Using response surface methodology as a tool for process optimization with the open-source software R: Subcritical water pretreatment of wheat straw

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Introduction

- The response surface methodology (RSM) is an efficient way of optimizing chemical processes when one or more responses are influenced by multiple variables. This methodology uses experimental data to fit a polynomial equation with mathematical techniques. To apply the RSM, a design of experiment (DOE) has first to be carried out in the range of interest of the variables for the studied response(s).
- Various software can be used to generate the DOE and calculate the RSM. One of those is R and its package "rms". The "rms" package is specific to DOE and RSM. The advantage of R is of being open-source, having access to a huge amount of statistical packages, being easy to use and to customize.
- In the present study, the optimization of subcritical water pretreatment of wheat straw (*Triticum aestivum* L.) has been assessed with a RSM using R. It has been optimized in the context of glucose production for its conversion into a cellulosic biofuels or biochemicals. For such productions, a thermic and/or chemical pretreatment of the biomass is needed to be able to enzymatically hydrolyze the cellulose to glucose. Without any biomass pretreatment, lignin prevents this hydrolysis [3].

Response surface methodology with R

- The subcritical water pretreatment of wheat straw (*Triticum aestivum* L.) has been realized with a 1.3 liters batch reactor (4540 reactor of Parr).
- The cellulose, hemicelluloses and lignin content of the solid residue after pretreatment has been estimated based on the Van Soest method.
- A rotatable central composite design has been chosen as DOE to have a RSM with isovariance of the predicted response, and a second-order polynomial quadratic equation made of an intercept, a linear, a quadratic and an interaction component.

Response surface: Solid residue after subcritical water pretreatment of wheat straw

- **R script for cellulose**

→ Generate design of experiment (DOE):

doe <- ccd(2, n0 = c(4,0), alpha="rotatable") # 2 → 2 variables # n0 = c(4,0) → 4 replicates of the central point

→ Generate response surface (RSM) and statistical analysis of the response surface:

rsm.cel <- rsm(formula = Cellulose ~ SO(Time, Temperature), data = doe) # SO → Second order polynomial equation

