Diyala Journal of Engineering Sciences

Journal homepage: https://djes.info/index.php/djes

ISSN: 1999-8716 (Print); 2616-6909 (Online)

Yield Optimization for the Extraction of Organic Compounds from Okra Leaves Wastes

Shaimaa Abod Hussain[1], Anees A. Khadom[1*] and Hameed B. Mahood[2]

[1] Department of Chemical Engineering, University of Diyala, 32001 Diyala, Iraq
[2] Centre for Sustainable Cooling, School of Chemical Engineering, University of Birmingham, UK

ARTICLE INFO

Article history:
Received December 24, 2023
Revised February 21, 2024
Accepted February 24, 2024
Available online May 6, 2024

Keywords:
Okra
Leaves
Extraction
Yield
Optimization

ABSTRACT

An important area of research is the extraction of organic chemicals from plants and herbs. Considering the fact that the extracts have numerous commercial and pharmacological uses. Furthermore, limiting the optimal working region is made easier by employing an appropriate experimental design. Solvent extraction is the technique most frequently used to separate organic components from plants. However, the conditions of extracting solvent that is utilized greatly affects the yields of the extract and, as a result, the organic activities of the plant parts. This research deals with the extraction of solid organic compounds from the okra leaves using water as a solvent. A Soxhlet apparatus was used for the extraction process. The influence of extraction time, solvent volume, and okra powder mass on the yield percentage was optimized. Two mathematical models were suggested: second-order polynomials and power models. A higher correlation coefficient was obtained with the polynomial model. The maximum extraction yield was obtained at optimum values of 200.3 min, 29.07 g, and 290.7 ml for time, okra powder mass, and solvent volume, respectively. It is evident from mathematical formulas that the impact of time was less significant than the effects of solvent volume and powder mass. On the other hand, the power model and the second-order quadratic interaction model had correlation coefficients of 0.4849 and 0.9707, respectively.

1. Introduction

Fruits and vegetables contain a variety of antioxidants and organic chemicals [1]. These chemicals can be used pharmacologically to treat diseases like cancer and other dangerous conditions [2]. A wide variety of bioactive chemicals can be produced by plant parts, such as leaves, flowers, seeds, stems, etc. Fruits and vegetables contain significant amounts of phytochemicals, which may offer protection against free radical damage [3]. Because they function as natural antioxidants, plants that contain advantageous phytochemicals may help the body meet its requirements [4]. Industrially, plant extracts can be used successfully as an alternative source for corrosion [5, 6].

Organic compounds are usually recovered from plants using a variety of extraction methods, such as soxhlet, heating under reflux, ultrasound extraction, microwave extraction, etc., which take into consideration their chemistry and uneven distribution within the plant tissue. For instance, the outer tissues of fruits and grains have larger quantities of soluble phenolics than the inner tissues [7]. The method most commonly employed to isolate organic components from plants is solvent

* Corresponding author.
E-mail address: draneesabdullah@uodiyala.edu.iq
DOI: 10.24237/djes.2024.17102

extraction. Nonetheless, the type of extracting solvent used has a significant impact on the extract yields and consequent organic activities of the plant components [8]. Water, ethyl acetate, acetone, cyclohexane, and methanol could be used to achieve the extraction process. Water is better than organic solvents in terms of both health and the environment because organic solvents can have detrimental effects on both [9, 10]. Several process parameters, such as time, temperature, and the ratio of solvent to material, are key factors influencing the extraction yield. Generally, the extraction yield will increase as the temperature, the time, and the solvent-to-material ratio increase. However, the decomposition of the heat-sensitive compounds is affected by raising the extraction temperature. Meanwhile, long extraction times are unnecessary since the maximum extraction rate is obtained in just a few minutes. In other words, the extra time could result in the degradation of active compounds [11, 12]. When solvent volume is increased, the extraction yield typically rises in relation to the solvent-to-material ratio. Therefore, the optimization of process parameters and their influence on the yield of the extraction process are very important and play an important role. The optimization technique can be applied to formulate a linear or quadratic model between responses and independent parameters. Limited work is available regarding the optimization of the extraction of organic materials from okra leaf wastes. Which, in turn, can be used in many pharmaceutical and industrial applications.

In the present work, the effect of time, initial okra powder mass, and volume of solvent on the yield of the extraction process was evaluated. Several previous studies concentrated on the bioactivity of extracts, organic compound diagnosis, etc. Thus, the present work represents an attempt in the direction of optimization and maximization of solid material produced in comparison with initial solid charged material.

