Ultrasonic-assisted Extraction of Sunflower Seeds Oil: Kinetic and Thermodynamics Study

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***Abstract* — This study has been conducted to evaluate the kinetic and thermodynamics of oil extraction from sunflower seeds using ultrasonic-assisted extraction (UAE). The first scope was one factor at a time (OFAT). The OFAT study involved different type of solvent, extraction time, temperature, and L/S ratio. The maximum oil yield (54.4%) was obtained with the used of ethyl acetate as solvent at 20 minutes, 40ºC, and 100 ml/g of L/S ratio. Second order kinetic model was used in describing the extraction process. The model coefficients were observed with determination coefficient ($R^2 \geq 0.9779$) and MRPD value 2.0692% – 9.5936%. Thermodynamics study showed that the activation energy in this extraction process was 1526.78 J mol^{-1}.**

***Keywords* — Sunflower (*Helianthus annuus L.*), ultrasonic-assisted extraction, seeds oil, one factor at a time (OFAT), second order kinetic model, activation energy**

I. Introduction

Sunflower (*Helianthus annuus L.*) as shown in Figure 1 is the cultivated oilseed plant that originated from Russia. [1] This unique plant is widespread to every region including Ukraine, Argentina, United States, Romania, Hungary, France, Bulgaria, Turkey, and China. [2] Sunflower tree is allied to Asteraceae family. [3] According to Sharma S. et al. (2009), worldwide sunflower crop is ranking third after groundnut and mustard. [4] In Europe, sunflower seeds oil is the second extensively utilized oil after rapeseeds. [5] Sunflower seed generally known as oil-rich seed which contain 25% to 48% of yield. But, the percentage oil yield may be reached up to 65% conditioning on the environment factors and genotypes used. [6,7] Sunflower oil is one of the well-known oils that can be applied in food consumptions as cooking oil and also used as biodiesel [8,9] Moreover, sunflower oil might be exploited in skin products including massage oils, lotion and creams. [4] Sunflower oil is being utilized as a supplement due to their antibacterial, antifungal, anti-inflammatory, and anti-cancer activity, as well as cardioprotection properties. [10]

Fig 1: Sunflower and its seeds (Madhavi et al.,2010)

According to Castejon et al. (2018), the oil from oleaginous plant can be extracted using various different techniques such as Soxhlet extraction, supercritical fluid extraction, microwave-assisted extraction, and ultrasonic-assisted extraction (UAE). All the introduced extraction method was aimed to increase the yield of oil and to reduce the extraction cost, same goes to UAE method. This extraction technique also can minimize extraction time with the aid of ultrasonic wave. [11,12] Bhaskaracharya et al. (2009) stated that UAE is a very good technique to achieved high yield of valuable compounds. The combination of ultrasonic wave and sufficient heat supplied is beneficial for extraction process because it can save a lot of energy by reducing the extraction time. [13] Recently, UAE have been extensively used in extraction of tea seeds, papaya seeds, raspberry seeds, and others. [14-16] Mohd. Fuad F. and Abd. Karim K. carried out an extraction of *Calophyllum inophyllum Seeds* using UAE and the maximum oil yield obtained from an experiment is around 56% at optimum condition. [17] Next, Goula M. A. (2012) had carried out an extraction of pomegranate seed and resulted the oil yield reach 45%. [18] Hence, regarding these two experimental results, UAE has high potential in giving high oil yield as well as minimized the extraction cost. [19]

The oil extraction using UAE was amplified by cavitation phenomenon. [20] Throughout the process, cavitation bubbles are formed and increase in size until its disintegrate instantaneously. The disintegration of bubbles will release a higher rate of energy and resulted in increasing of temperature and pressure in the system. [17,21] As a consequence, the rapid adiabatic compression in UAE will lead to the greater of oil escaping from seeds into the solvent. [17,22] Although the sunflower seeds oil has been extracted using UAE before, the study was sparsely done.

In the extraction process, the kinetic models have been used broadly. Numerous types of kinetic models are available including Patricelli's model, diffusive model (Fick's Second Law), first order kinetic model, and second order kinetic model. Nevertheless, Pongmalai et al. (2015) claimed that second order model is the most suitable for solid-liquid extraction process. [23] Second order kinetic model is appertained based on two simultaneous process. [24] First process is the solute extracted abruptly due to the driving force of fresh solvent. During this step, the oil from internal seeds will diffuse out rapidly. For the second step, extraction process become slower caused by the external diffusion of remainder oil into the solvent. [23] In terms of thermodynamics, activation energy for second order system were determine by the Arrhenius law. [25] From an engineering scope, the understanding obtained from kinetic and thermodynamics study is advantageous for scaling up and operation in industry. [26]

This study deals with UAE of oil from sunflower seeds. The major purpose of this study is to evaluate the relevancy of second order kinetic model in illustrating the kinetic behavior of sunflower seeds oil using UAE along with diagnosing the effect of solvent used, extraction time, temperature, and liquid to solid ratio (L/S) on the oil yield. Thermodynamics of the extraction process was also evaluated.