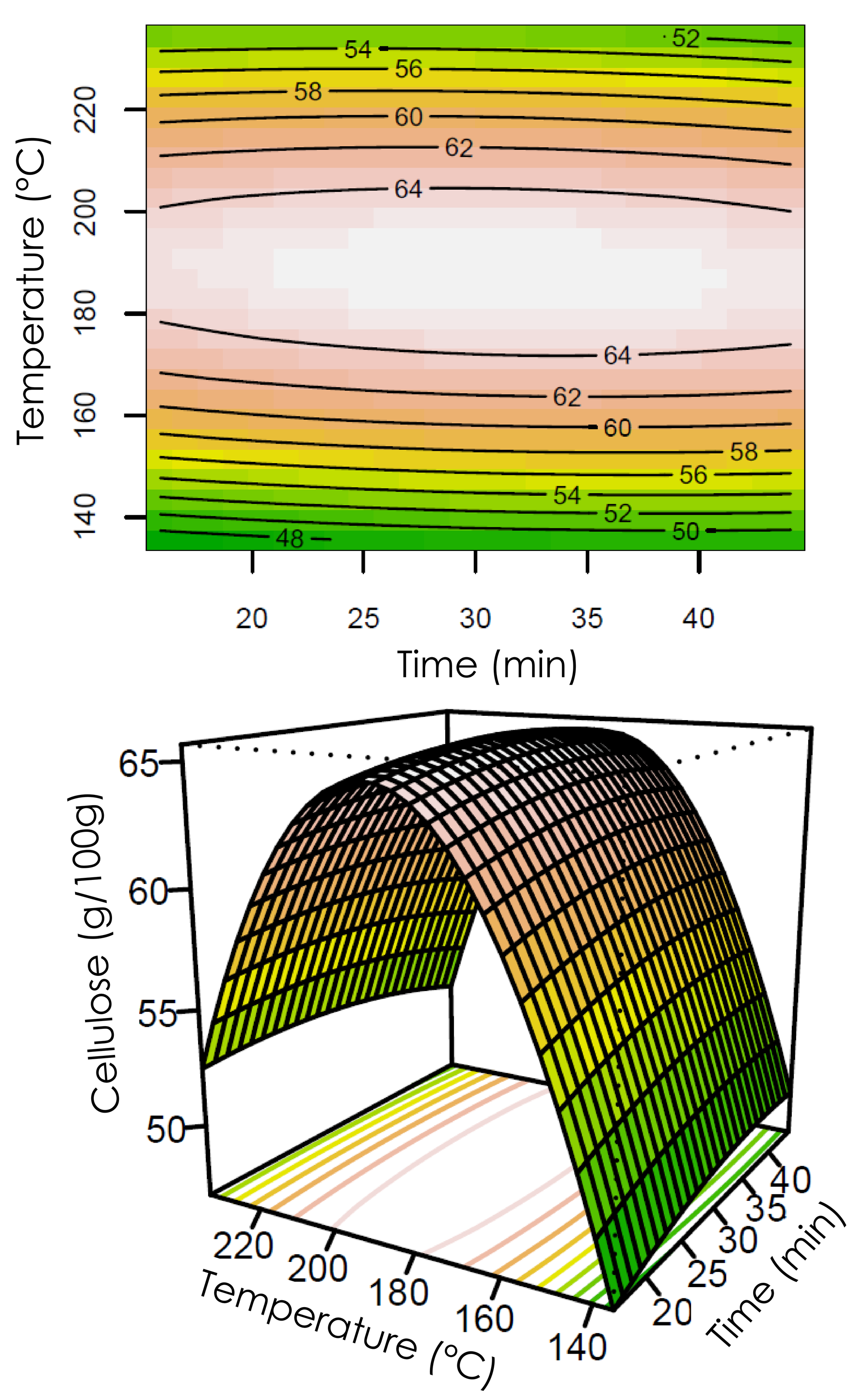
summary(rsm.cel) # Value of the coefficients of the RSM and their significance

→ Generate plots (2D and 3D):

contour(rsm.cel, ~ Time + Temperature, atpos = 3, main="Cellulose (g/100g)", image = TRUE, img.col = terrain.colors(40), xlabs = c("Temperature (°C)", "Time (min)", "Time..min.", "Temperature...C."))

