A COMPUTER SIMULATION OF RESPONSE SURFACE METHODOLOGY FOR OPTIMIZATION OF ULTRASOUND-ASSISTED EXTRACTION OF OLIVE LEAVES

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Abstract

Modelling by computers is preferred in lots of study fields, since it reduces waste of material, labour and time. It also sends away people from the danger in the experiments or physical studies. Apart from those, its supporting the determination of experiments and results are continuing properly can be said as another advantage. According to the study field and science discipline, there are many various modelling techniques and methods. In this paper, a study made in chemical engineering discipline is modelled by the help of mathematics, statistics and computer science.

Keywords : Response Surface Methodology, extraction, modelling

1. Introduction

Natural plant extracts are getting high added value in the world. There is a susceptibility more than before in demand on natural products in food, pharmaceutical and cosmetic industries. It is the consequence of consumers growing care about health and environment (1-3). This is mostly attributed to its polyphenolic content and its antioxidant activity. In this paper, several experiments carried out in order to increase the amount of phenolic content in olive leaf extract obtained by ultrasound-assisted extraction. Additionally, the experimental results were modelled with Response Surface Methodology (RSM). During this modelling a simulation in MATLAB simulation program was constituted by using least squares method. The effects of the parameters to phenolic content were calculated and formulated, respectively.

2. Extraction Process

An important process of chemical engineering is revealing the phenolic content of natural plant extracts. The more phenolic material in a plant extract, the more industrial value. There are many parameters that can affect the extraction process. Plant material, extraction method, preliminary preparations, solvent type, solid to solvent ratio, extraction temperature and

pressure, extraction time and pH are some of these parameters. A lot of researchers study the effects of these parameters on the extract. That's why, experiments with two, three, four or more parameters and different conditions were made.

In this study, as an agricultural and industrial waste of olive oil and table oil productions, olive tree (*Olea europaea*) leaves were used as the material. The purpose of the study is to investigate the extraction parameters such as time (20, 40, 60 min), temperature (25, 35, 45 ℃) and pH (3, 7, 11) and to observe the total phenolic content with different combinations of these parameters. Totaly, 27 experiments were performed. The results are given below.

Experiment No	Time(min)	T(℃)	pH	Total phenolic content (mg-GAE/g-dried leaf)
1	60	25	3	28.37525361
2	90	25	3	30.85166135
3	120	25	3	33.42621939
4	60	25	7	20.22347134
5	90	25	7	23.09649878
6	120	25	7	24.44219842
7	60	25	11	8.686059414
8	90	25	11	10.36635493
9	120	25	11	12.07093217
10	60	35	3	25.84592294
11	90	35	3	27.0173689
12	120	35	3	29.48776513
13	60	35	7	18.22195617
14	90	35	7	18.94426521
15	120	35	7	22.11482171
16	60	35	11	9.615632406
17	90	35	11	10.13668612
18	120	35	11	11.78750117
19	60	45	3	27.22277895
20	90	45	3	28.52836874
21	120	45	3	30.80288682
22	60	45	7	20.09370254
23	90	45	7	22.09506734
24	120	45	7	23.09952589
25	60	45	11	14.26655863
26	90	45	11	15.67107964
27	120	45	11	17.23937872

3. Response Surface Methodology

Response Surface Method (RSM) is an experimental optimization procedure based on physical experiments or computer experiments (simulations) [4,5] and experimented observations. It was first developed in 1951 by G.E.P. Box and K.B. Wilson [6].

In most of the experiment investigations, there are many parameters but in many methods the effects of parameters to each other are ignored and and only the effects of distinct parameter to the results are calculated. However, parameters usually affect each other especially in physical experiments. For example if the parameters are density and temperature, handling only the distinct effect cannot be enough because temperature can also affect density. Thus, the more suitable standpoint investigates the effect of these parameters to each other and to results together. RSM is a method that aims to remove this lack.

