

INVESTIGATION OF THE SOLID-LIQUID EXTRACTION PARAMETERS OF THE POLYPHENOLS FROM FEIJOA SELLOWIANA PEELS: MASS TRANSFER, KINETICS, AND THERMODYNAMICS STUDIES

FEIJOA SELLOWIANA KABUKLARINDAN POLİFENOLLERİN KATI-SIVI EKSTRAKSİYONUNDA ÇEŞİTLİ PARAMETRELERİNİN İNCELENMESİ: KÜTLE İLETİMİ, KİNETİK VE TERMODİNAMİK ÇALIŞMALAR



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ABSTRACT

In this study, polyphenol recovery from feijoa (*Feijoa sellowiana*) peels was investigated in terms of mass transfer, kinetics and thermodynamics approaches. Ultrasound-assisted extraction (UAE) was employed. 3 different amplitudes (10, 15 and 20%) were use. It was observed that all three systems reached equilibrium within the first five minutes. Increasing the amplitude of the energy increased the temperature of the environment, and in this case, the efficiency increased slightly (≈ 17 mg-GAE/g-DP to ≈ 18 mg-GAE/g-DP). In the UAE system, the diffusion coefficient varied between 2.120×10^{-8} and 3.995×10^{-8} m²/min. The Biot number changed between 150.333×10^{3} and 206.867×10^{3} , while the mass transfer coefficient was calculated between 9.507 and 14.050 m/min. The kinetic data of the UAE system was represented by both kinetic models (R²>0.97). Additionally, it was observed that the rate constants (k₁ and k₂) of both kinetic models generally increased with temperature. When the thermodynamic structure of the UAE system was evaluated with parameters such as ΔH (>0), ΔS (>0) and ΔG (<0), the system has been endothermic, spontaneous and moving towards disorder.

Keywords: Bioactive Metabolites; Biot Number; Biowaste; Effective Diffusivity; Ultrasound Technology

ÖZET

Bu çalışmada feijoa (Feijoa sellowiana) kabuklarından polifenol geri kazanımı kütle transferi, kinetik ve termodinamik yaklaşımlar açısından incelenmiştir. Ultrason destekli ekstraksiyon (UDE) kullanılMIŞTIR. 3 farklı genlikte (%10, 15 ve 20) çalışılmıştır. İlk beş dakika içinde her üç sistemin de dengeye ulaştığı gözlendi. Enerjinin genliğinin arttırılması ortamın sıcaklığını arttırdı ve bu durumda verim bir miktar arttı (≈ 17 mg-GAE/g-DP'den ≈ 18 mg-GAE/g-DP'ye). UDE sisteminde difüzyon katsayısı 2,120×10⁻⁸ ile 3,995×10⁻⁸ m²/dk arasında değişiyordu. Biot sayısı 150.333×10³ ile 206.867×10³ arasında değişirken, kütle transfer katsayısı 9.507 ile 14.050 m/dk arasında hesaplandı. UDE sisteminin kinetik verileri her iki kinetik modelle (R2>0.97) temsil edildi. Ayrıca her iki kinetik modelin hız sabitlerinin (k1 ve k2) genel olarak sıcaklıkla arttığı gözlenmiştir. UDE sisteminin termodinamik yapısı ΔH (>0), ΔS (>0) ve ΔG (<0) gibi parametrelerle değerlendirildiğinde sistemin endotermik, kendiliğinden ve düzensizliğe doğru ilerlediği görülmektedir.

Anahtar Kelimeler: Biot Sayısı; Biyoaktif Metabolitler; Biyoatık; Etkin Yayılma; Ultrason Teknolojisi

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Submission Date Başvuru Tarihi	Revision Date Revizyon Tarihi	Accepted Date Kabul Tarihi	Published Date Yayım Tarihi
15.11.2023	08.12.2023	26.12.2023	27.12.2023

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1. INTRODUCTION

Biorefinery approach simply comprises the conversion processes where raw materials of biological origin are processed to produce high value-added products such as heat, electricity, fuel and fine chemicals. Therefore, particularly biowaste by-products have recently attracted the attention of researchers working in natural products chemistry. Agro-food industry produces huge amounts of solid waste, which is generally employed as feed or fertilizer [1]. Actually, valorization of this biowaste is of great value from an economic perspective. Moreover, recovery of high-added value materials from waste of agro-food industries attracts interest because of the novel technologies in process engineering [2]. The peel, which is almost half the weight of the fruit, is also a rich source of antioxidant polyphenols [3]. So, the recent studies on the bioactive extraction from peels of several fruits show the related source is of significant importance. Suleria et al. screened the bioactive properties of the 20 different fruit peels [4]. The peels of apple, apricot, avocado, banana, custard apple, dragon fruit, grapefruit, kiwifruit, mango, lime, melon, nectarine, orange, papaya, passionfruit, peach, pear, pineapple, plum and pomegranate were compared in terms of total polyphenols, flavonoids and antioxidant activity. Phaleria macrocarpa peels were also investigated as raw material with respect to antioxidant activity and total polyphenols [5]. Microwave-assisted extraction was used as the obtaining method. Guthrie et al. recovered phenolic antioxidants from green kiwifruit peel using subcritical water extraction [6]. Hanafy et al. used pomegranate, orange and banana peels to extract bioactive metabolites [7].