2. Experimental work

2.1 Materials and powder preparation

The fresh okra leaves are collected from a local market in Diyala province, Iraq. The leaves were washed to remove suspended objects, soil, and dust. This was done by submerging the leaves with tap water inside clean plastic tubs. Then, the leaves were dried and washed again with distilled water. In the next stage, the leaves were dried with a clean cloth and left in a shadow for two weeks at room temperature. After drying, the leaves were ground using an electrical grinder, and then the powder was sieved and prepared for the extraction process.

2.2 Soxhlet extraction

Extraction is achieved by using a conventional Soxhlet apparatus (1000 ml capacity). It consists of a distillation flask placed in a water bath, a condenser, and a thimble holder. The okra powder is packed in filter cloth and placed in a thimble holder, which is filled with the condensed water (solvent) from the distillation flask. The liquid moves through the siphon and unloads it back into the distillation flask when it reaches the overflow level. Then, it carries the extracted solute into the bulk of the liquid. In a solvent flask, the solute is separated from the solvent by filtration and evaporation. Other glasses and instrumentation, such as conical glasses, beakers, electronic balances, graduated cylinders, etc., used distilled water as a solvent. The powder was weighted and mixed with distilled water at different mixing ratios. The extraction process was carried out at different periods, depending on the completion of the extraction process. The yield was obtained by calculating the amount of solute in the solution. To achieve that, 10 ml of the extract was weighted by an electronic balance, and then the extract was weighted again after drying in an oven at 60 ^{o}C. The weight difference represents the amount of sold okra in the extracted solution, which can be used in yield calculations.

2.3 Mathematical regression

The mathematical and statistical analysis were achieved using STATISTIC software, version 10 was used. Advanced, nonlinear models based on nonlinear estimation method was used in the regression process. It is based on

the least-squares Levenberg-Marquardt estimation method. The maximum number of iterations was 1000, the convergence criterion was 1×10^{-6}, the p-level was 0.05, and the confidence intervals for parameter estimation were 95%.

3. Results and discussion

3.1 Yield of extraction process

The percentage yield (%Y) of the extraction process was evaluated at different process variables. The time of the extraction process (t), initial okra powder mass (m_p), and solvent volume (v_s) were varied in order to optimize the yield of the extraction process. The percentage yield (%Y) of the extraction process was calculated using Eq. 1.

$$\%Y = \frac{m_p^f}{m_p} \times 100 \quad (1)$$

In Eq. 1, m_p^f represents the mass of extract produced from the extraction process. Table 1 shows the %Y at different values of t, m_p, and v_s. The runs were distributed randomly at different values of process variables. Some runs were repeated in order to increase the number of equations required to evaluate the coefficients of the optimized model. Table 1 shows the %Y as a function of process variables. Some tests were repeated twice to ensure the repeatability and accuracy of the results. In addition, repeated test are also necessary in the evaluation of model coefficients since they make the degree of freedom equal to zero.

Table 1: Variation of extraction yield with time, powder mass, and solvent volume

Run	Time (min)	Powder mass (g)	Solvent volume (ml)	Y (%)
1	210	15	150	10.7
2	210	50	500	11.6
3	180	10	100	10.0
4	205	40	400	7.5
5	200	15	150	9.8
6	215	40	400	8.3
7	160	15	150	9.7
8	200	40	400	7.4
9	210	15	150	11.3
10	230	40	400	7.5
11	190	15	150	9.1

3.2 Mathematical model and optimization

The mathematical and statistical analysis represent powerful tools in the correlation of dependent and independent variables [13, 14]. In the present work, two models were suggested: the second-order quadratic interaction model and the power model. The mathematical representation of these two models is shown in Eqs. 2 and 3.

$$\%Y = a_o + a_1 t + a_2 m_p + a_3 v_s + a_{11} t^2 + a_{22} m_p^2 + a_{33} v_s^2 + a_{12} t m_p + a_{13} t v_s + a_{23} m_p v_s \quad (2)$$

$$\%Y = a_o t^{a_1} m_p^{a_2} v_s^{a_3} \quad (3)$$

In order to normalize the independent variable and understand the influence of each variable, coding was used. Independent real variables were transformed to coding once (X_1, X_2, and X_3) using Eq. 4. Then, Eqs. 2 and 3 were rewritten as shown in Eqs. 5 and 6.