It is very important to fomulate the effects of parameters to the result by the experiments and to calculate the conditions that renders the phenolic content maximum. When this can be done, the material can be used in industrial applications easily.

In Response Surface Method a function that is tried to be obtained for prediction. This function's general formula is

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i<j}^{k} \beta_i X_i X_j + \sum_{i=i}^{k} \beta_{ii} X_i^2 + \cdots$$

where Y represents the result (or with the name in RSM – response), $\beta_i,\ \forall i$ are regression coefficients, , $X_i,\ \forall i$ are the independent parameters.

In this study, three independent parameters were used. Those were X_1 (time, min), X_2 (temperature, ℃) and X_3 (pH). The result named response in this method is Y (mg GAE/g dried leaf). Thus, the function of this study containing three independent variables is expressed with equation below :

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3$$

When least squares method, RSM and MATLAB simulation are applied to these experiment results, the coefficients of this equation are calculated as

$$\beta_0 = 66.1766, \beta_1 = -0.0757,\ \beta_2 = -1.7392,\ \ \beta_3 = -3.0641,\ \beta_{11} = 0.0012$$
$$\beta_{22} = 0.0251, \beta_{33} = -0.0333, \beta_{12} = -0.0022,\ \beta_{13} = 0.0019,\ \beta_{23} = 0.0368$$

and the expresiion of the whole mg-GAE/g-dried leaf equation as terms of time, temperature and pH becomes as

$$Y = 66.1766 + (-0.0757.x_1) + (-1.7392.x_2) + (-3.0641.x_3) + 0.0012.x_1^2 + 0.0251.x_2^2 + (-0.0333.x_3^2) + (-0.0022.x_1.x_2) + 0.0019.x_1.x_3 + 0.0368.x_2.x_3.$$

The results obtained by experiments and these RSM equations are summarized as below.

Experiment No	Time(min)	T(℃)	pH	Experimental Result	RSM Result
1	60	25	3	28.37525361	28.4721
2	90	25	3	30.85166135	30.1221
3	120	25	3	33.42621939	33.9321
4	60	25	7	20.22347134	19.0197
5	90	25	7	23.09649878	20.8977
6	120	25	7	24.44219842	24.9357
7	60	25	11	8.686059414	8.5017
8	90	25	11	10.36635493	10.6077
9	120	25	11	12.07093217	12.8437
10	60	35	3	25.84592294	25.9241
11	90	35	3	27.0173689	26.9141
12	120	35	3	29.48776513	30.0641
13	60	35	7	18.22195617	17.9437
14	90	35	7	18.94426521	19.1617
15	120	35	7	22.11482171	22.5397
16	60	35	11	9.615632406	9.5247
17	90	35	11	10.13668612	10.3437
18	120	35	11	11.78750117	10.7467
19	60	45	3	27.22277895	28.3961
20	90	45	3	28.52836874	28.7261
21	120	45	3	30.80288682	31.2161
22	60	45	7	20.09370254	20.4157
23	90	45	7	22.09506734	22.4457
24	120	45	7	23.09952589	23.9637
25	60	45	11	14.26655863	14.3137
26	90	45	11	15.67107964	15.0997
27	120	45	11	17.23937872	18.0457

4. Conclusion

As an agricultural and industrial waste, olive leaves are a potential cheap, renewable and abundant source of polyphenols. These valuable compounds in olive leaf powders and leaf extracts are responsible for many health benefits, and therefore there is a growing interest to utilize olive leaf powders or extracts in various industrial applications such as food supplements, cosmetic and pharmaceutical industries. In this study olive leaves are chosen as a research material. The phenolic content in the leaf extracts was acquired by ultrasound-assisted extraction through different time, temperature and pH conditions. Therefore, 27 experiments with different combinations of 3 time, 3 temperature and 3 pH values were carried out. After chemical experimental studies were completed, a simulation in MATLAB program was coded for applying RSM method to these results. Each parameter effect was formulated.

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