II. METHODOLOGY

A. Materials

Raw and dried sunflower seeds were purchased from local market in Shah Alam, Selangor. The seeds were grind by using a blender and sieved by using sieve shaker with the size of 850 µm. The uniform size of seeds sample or size below 850 µm were used in this study.

B. Ultrasound-assisted Extraction

An ultrasonic bath (S 80 H Elmasonic) was used for generating indirect ultrasonication. The internal dimension of tank is 505 mm width, 137 mm depth, and 264 mm of height. The total power consumption is 750 W with 37 kHz of ultrasonic frequency. This type of ultrasonic bath is equipped with timer and temperature controller. During the experiment, ultrasonic bath was filled with distilled water roughly 2/3 from the internal volume and the temperature of water is being controlled at desired point. 5 g of seeds sample were mixed with 100 ml of ethyl acetate in an Erlenmeyer flask (250 ml). The flask is covered with aluminium foil. Next, the flask was submerged with specific time in the ultrasonic bath. When the extraction process has completed, the solution was centrifuged at 6000 rpm for 30 minutes to separate the mixture of liquid from solid residue. Afterwards, the mixture of solid was being separated using rotary evaporator (Heidolph). The extracted oil obtained was placed into the desiccator in order to remove the moisture. Next, the oil was weighed until it reached the constant mass and the final reading were recorded. In this study, type of solvents used were ethyl acetate, ethanol, methanol, and acetone. In addition, extraction time, extraction temperature, along with L/S ratio were also varied from 5 – 30 minutes, 30 – 45ºC and 10 – 50 ml/g respectively.

C. Determination of Oil Yield from Sunflower Seeds

The sunflower seeds oil yield was calculated using Equation 1. [23]

$$\text{Oil yield (\%)} = \frac{\text{Mass of oil obtained}}{\text{Mass of plant material}} \times 100\% \quad (1)$$

D. Mathematical Model

Second order kinetic model is expressed in equation 2:

$$\frac{dC_t}{dt} = k_t(C_s - C_t)^2 \quad (2)$$

where;
k_t = Extraction rate constant (mL g^{-1} min^{-1})
C_s = Concentration of oil at saturation (g mL^{-1})
C_t = Concentration of oil in solution at any time (g mL^{-1})

By integrate Equation 2,

$$\int_0^t k_t\, dt = \int_0^{C_t} \frac{1}{(C_s - C_t)^2}\, dC_t \quad (3)$$

Second order extraction obtained,

$$C_t = \frac{C_s^2 k_t t}{1 + C_s k_t t} \quad (4)$$

Rearrange equation 4,

$$\frac{t}{C_t} = \frac{1}{k_t C_s^2} + \frac{t}{C_s} \quad (5)$$

By plotting a graph of t/C_t versus t, the extraction rate constant, k_t and concentration of oil at saturation, C_s can be determined. [25]

E. Activation Energy

Activation energy for second order system were determine by Arrhenius law:

$$k = Aexp\left(-\frac{E}{RT}\right) \quad (6)$$

where:
k = Extraction rate constant (mL g^{-1} min^{-1})
A = Temperature independent factor (mL g^{-1} min^{-1})
E = Activation energy (J mol^{-1})
R = Gas constant (8.314 J mol^{-1} K^{-1})
T = Absolute suspension temperature (K)

Linearize and rearrange equation (6) to find the value of activation energy by plotting the graph of ln (k) versus 1/T. [25]

$$ln(k) = ln(A) + \left(\frac{-E}{R}\right)\frac{1}{T} \quad (7)$$

F. Statistical Analysis

The validation of second order model was determined by evaluating the differences between the experimental and predicted oil yield via mean relative percentage deviation (MRPD) value that defined in equation 8:

$$\text{MRPD}(\%) = \frac{100}{\text{N}} \times \sum \frac{|\text{Y} - \text{Y}_\text{P}|}{\text{Y}} \quad (8)$$

Where:
Y : Experimental yield of sunflower seeds oil
Y_p: Predicted yield of sunflower seeds oil
N : Number of experimental data
Mathematical model is considered acceptable to be used in describing extraction process if MRPD value less than 10%. [17, 27]