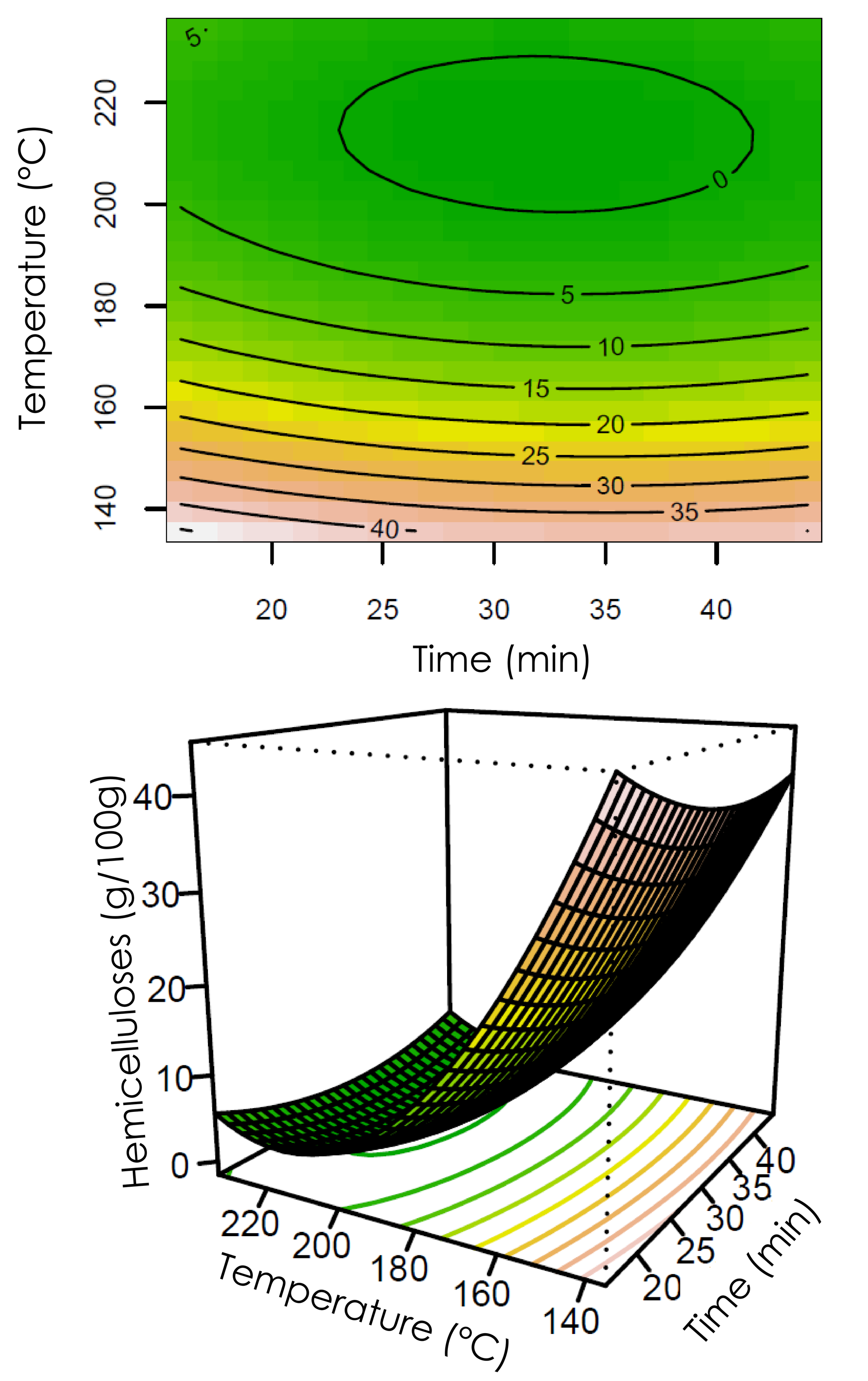
persp(rsm.cel, ~ Time + Temperature, atpos = 0, theta = -60, phi = 15, zlab="Cellulose (g/100g)", main="Cellulose (g/100g)", contour="colors", col = terrain.colors(40), xlabs = c("Temperature (°C)", "Time (min)", "Time..min.", "Temperature...C."))

Cellulose (g/100g)



