologie proposée dans cette étude, permet de caractériser la broyabilité des plaquettes de bois à l'aide du seul paramètre M. Dans les limites de cette étude, M = 5,11 pour le Hêtre, M = 8,54 pour le Chêne, M = 9,65 pour le Pin et M = 11,85 pour l'Epicéa. L'unité associée à la constante M équilibre les unités de l'équation lorsque les médianes des distributions granulométriques sont exprimées en mm, la masse en kg, l'humidité en % et l'énergie consommée en Wh/kg de matière sèche.

L'énergie nécessaire au broyage des pellets répond aux mêmes facteurs qui influencent le broyage des plaquettes : l'humidité, une différence de granulométrie entre matière entrante et produit broyé ainsi que le type de pellets broyés. Cependant, pour des humidités comparables, et des grilles aux mailles de diamètres équivalents, le broyage des pellets est moins énergivore que celui des plaquettes. De plus, il apparait que l'énergie nécessaire au broyage des pellets indemnes est supérieure à celle nécessaire au broyage de la matière qui les constitue. Lorsque des pellets sont broyés sur des grilles aux mailles de diamètre de 5, 6 et 8 mm, l'énergie nécessaire est stable et est de l'ordre de 2,0 Wh/kg. Pour des broyages plus fins, sur des grilles aux mailles de diamètre de 2, 3 et 4 mm, les consommations énergétiques et leur étendue augmentent. Dans ce cas, il est possible, pour chaque type de pellets, de proposer un modèle exprimant la consommation énergétique des pellets et de la matière qui le constitue sur base d'une énergie de déstructuration des pellets (estimée à 2,5 Wh/kg) et d'un paramètre de Von Rittinger. Ce paramètre est calculé en considérant la médiane de la distribution granulométrique du produit et la médiane de la granulométrie des constituants des pellets. Les paramètres de ce modèle sont caractéristiques de l'humidité à laquelle les pellets sont broyés. L'hypothèse de l'influence de la durabilité et de la masse volumique nette des pellets sur l'énergie nécessaire pour les broyer n'a pu être confirmée.

Cependant, il faut noter qu'à la mesure de chaque propriété, est associée une source de variation (caractérisée par les répétabilités et reproductibilités). Or, le modèle prend en compte plusieurs propriétés de la matière. Ainsi la multiplication des variabilités associées à chacun des paramètres pris en compte influence la précision du modèle global.

Les méthodes de mesure de la durabilité, de la masse volumique nette et de la distribution granulométrique des constituants des pellets, préconisées par ce travail, ont

déjà été reprises dans les travaux du comité technique 235 (Biocombustibles solides) du comité européen de normalisation (CEN TC 335 – Solid Bio fuels) et sont retranscrites dans les normes européennes. Depuis peu, elles servent de base à la rédaction de normes ISO (International Standard Organisation) dans le cadre des travaux de son comité technique 238. Mais les normes sont appelées à évoluer et demain, la démocratisation des récentes évolutions techniques notamment en matière d'analyse d'image et de spectrométrie infrarouge offriront probablement plus de précision, un gain de temps, une facilité d'utilisation et un éventail de paramètres mesurés plus large. À titre d'exemple, la médiane des distributions granulométriques obtenue par tamisage permet d'expliquer la consommation énergétique du broyage. L'analyse d'image, outre la facilité de mesure qu'elle apporte, offre une nouvelle gamme de paramètres mesurables qui permettront de caractériser le broyage par des paramètres qualitatifs : état de surface, prise en compte de deux (ou trois) dimensions de la particule plutôt que de la seule largeur.

La modélisation du broyage du bois permettra aussi dans un avenir proche de rédiger une méthode de mesure de la broyabilité du bois et d'autres biomasses notamment à destination énergétique comme le bambou ou le Miscanthus. En effet, des observations restent nécessaires pour caractériser la broyabilité de ces biocombustibles. Pour ce faire, les procédures appliquées lors des essais réalisés ici devront probablement être simplifiées, au risque d'une diminution de la précision par rapport à la méthode de référence, afin d'être utilisables à des coûts raisonnables dans la pratique et permettre l'acquisition de ces données dans des conditions standardisées. Des essais très récents démontrent que le broyeur utilisé influence la valeur de la constante de broyabilité. Néanmoins, la possibilité d'utiliser le paramètre de Von Rittinger pour expliquer la consommation énergétique d'une matière est confirmée, ainsi que le lien de ce paramètre avec l'humidité. Le choix d'une méthode de mesure impliquera donc la définition du broyeur à utiliser. En effet, tous les broyeurs ne sont pas adaptés à la réalisation de tests de broyabilité. Celui qui sera sélectionné, à l'image de celui utilisé dans cette étude, sera de faible puissance, ne nécessitera pas de ventilation autre que celle qu'il génère lui-même et sera capable de broyer complètement l'échantillon, ce qui n'est pas le cas de la totalité des broyeurs de laboratoire disponibles dans le commerce. Des essais inter-laboratoires pourront alors être entrepris afin de caractériser les répétabilités et les reproductibilités de la méthode décrite. Il sera dès lors possible de constituer une base de données reprenant la broyabilité des différents biocombustibles solides.