In the current study, we applied ultrasound-assisted extraction (UAE). Castañeda-Valbuena et al. and Safdar et al. extracted polyphenols from mango peels by means of UAE [8,9]. Pomegranate peels were also extracted by the same method (UAE) [10–12]. Mandarin peels were used as polyphenol source by application of UAE [13,14]. Ultrasound treatment was also used for the extraction of polyphenols from orange peels [15]. In this study, we used feijoa (*Feijoa sellowiana*) peels as polyphenol source. This research material was used as potential antioxidant source in previous studies. Santos et al. used pressurized liquid extraction and supercritical fluid extraction for the recovery of polyphenols [17]. The polyphenols have been recovered from feijoa peels by means of UAE. The UAE system has been investigated in terms of mass transport, kinetics and thermodynamics. The response of the UAE system was selected as total polyphenol content (TPC). The aim of this study is to produce findings on effective diffusivity, kinetics and thermodynamics for the first time. So, this study will serve as a guide for further similar studies on the recovery processes of several biomass.

2. MATERIALS AND METHODS

2.1. Materials

Fruit samples were brought from Azerbaijan. The peels were separated from the fruit samples after the fruit was cleaned by distilled water. The peels were dried at ambient conditions before grounded. The moisture content was $\approx 16\%$. The particle size of the solid material was adjusted to 855 µm. Additionally, ethanol, methanol, Folin reagent, gallic acid and Na₂CO₃ were from Merck (Darmstadt, Germany).

2.2. Extraction Procedure

The UAE method was used to examine the kinetics, thermodynamics and mass transfer mechanisms of the extraction process. The UAE device was produced by Sonics and Materials Inc (Newtown, USA; 750 W and 20kHz). The device had a probe system. Ethanol-water solution (60%, v/v) was used as the solvent. The solvent volume was kept at 35 mL, and the weight of the solid was set at 0.5 g. Extract samples with a volume of approximately 30 mL were stored in centrifugal plastic test tubes with a capacity of 50 mL.

2.3. Quantification of Total Polyphenols

Total polyphenol content (TPC) was measured by Folin method as described earlier [18]. The spectrophotometric measurements were performed by UV-Visible spectrophotometer (PG Instruments, T60/Leicestershire, UK). Incubation time was adjusted as 30 min. After 30 min, the absorbance of the solution was read at 765 nm. The data was given as the gallic acid equilance per g dried peel (mg-GAE/g-DP).

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2.4. Mass Transfer, Kinetics and Thermodynamic Calculations

Diffusion coefficient (D_e), mass transfer coefficient (K_T) and Biot number (Bi) were calculated for the UAE of polyphenols from feijoa peels by using the Eqs.1-3. Eq.1 is derived from Fick's second law, while Bi determines the ratio of internal and external resistances of mass transfer by Eq.2 [19].

$$\ln\left(\frac{Y_{\rm S}}{Y_{\rm S}-Y_{\rm t}}\right) = \left(\frac{D_{\rm e}\,\pi^2}{r\,\pi^2}\right)t + \frac{\ln\pi^2}{6}\tag{1}$$

 Y_s = TPC yield at equilibrium (mg-GAE/g-DP)

 Y_t = TPC yield at any time (mg-GAE/g-DP)

r= Particle radius (m)

 D_e = Diffusion coefficient (m²/sec)

$$ln\left(\frac{C_{\infty}}{C_{\infty} - C_T}\right) = \left(\frac{K_T A}{V_S}\right) \tag{2}$$

 C_{∞} = TPC concentration at equilibrium (mg-GAE/L)

 C_T = TPC concentration at any time (mg-GAE/L)

K_T= Mass transfer coefficient (m/min)

A= Particle total surface area (m^2)