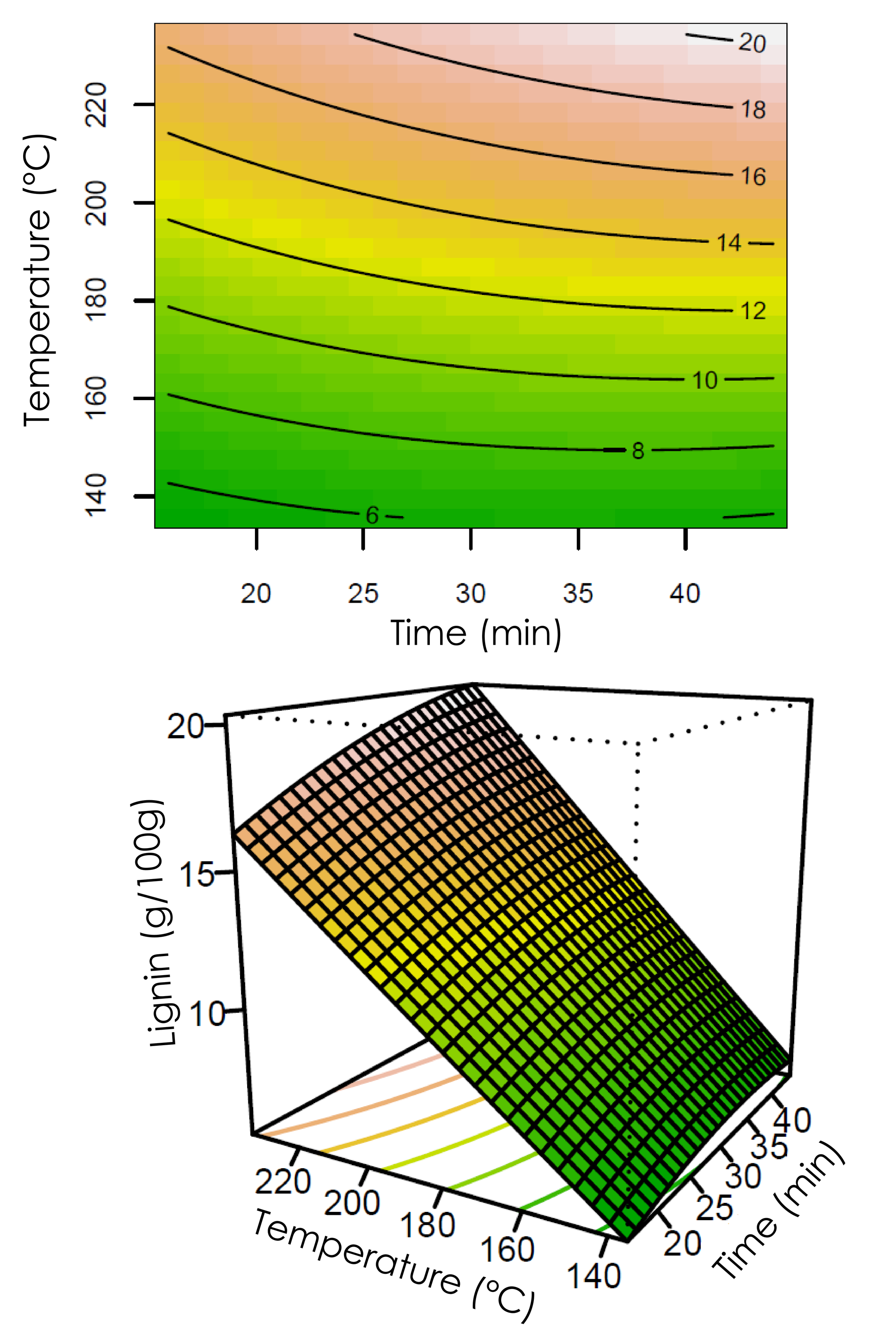
R^2=0.911 ; SEC=2.5 ; RPD=SDy/SEC=2.5

Hemicelluloses (g/100g)



R^2=0.955 ; SEC=3.8 ; RPD=SDy/SEC=3.5

Lignin (g/100g)



R^2=0.977 ; SEC=0.8 ; RPD=SDy/SEC=4.9

- **Temperature**
  → Significant effect
  → Quadratic response for cellulose and hemicelluloses
- **Time**
  → No significant effect

- **Cellulose**
  → Content increase until 190°C because of hemicelluloses degradation
  → Degradation above 190°C
- **Hemicelluloses**
  → Complete degradation above 210°C
- **Lignin**
  → Content increase because of hemicelluloses degradation and also cellulose from 190°C

Conclusions

- Temperature is an important parameter to optimize and control for the subcritical water pretreatment of fibrous biomass. For wheat straw, the maximum cellulose content after pretreatment is obtained at 190°C of subcritical water pretreatment. This temperature of pretreatment will also enable to have a high yield of enzymatic hydrolysis to get glucose for the production of cellulosic biofuels or chemicals.

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