Disposer d'une méthode de mesure normalisée permet d'abord de caractériser la broyabilité de diverses biomasses en conditions standardisées. Elle est ensuite une référence qui permet de comparer entre eux différents systèmes de broyage (vis, disque, marteaux…) différentes conception de broyeurs, différents outils de coupe ou marteaux, pour juger de leur efficacité énergétique.

Les observations, les hypothèses et le modèle proposés par cette étude devront être confirmés et étendus. En effet, la gamme considérée par ce travail est relativement restreinte. Les 4 essences tempérées et les 5 pellets de bois, sont certes représentatifs du marché wallon, belge et peut-être européen, mais qu'en sera-t-il d'autres essences de bois (par exemple tropicales), d'autres biomasses plus filandreuses (du chanvre ou du lin) ou de productions agricoles classiques (paille, maïs, fétuque…).

L'humidité des échantillons traités par les essais décrits ici était comprise entre 0 et 22,4 %. Comment réagira le modèle avec des matières plus humides, ou ayant subi un traitement thermique plus sévère que celui

de l'étuve de séchage, comme la pyrolyse à basse température par exemple ? Un des avantages souvent mis en avant à propos de la torréfaction est son impact sur la diminution énergétique du broyage de la matière. Les broyabilités de ces matières pourraient-elles être exprimées à partir de ce modèle en considérant par exemple des humidités négatives ? C'est-à-dire des matières ayant atteint, lors de leur stabilisation en masse, du fait de la température plus haute, une masse inférieure à celle qui aurait été obtenue par un séchage à 105 °C.

La gamme de granulométrie pourrait également être étendue. Les matières considérées par cette étude sont caractérisées par des médianes de distribution, obtenues par tamisage, de 0,40 mm à 7,38 mm. Le modèle conservera-t-il sa validité en dehors de cette gamme ? Pourra-t-il être étendu ? La méthode proposée restera-t-elle valide ou devra-t-elle être adaptée ? Les distributions granulométriques des matières utilisées dans l'expérimentation relative au broyage sont décrites par leur médiane. L'impact de l'utilisation d'autres quantiles (par exemple les déciles 80 ou 90) sur la valeur du paramètre M et sur la qualité des relations constituant le modèle global pourrait être investigué également.

Très probablement, les principes fondamentaux du modèle mis en évidence à l'aide d'un broyeur de laboratoire s'appliqueront aussi aux broyeurs industriels. La relation entre ces deux échelles reste à déterminer, pour les broyeurs industriels qui seront considérés. La difficulté sera alors de surmonter l'aspect expérimental en industrie. D'abord, les possibilités d'expérimentation y sont rares, puis la logistique associée à ces essais rend souvent illusoire la mise en place d'un plan expérimental satisfaisant. L'analyse se base donc généralement sur des mesures réalisées en production, celle-ci étant caractérisée par des conditions volontairement stables. Cependant lorsque sera établie la relation entre les résultats de broyabilité obtenus en laboratoire et les consommations observées en conditions industrielles, il deviendra possible de déterminer si une matière nouvelle peut être acceptée par une installation existante ou si une installation peut être dimensionnée sur la base de la broyabilité déterminée pour cette matière.

Enfin, le coût de l'énergie rend nécessaire l'optimisation énergétique des procédés industriels, la méthode utilisée ici pour caractériser le broyage pourrait l'être également pour d'autres étapes du conditionnement des biocombustibles solides : séchage, densification, torréfaction… Celles-ci sont, en effet, dépendantes des mêmes paramètres que le broyage. Globalement lorsque ces modélisations seront réalisées, c'est la chaîne globale de la préparation des biocombustibles qui pourra être optimisée.