# Dilute Acid Hydrolysis of Spent Coffee Grounds at Mild Conditions: A Response Surface Methodology Approach

Panagiota Tsafrakidou[1], Athanasia Goula[1], Ioannis Mourtzinos[1], Irene Pieria[1], Charilaos Voulgaridis[1], George Menexes[1], Costas G. Biliaderis[1], Alexandra-Maria Michaelidou[1*]

[1]Faculty of Agriculture, Aristotle University, Thessaloniki, Greece
*corresponding author: amichail@agro.auth.gr

## INTRODUCTION

More than 6 million tons of spent coffee grounds (SCG) are produced annually during coffee beverage preparation. Up to date, they are being discarded in landfills due to the lack of efficient waste management methods. As a lignocellulosic waste stream, SCG could serve as an excellent sustainable raw material in biotechnological processes for the production of added-value chemicals/commodities.

## OBJECTIVES

The present study examines the potential for sugars recovery from SCG, as a stepping stone to explore the use of this biomass in biotechnological processes. Response surface methodology was used to develop a prediction model and identify the optimum conditions for the recovery of fermentable sugars.

## METHOD / DESIGN

The experiments were conducted by employing dilute acid hydrolysis under mild conditions (temperature below 100 °C, ambient pressure) in order to prevent the acid mediated thermal decomposition of monomeric sugars (intermolecular dehydration and polymerization processes).

A central composite design was employed to evaluate the four selected variables, for the determination of the optimum dilute acid hydrolysis conditions, in order to maximize the total sugars yield (**Table**). The statistical software package of Design Expert (Release 6.0.8) was used.

**Table.** Experimental values and coded levels of the independent variables used for the central composite of the present research experiment

| Independent variable | Levels | | | | |
|---|---|---|---|---|---|
| | **−2** | **−1** | **0** | **+1** | **+2** |
| **Reaction time** (min), $X_1$ | 60 | 90 | 120 | 150 | 180 |
| **Liquid/solid ratio** (L/S ratio, mL/g), $X_2$ | 5 | 7.5 | 10 | 12.5 | 15 |
| **Temperature** (°C), $X_3$ | 60 | 70 | 80 | 90 | 100 |
| **$H_2SO_4$ concentration** (C $H_2SO_4$, %v/w), $X_4$ | 0.5 | 1 | 2 | 3 | 4 |

Sugar concentration was determined as glucose equivalent spectrophotometrically using the phenol-sulfuric method [1] and the total sugar yield ($Y_{TS}$) was calculated according to the equation:

$$Y_{TS} = \frac{C_{TS} V_H}{m_{dSCG}} \times 100$$

*$C_{TS}$: total sugar concentration obtained from the calibration curve, VH: hydrolysate volume, $m_{dSCG}$: mass of the initial dried SCG before hydrolysis [2]*

## RESULTS & DISCUSSION

Regression analysis was used to identify the influence of the independent variables on the sugars yield. In the final model, the non-significant factors ($p > 0.05$, **Fig. 1**) were excluded. The following regression equation was obtained:

$$\mathbf{Y_{TS} = -51.27 + 0.05X_1 + 3.37X_2 + 0.43X_3 + 2.12X_4 - 0.16X_2^2}$$

*$X_1$ :Time, $X_2$: L/S ratio,*
*$X_3$: Temperature, $X_4$: $H_2SO_4$ concentration*

**Determination coefficient ($R^2$)=0.816 ($p < 0.001$)**

The effect of each parameter on the total sugar yield obtained is presented in **Fig. 2**. According to the analysis, maximization of the sugar yield can be obtained at 149.73 (~150) min, an L/S ratio of 10.25, 90°C, and 3% $H_2SO_4$.

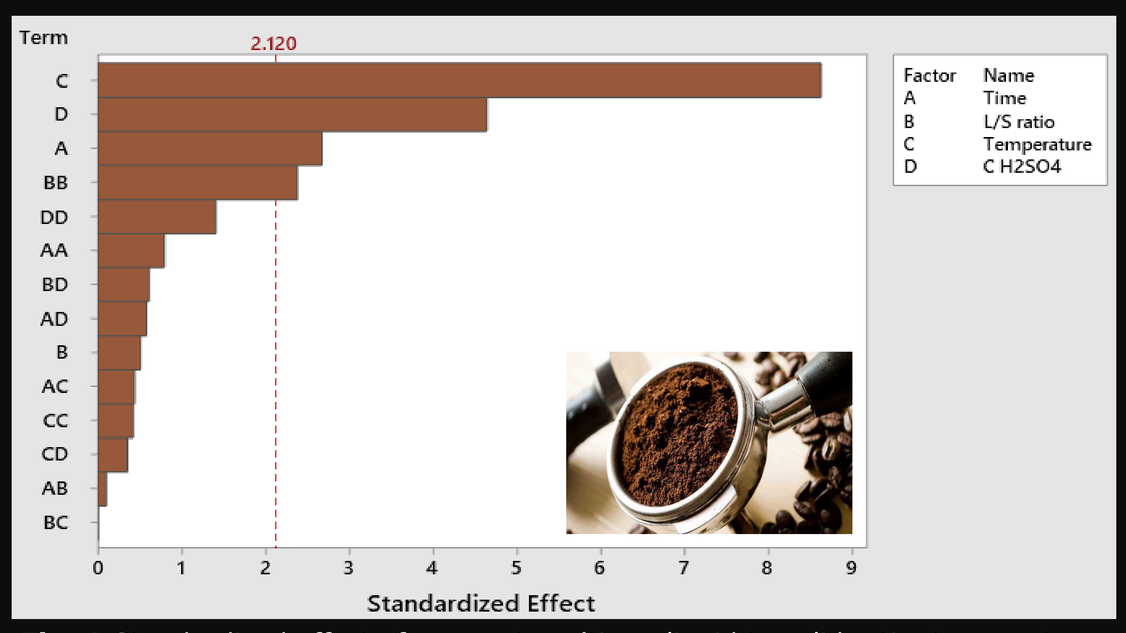


**Fig. 1** Standardized effect of parameters (time, liquid to solid ratio, temperature, on total sugar yield ($Y_{TS}$, $g/100_{gSCG}$)