$$X_1 = \sqrt{n}\left(\frac{t - t^{center}}{t^{center} - t^{\text{minimum}}}\right)$$
$$X_2 = \sqrt{n}\left(\frac{m_p - m_p^{center}}{m_p^{center} - m_p^{\text{minimum}}}\right) \quad (4)$$
$$X_3 = \sqrt{n}\left(\frac{v_s - v_s^{center}}{v_s^{center} - v_s^{\text{minimum}}}\right)$$

$$y = a_o + a_1 X_1 + a_2 X_2 + a_3 X_3 + a_{11} X_1^2 + a_{22} X_2^2 + a_{33} X_3^2 + a_{12} X_1 X_2 + a_{13} X_1 X_3 + a_{23} X_2 X_3 \quad (5)$$

$$y = a_o {X_1}^{a_1} X_2^{a_2} X_3^{a_3} \quad (6)$$

The notation center stands for the center point of the variable, which represents the sum of the lower and higher values of the variable divided by two. The notation minimum stands for the lower value of the variable. The constants a_o, a_1, …, a_{33} represent the coefficients of the models. After regressions, a model was obtained in terms of coded variables, as shown in Eq. 7, with a 0.9877 correlation coefficient (R^2). The optimum conditions in terms of coded variables were X_1 = 0.26, X_2 = -0.08, and X_3 = -0.08.

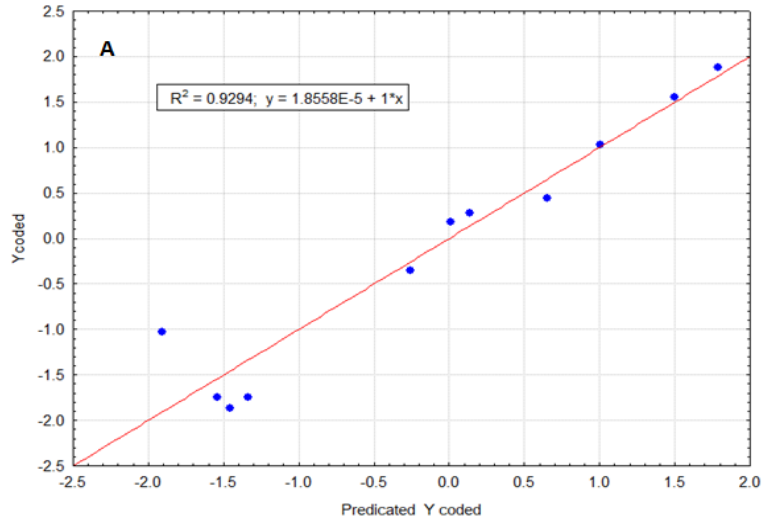
$$y = -2.75 - 0.6x_1 + 1180.8x_2 - 1180.1x_3 + 0.96x_1^2 - 3785.5x_2^2 + 1650.4x_3^2 - 6177.6x_1x_2 + 6176.1x_1x_3 + 2136.9x_2x_3 \quad (7)$$

It was clear that the effect of time was lower in comparison with the effect of powder mass and solvent volume. However, in terms of real variables, two equations were estimated (Eq. 8 and Eq. 9), with 0.9707 and 0.4849 correlation coefficients, respectively. The optimum real variables were 200.3 min, 29.07 g, and 290.7 ml for time, powder mass, and solvent volume, respectively.

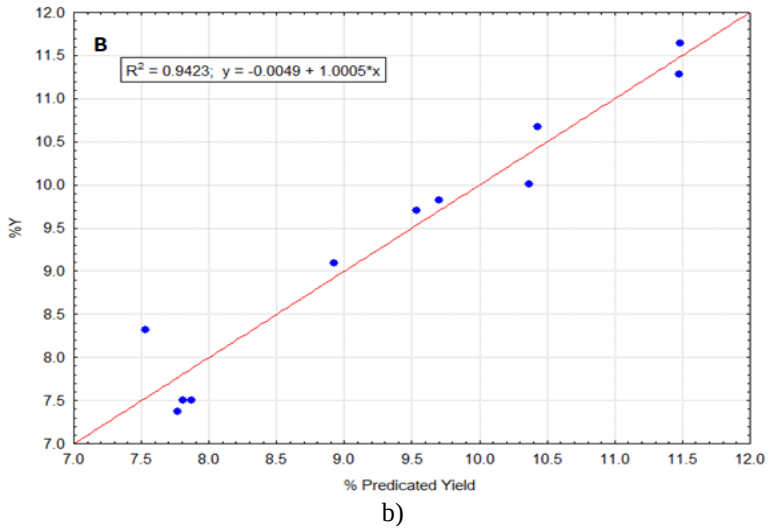
$$\%Y = 74.1 - 0.7t - 11138.5m_p + 1113.9v_s + 0.0023t^2 + 2.98m_p^2 + 0.6v_s^2 + 64.3tm_p - 6.4tv_s - 6.4m_pv_s \quad (8)$$