 V_{S} = Solution volume (m³)

$$B_i = \frac{rK_T}{D_e} \tag{3}$$

The kinetic data was evaluated by pseudo-first-order model (Eq.4), pseudo-second-order model (Eq.5) and Arrhenius equation (Eq.6), respectively [20]:

$$\ln\left(\frac{c_{\infty}}{c_{\infty} - c_t}\right) = k_1 t + \ln\left(\frac{c_{\infty}}{c_{\infty} - c_0}\right) \tag{4}$$

$$\frac{t}{C_{t}} = \frac{1}{k_{2}C_{e}^{2}} + \frac{t}{C_{e}}$$
(5)

$$lnk = \left(-\frac{E_a}{R}\right) \times \frac{1}{T} + lnk_0 \tag{6}$$

 C_0 = Initial TP concentration (mg-GAE/L)

 C_{∞} = TPC concentration at equilibrium (mg-GAE/L)

Ct= TPC concentration at any time (mg-GAE/L)

t= Time (min)

 k_1 = First-order rate constant (1/min)

 k_2 = Second-order rate constant (L/mg min)

k= Extraction rate constant (L/mg sec) or (1/min)

E_a=Activation energy (kj/mol)

k₀= Frequency factor

T= Temperature (K)

R= Universal gas constant (8.314 J/mol K)

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The thermodynamics of the system was evaluated in terms of enthalpy (Δ H), entropy (Δ S) and Gibbs free energy (Δ G) [21]. Eqs.7-9 were used to calculate the related terms:

$$lnK_e = -\frac{\Delta H}{R} \times \frac{1}{T} + \frac{\Delta S}{R} \tag{7}$$

$$K_e = \frac{Y_s}{Y_{max} - Y_s} \tag{8}$$

$$\Delta G = \Delta H - T \Delta S \tag{9}$$

K_e= Equilibrium constant rate

 $Y_s = TPC$ yield at equilibrium (mg-GAE/g-DP)

Y_{max}= TPC yield at maximum (mg-GAE/g-DP)

 ΔG = Gibbs free energy change (kj/mol)

 ΔH = Enthalpy change (kj/mol)

 ΔS = Entropy change (kj/mol K)

3. RESULTS AND DISCUSSIONS

3.1. Ultrasound-Assisted Extraction

Table 1 and Figure 1 demonstrate the polyphenol levels in the peel extracts obtained by UAE under several amplitude values depending on the time. The amplitude of the ultrasound indicates the energy. The temperature values were also recorded after each run. The given temperature values are the arithmetic mean of the all extracts under constant amplitude.

Amplitude (%)	Time (min)	TPC (mg-GAE/g-DP)	Temperature (°C)
	1	9.9±0.01	
	2	15.3±0.03	
	3	16.5±0.01	
10	4	16.6±0.01	21
10	5	16.9±0.02	21
	10	16.9±0.01	
	15	16.89 ± 0.05	
	20	16.88 ± 0.04	
	1	12.87±0.01	
	2	16.07 ± 0.01	
	3	17.057 ± 0.06	
15	4	17.26 ± 0.01	25
15	5	17.23 ± 0.05	25
	10	$17.30{\pm}0.01$	
	15	17.25 ± 0.01	
	20	17.23 ± 0.01	
20	1	$12.34{\pm}0.01$	
	2	16.12 ± 0.01	20
	3	18.23 ± 0.06	30
	4	18.3 ± 0.01	

Table 1. TPC levels of the *Feijoa sellowiana* peel extracts obtained by UAE under different amplitude and time values.

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	5	18.3±0.01	
	10	18.3±0.05	

As seen in Figure 1, the related system is in equilibrium within the first five minutes. Increasing the amplitude of the energy increased the temperature of the environment, and in this case, the efficiency increased slightly. The TPC yield increased from approximately 16.5 (10%) to \approx 17 mg-GAE/g-DP (15%), and finally to \approx 18 mg-GAE/g-DP (20%). These findings are superior than those of the supercritical fluid extraction (with CO₂ and ethanol) [16]. Our results were also in convenient with the values of microwave-assisted extraction [17].



Figure 1. Identification of the equilibrium time under different amplitude values.

3.2. Mass Transfer Studies

The mass transfer mechanism in obtaining extract from *F. sellowiana* fruit peels using UAE was expressed by diffusion coefficient, mass transfer coefficient and Biot number, respectively. The UAE system operated under three different temperatures (294, 298 and 304 K). Table 2 shows the diffusion coefficient, mass transfer coefficient and Biot number values for the system depending on the temperature.

Table 2. Mass transport parameters of the UAE system for the recovery of *F. sellowiana* fruit peels.

