EXPERIMENTAL DESIGN TO OPTIMIZE YIELD IN THE ULTRASOUND ASSISTED EXTRACTION OF HEATHER (*Calluna vulgaris*)

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INTRODUCTION

Heather (*Calluna vulgaris*) is an excellent feedstock for natural extracts production with a wide range of biological properties such as anticancer and antimicrobial activities [1]. Heather is also rich in phenolic acids which are well known for their antioxidant capacity and their stable radical intermediates can prevent the oxidation of many food ingredients especially fatty acids and oils [2].

The use of ultrasound assisted extraction (UAE) is an economical alternative to traditional extraction processes which is an industry demand for a sustainable development [3]. The major advantage of UAE is the reduction of processing time in addition to other benefits such as lower operational temperatures and lower requirement of solvents [4].

The target of this work was to ascertain the optimum conditions of heather UAE which maximize extraction yield, using ethyl acetate as extractive solvent. The effect of variables such as the mass:solvent ratio and the extraction time was investigated to evaluate their influence on the overall yield using a central composite design (CCD) 2^k with two central and two axial points.

MATERIAL AND METHODS

C. Vulgaris dried and grinded
d_p = 0.5mm

Fixed variables
Solvent: Ethyl Acetate
T(℃): 45

Ultrasound bath
From JP Selecta, S.A., Barcelona, Spain

Table 1. CCD for Ultrasound Assisted Extraction of Heather *Calluna vulgaris*

Experiment	Code	Ratio	Time
1	0	10	30
2	0	10	30
3	0	10	9
4	-1	5	45
5	(-α)	3	30
6	-1	5	15
7	1	15	45
8	1	15	15
9	0	10	51
10	(+α)	17	30

RESULTS

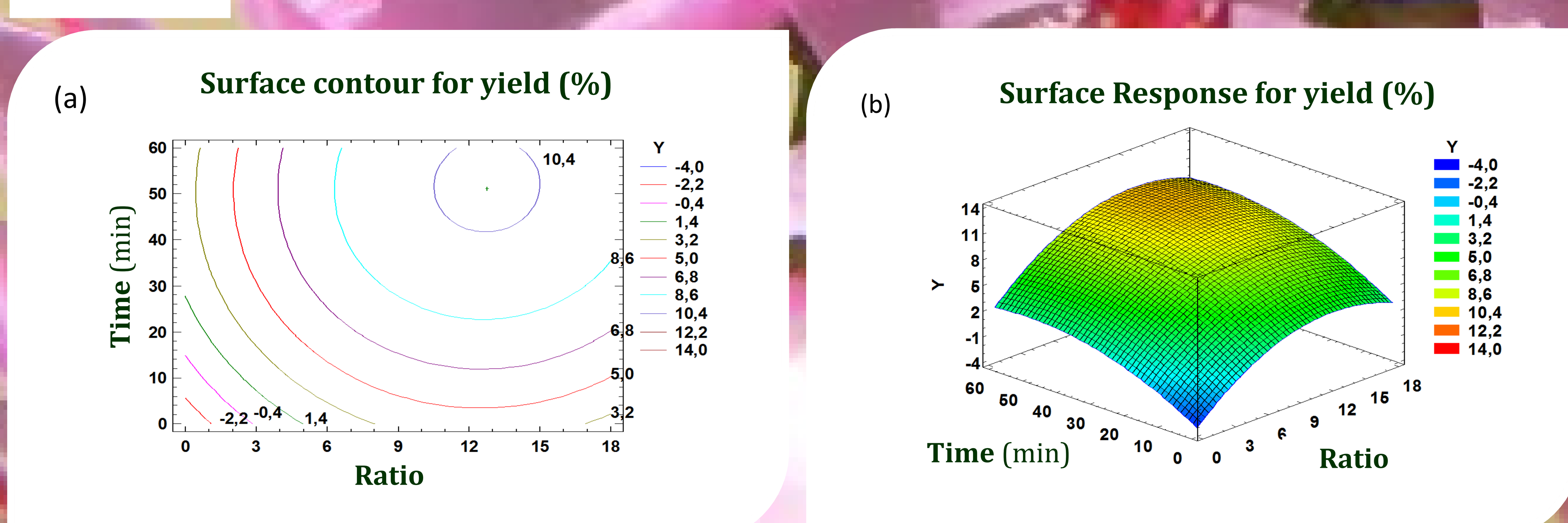

Figure 1. Contour (a) and Response Surface plot (b) for Heather UAE: dependence of yield on time and solvent ratio.

Table 2. Analysis of Variance ANOVA

Variable	Square sum	FG	Mean square	F-Reason	*p*-value
A:Ratio	13.545	1	13.545	283.72	0.0378
B:Time	19.425	1	19.425	406.88	0.0315
AA	7.0121	1	7.0121	146.88	0.0524
AB	0.0066	1	0.0066	0.14	0.7740
BB	1.338	1	1.3380	28.02	0.1190
Error	0.0477	1	0.0477		
Total (corr.)	52.015	9			

Regression Model

$$Y(\%) = -3.594 + 1.251r + 0.242t - 0.05r^2 + 0.0005r \cdot t - 0.002t^2 \qquad R^2 = 0.77$$

(*r:* mass:solvent ratio; *t: time*)

CONCLUSIONS

➔ The best conditions were found to be an extraction time of 15 min and mass:solvent ratio of 1:10, obtaining a mean yield of 9% (R^2 = 0.77). Time (*p= 0.0032*) and mass:solvent ratio (*p=0.0038*) factors have significance over the yield.

➔ The maximum yield was found at 50 min and mass:solvent ratio of 1:10 (10%) although extraction time had no significance over the maximum yield.

➔ The statistic R^2 shows that the model explains 77% of variability in the experimental yields obtained, this value means there are another variables not controlled that have and effect on the yield extraction, i.e. homogeneous particle size distribution or heat transfer effects.

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