$$\%Y = 0.86t^{0.29}m_p^{-0.75}v_s^{0.59} \quad (9)$$

Figure 1 shows the experimental versus predicated yield in terms of coded variables (Figure 1 a) and real variables (Figure 1 b). In both cases, the slope and intercept of the fitting lines were close to one and zero, respectively. This indicates a high correlation between the experimental and predicted values (correlation coefficients close to one).



a)



b)

Figure 1. Experimental against predicated yield. a) In term of coded variable, b) In term of real variable

In Figure 2, yield from Eq. 8 was illustrated as a function of process parameters in three-dimensional and contour plots. In Figure 2a, yield varies with powder mass and time. It is clear that the mass of powder and time maximize the yield percentage. In Figure 2b, yield varies with solvent volume and time. Again, solvent volume and time maximize the yield percentage.

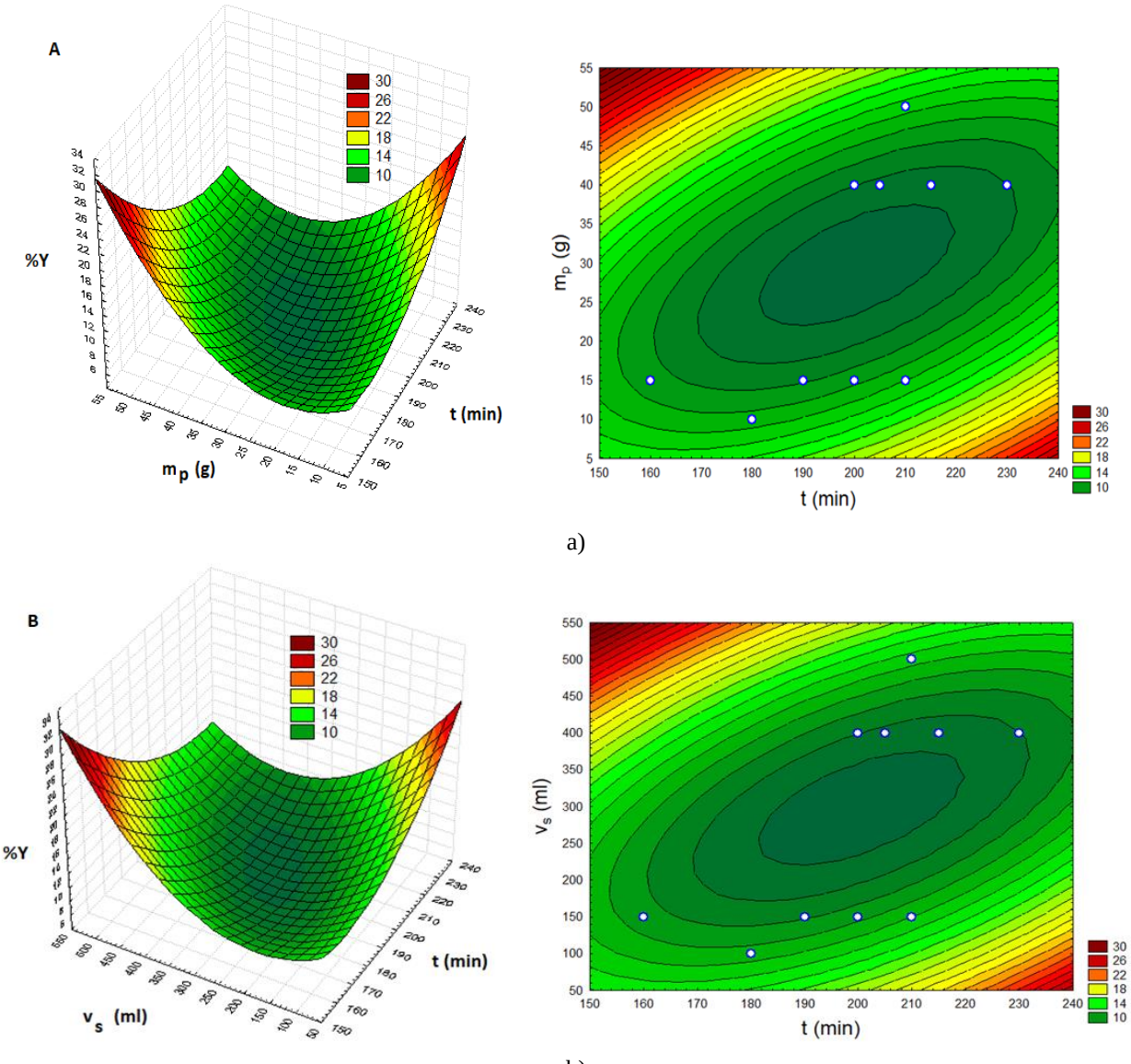
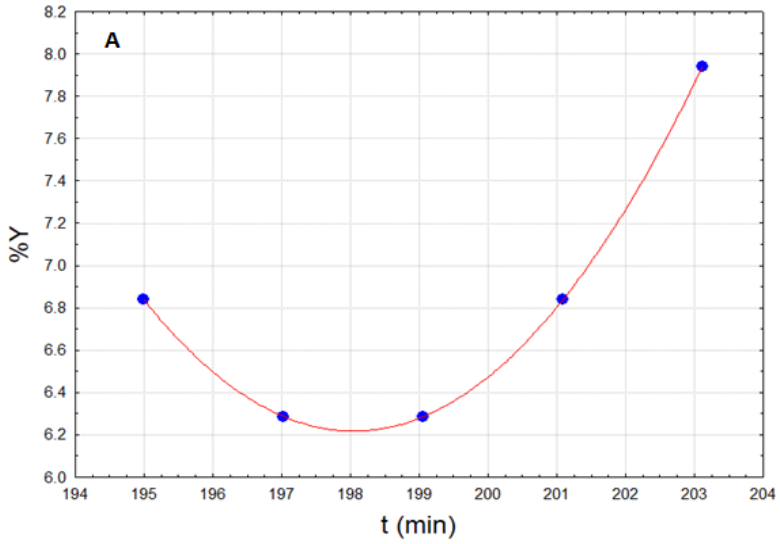
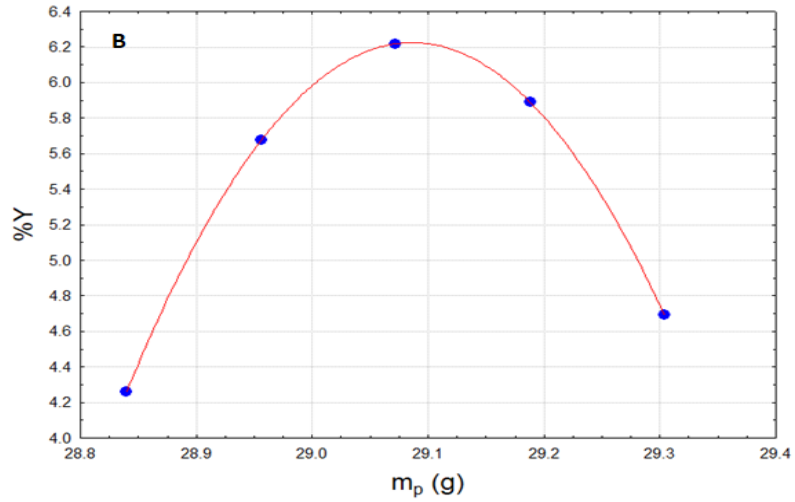


Figure 2. Yield as a function of process parameters. a)Three-dimensions and countour plots for yield as a function of powder mass and time. b)Three-dimensions and countour plots for yield as a function of solvent volume mass and time