Parameter	Temperature (K)			
	294	298	304	
$D_{e} (m^{2}/min) \times 10^{-8}$	2.120	2.823	3.995	
Bi ×10 ³	191.710	206.867	150.333	
K _T (m/min)	9.507	13.661	14.050	

As seen in Table 2, the diffusion coefficient varies between 2.120×10^{-8} and 3.995×10^{-8} m²/min (Figure 2). The mass transfer coefficient was calculated between 9.507 and 14.050 m/min (Figure 3). It is observed that mass transfer increases with temperature. This is an expected result, since increasing the temperature decreases the viscosity. In this case, it increases the transmission of the target component to the solvent system.



Figure 2. Diffusion coefficients under different temperature values.

The Biot number varies between 150.333×10^3 and 206.867×10^3 (Figure 4). The level of Bi gives an idea about the resistance between the internal and external. So, our Bi was calculated as high (>100), indicating that internal resistance is higher than external resistance. This means that the contact between the feijoa peel and the solvent (60% of ethanol solution) [22].



Figure 3. Mass transfer coefficients under different temperature values.



Figure 4. Biot numbers under different temperature values.

3.3. Kinetics Studies

Within the scope of this study, the kinetics mechanism for the obtaining extract from *F. sellowiana* fruit peels using UAE was explained with 2 different mathematical models. These models are pseudo-first-order kinetic model and pseudo-second-order kinetic model. The kinetics were studied at three different temperatures (294, 298 and 304 K). Table 3 shows the first- and second-order rate constants, activation energy and R^2 values for the related equations.

Pseudo-first-order constant increases by temperature as seen in Table 3. Similar observation on pseudo-first-order constant tendency was also recorded by Sant'Anna et al, who investigated polyphenol extraction from grape marc [23]. Additionally, the R^2 values of the model are satisfactory. The kinetic data was represented much better with pseudo-second-order kinetic model depending on the R^2 values (>0.99). Hence, activation energy was calculated by Arrhenius equation using the constant of pseudo-second-order kinetic model (k₂).

Model	Parameter	Ten	nperature	(K)
Pseudo-first-order		294	298	304
	k ₁ (1/sec)	0.6235	0.8959	0.9215
	R ²	0.9949	0.9985	0.9757
Pseudo-second-order		294 298 304		304
	k ₂ (L/mg sec)	0.0023	0.0029	0.0024
	R ²	0.9976	0.9984	0.9960
E _a ((kJ/mol)		4.90	

Table 3. Kinetic parameters of the UAE system for the recovery of *F. sellowiana* fruit peels.

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	R ²	0.9520	

The relationship between 1/T and lnk_2 is indicated by Figure 5. E_a was calculated as 4.9 kJ/mol as seen in Table 3. 3.52 kJ/mol was reported for the UAE of polyphenols from moringa leaves [24].



Figure 5. Application of Arrhenius equation to calculate activation energy.

3.4. Thermodynamics Studies

The thermodynamic structure of the system was evaluated with parameters such as ΔH , ΔS and ΔG in obtaining extract from *F. sellowiana* fruit peels using UAE. Table 4 shows the relevant parameters.

Parameter	Te	Temperature (K)	
	294	298	304
K _e	3.8259	4.1276	6.2390
$\Delta H (kJ/mol)$		37.45	
$\Delta S (kJ/mol K)$		0.138	
$\Delta G (kJ/mol)$	-3.122	-3.674	-4.502
R ²		0.9272	

Table 4. Thermodynamics parameters of the UAE system for the recovery of F. sellowiana fruit peels.

A positive enthalpy change indicates that the system is endothermic. A positive entropy change is an indication that the system is heading towards disorder. Gibbs free energy was calculated as negative at all temperature values. This indicates that the UAE system studied behaves spontaneously. Similar thermodynamic tendency in case of entropy, enthalpy and Gibbs free energy changes was also reported for the UAE of polyphenols from moringa leaves [24], microwave-assisted extraction of metabolites from *Terminalia bellerica* [25], and UAE of bioactive substance from UAE *Taxus chinensis* leaves [26].

4. CONLCUSION

Feijoa fruit (*Feijoa sellowiana*) peel was chosen as the source of polyphenol within the scope of this study. Ultrasound-assisted extraction was chosen to achieve the target material. The extraction time was

determined by obtaining the kinetic data of the ultrasound-assisted extraction method. The kinetic mechanism of the method was explained by two different mathematical models: pseudo-first-order kinetic model and pseudo-second-order kinetic model. Additionally, the activation energy of the relevant system was calculated using the kinetic data. The mass transfer mechanism of the ultrasound-assisted extraction process was expressed by the diffusion coefficient, mass transfer coefficient and Biot number under different temperature values. Thermodynamic parameters (enthalpy, entropy and Gibbs free energy changes) of the same system were also calculated. To conclude, the outcome of the current study indicates that the peel of feijoa fruit is a rich source of bioactive components. The data produced is of great value for the prediction of process conditions in industrial scales.

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