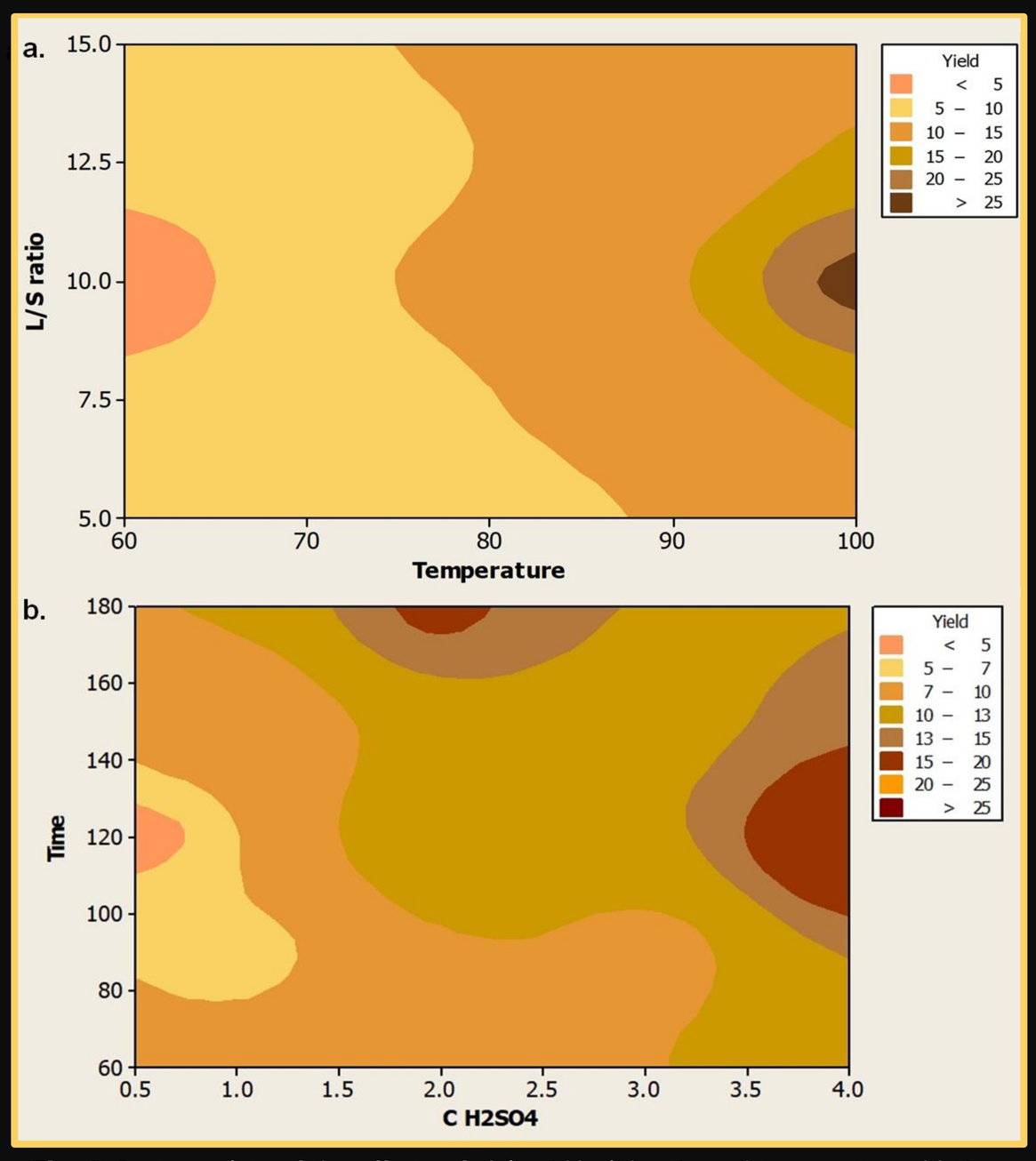


**Fig. 2** Contour plots of the effects of a) liquid/solid ratio and temperature, b) time and $H_2SO_4$ concentration on total sugar yield ($g/100g_{SCG}$)

## CONCLUSION

- Mild conditions for SCG acid hydrolysis pretreatment proved to be an efficient method for the extraction of hemicellulose type carbohydrates (soluble sugars).
- Response Surface Methodology (RSM) was successfully implemented to optimize the conditions of the process and allowed the rapid screening for a large experimental domain.
- Under the optimal conditions of this study, hydrolysis efficiency reached 73.88% expressed in glucose equivalents, corresponding to an average of 0.1876 $g_{gluc.eq}/g_{scg}$ yield.
- The optimized process points to new possibilities for SCG's biotechnological valorization through the production of several chemical compounds by chemical and/or fermentation processes.

**REFERENCES**

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