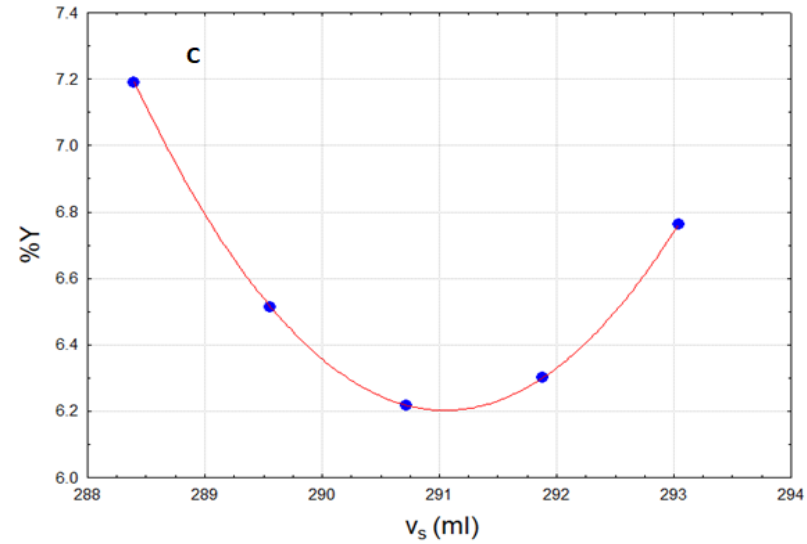
In Figure 3, the variation of yield at optimum conditions is illustrated. Figure 3a shows the yield against time at optimum values of powder mass and solvent volume. The yield initially decreased with time up to the optimum magnitude of 200.3 min, then increased with time. This may be attributed to the diffusion of solvent to the adsorbate; after that, the diffusion of organic material increased as time increased. In Figure 3b, the relationship between yield and powder mass at optimum values of time and solvent volume is depicted. As the powder mass increased, the yield of organic materials also increased, reaching an approximate value of 6.2% at 29.07 g. Subsequently, the yield decreased, a phenomenon attributable to the reduction in organic material content. Finally, Figure 3C shows the yield against solvent volume at optimum values of powder mass and time. The optimum yield was at 290.7 ml.



a)



b)



c)

Figure 3. Variation of yield at optimum conditions. a) Yield against time at optimum values of powder mass and solvent volume. b) Yield against powder mass at optimum values of time and solvent volume. c)Yield against solvent volume at optimum values of powder mass and time

Generally, the maximum yield of total organic materials from okra leaf was 6.2% at the optimum conditions. This value was in agreement with the yield of the extraction process of organic materials from plants and herbs. Dhanani et. al. [15] investigated the yield of the extraction process and the phytochemical constituents of Withania somnifera herb using a Soxhlet extractor. The total yields were 9.08, 9.43, and 9.51% using ethanol, ethanol-water, and water solvents, respectively. Hayouni et. al. [16] studied the effects of extraction method and solvent type yield on organic material from Tunisian Quercus coccifera leaves and Juniperus phoenicea leaves. For Tunisian Quercus coccifera leaves, the yields were 4.21, 5.75, and 3.7 using water, acetone, and chloroform solvents, respectively. While Juniperus phoenicea leaves. For Tunisian Quercus coccifera leaves, the yields were 6.32, 9.65, and 4.14 using water, acetone, and chloroform solvents, respectively. Sharma and Cannoo [17] studied the extraction yield of *Nepeta leucophylla* stems. The percentage yield of organic compounds varied from 0.99 to 12.16%.

4. Conclusion

In this study, solid organic chemicals are extracted from okra leaves using water as a solvent. The extraction procedure was carried out using Soxhlet equipment. The yield percentage was maximized by maximizing the effects of extraction time, solvent volume, and okra powder mass. There were two proposed mathematical models: power models and second-order polynomials. With the polynomial model, a greater correlation coefficient might be achieved. The ideal values for time, okra powder mass, and solvent volume were found to be 200.3 min, 29.07 g, and 290.7 ml, respectively, yielding the greatest extraction yield. The extraction of organic compounds from plants and herbs represents a significant field of study. Since the extracts can be used in many pharmaceutical and industrial applications. In addition, using a suitable experimental design facilitates the limitation of the optimum operating region.

References

[1] Alara, O. R., Abdurahman, N. H., Mudalip, S. K. A., & Olalere, O. A. (2018). Characterization and effect of extraction solvents on the yield and total phenolic content from Vernonia amygdalina leaves. *Journal of Food Measurement and Characterization*, *12*, 311-316.

[2] Charpe, T. W., & Rathod, V. K. (2012). Extraction of glycyrrhizic acid from licorice root using ultrasound: process intensification studies. *Chemical Engineering and Processing: Process Intensification*, *54*, 37-41.

[3] Saraiva, A., Carrascosa, C., Raheem, D., Ramos, F., & Raposo, A. (2020). Natural sweeteners: The relevance of food naturalness for consumers, food security aspects, sustainability and health impacts. *International Journal of Environmental Research and Public Health*, *17*(17), 6285.

[4] Raj, G. B., & Dash, K. K. (2020). Ultrasound-assisted extraction of phytocompounds from dragon fruit peel: Optimization, kinetics and thermodynamic studies. *Ultrasonics Sonochemistry*, *68*, 105180.

[5] Abdullah, H. A., Anaee, R. A., Khadom, A. A., Abd Ali, A. T., Malik, A. H., & Kadhim, M. M. (2023). Experimental and theoretical assessments of the chamomile flower extract as a green corrosion inhibitor for aluminum in artificial seawater. *Results in Chemistry*, *6*, 101035.

[6] Khadom, A. A., Abd, A. N., Ahmed, N. A., Kadhim, M. M., & Fadhil, A. A. (2022). Combined influence of iodide ions and Xanthium Strumarium leaves extract as eco-friendly corrosion inhibitor for low-carbon steel in hydrochloric acid. *Current Research in Green and Sustainable Chemistry*, *5*, 100278.

[7] Lanjekar, K. J., & Rathod, V. K. (2021). Green extraction of Glycyrrhizic acid from Glycyrrhiza glabra using choline chloride based natural deep eutectic solvents (NADESs). *Process Biochemistry*, *102*, 22-32.

[8] Jadhav, D., Rekha, B. N., Gogate, P. R., & Rathod, V. K. (2009). Extraction of vanillin from vanilla pods: A comparison study of conventional soxhlet and ultrasound assisted extraction. *Journal of food engineering*, *93*(4), 421-426.

[9] Kulkarni, V., & Rathod, V. (2016). Green process for extraction of mangiferin from Mangifera indica leaves. *Journal of Biologically Active Products from Nature*, *6*(5-6), 406-411.

[10] Horozić, E. (2020). Effects of extraction solvent/technique on the antioxidant and antimicrobial activity of spring saffron (Crocus vernus (L.) Hill). *Acta Medica Saliniana*, *49*(2).

[11] Wen, C., Zhang, J., Zhang, H., Dzah, C. S., Zandile, M., Duan, Y., ... & Luo, X. (2018). Advances in ultrasound assisted extraction of bioactive compounds from cash crops–A review. *Ultrasonics sonochemistry*, *48*, 538-549.

[12] Chemat, F., Rombaut, N., Meullemiestre, A., Turk, M., Perino, S., Fabiano-Tixier, A. S., & Abert-Vian, M. (2017). Review of green food processing techniques. Preservation, transformation, and extraction. *Innovative Food Science & Emerging Technologies*, *41*, 357-377.

[13] Rashid, K. H., Khadom, A. A., & Mahood, H. B. (2021). Aluminum ASA 6061 anodizing process by chromic acid using Box–Wilson central composite design: optimization and corrosion tendency. *Metals and Materials International*, *27*, 4059-4073.

[14] A Khadom, A., Khudhair Al-Jiboory, A., S Mahdi, M., & B Mahood, H. (2021). Regression and validation studies of the spread of novel COVID-19 in Iraq using mathematical and dynamic neural networks models: A case of the first six months of 2020. *Caspian Journal of Environmental Sciences*, *19*(3), 431-440.

[15] T. Dhanani, Shah, S., Gajbhiye, N. A., & Kumar, S. (2017). Effect of extraction methods on yield, phytochemical constituents and antioxidant activity of Withania somnifera. *Arabian journal of chemistry*, *10*, S1193-S1199.

[16] Hayouni, E. A., Abedrabba, M., Bouix, M., & Hamdi, M. (2007). The effects of solvents and extraction method on the phenolic contents and biological activities in vitro of Tunisian Quercus coccifera L. and Juniperus phoenicea L. fruit extracts. *Food chemistry*, *105*(3), 1126-1134.

[17] Sharma, A., & Cannoo, D. S. (2017). A comparative study of effects of extraction solvents/techniques on percentage yield, polyhenolic composition, and antioxidant potential of various extracts obtained from stems of N epeta leucophylla: RP-HPLC-DAD assessment of its polyhenolic constituents. Journal of food biochemistry, 41(2), e12337.