III. RESULTS AND DISCUSSION

A. The Effects of Extraction Solvent

Figure 2 showed the total amount of extracted oil from sunflower seeds using a different solvent. The extraction using ethyl acetate gave the highest average oil yield which was 37.62% and followed with acetone 24.17%, methanol 3.5%, as well as ethanol 3.2%. According to Dasari and Goud (2013), the oil extraction from castor seeds using ethyl acetate also give the greater yield which was 49.1% compared to hexane, petroleum ether, n-pentane, cycloxane, isopropanol, and methanol. [28] Norshazila et al. (2017) extracted carotenoid from pumpkin by using three different solvents including ethyl acetate, acetic acid and ethanol. Ethyl acetate gave the prohibitive carotenoid 77.30 μg/g, contrasted with 54.98 μg/g using ethanol and 68.10 μg/g using acetic acid. [29] Reichardt (2003) reported that ethyl acetate is good for extraction because it has less polarity with only 0.228. [29,30] According to Dutkiewicz M. (1990), the polarity of acetone, ethanol and methanol are 0.355, 0.654 and 0.762 respectively. [31] While according to Iwata and Shimada (2012), vegetable oil has low non polarity. [29,32] Therefore, ethyl acetate is suitable for vegetable oil extraction due to the polar and non-polar interaction. [33]

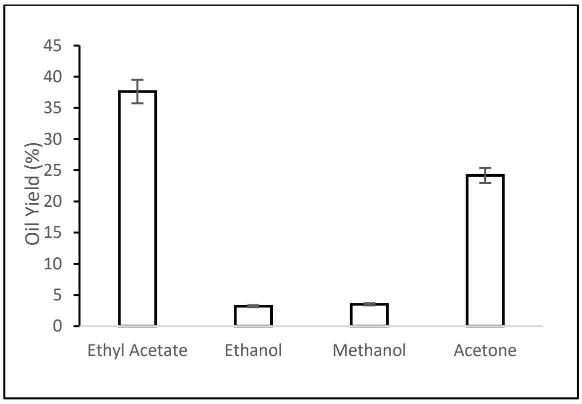

Fig. 2: Effect of different solvents on oil yield extracted from sunflower seeds (condition: extraction time 30 minutes, extraction temperature 40ºC, and L/S ratio 10 ml/g).

B. The Effects of Extraction Time

Figure 3 illustrated oil yield from sunflower seeds obtained from a different extraction time. As the extraction time increase, the oil yield keep increasing until its optimum condition. Oil yield increase rapidly from 42.77% to 51.07% at time 5 to 20 minutes. After 20 minutes, the oil yield decrease to 47.98%. According to Saxena et al. (2011), the higher extraction rate during the initial stage due to the lower oil concentration in the solvent. Hence, it will result the higher driving force and a great solubility of oil into the solvent. At time 20 to 30 minutes, the extraction rate is slow because of the lower mass transfer due to the increasing oil concentration in the solvent. [34] Zhang et al. (2008) extracted of oil from flaxseed by using UAE. As a resulted, the oil yield was optimum at 30 minutes. [35] Next, Wang and Wei (2015) optimized UAE of oil from *Physalisalkekengi L.* seeds. The highest extraction yield (22.78%) was analyzed at 40 minutes. [36] Consequently, all the experiments showed that ultrasonic wave can reduce extraction time by initiating the greater contact area between the solute and the solvent. [37]

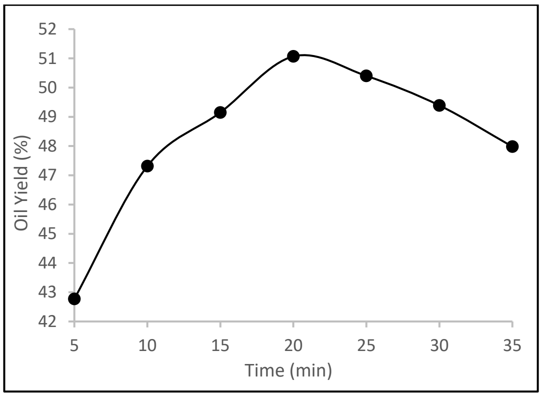

Fig. 3: Effect of extraction time on oil yield extracted from sunflower seeds (condition: extraction solvent ethyl acetate, extraction temperature 40ºC, and L/S ratio 10 ml/g).

C. The Effects of Extraction Temperature

Figure 4 demonstrated the overall data of extracted oil from sunflower seeds using a different extraction temperature. During the initial phase which is 30ºC to 40ºC, the oil yield increase speedily from 40.34% to 53.74%. According to Panda et al. (2019), when the temperature increase, the cavitational intensity increase and promote the greater bubbles penetration from solvent into the cells. Hence, the oil escaped from the cell is higher and give the optimum extraction yield at 40ºC (53.74%). [21] Beyond 40ºC, the oil yield decreased steadily until it reached 44.15% at 60ºC. According to Oniya et al. (2017), the oil yield decreased when the temperature almost reached the boiling point of the solvent. This is due to the evaporation of the solvent occurred as well as temperature increased. [39] Besides, the decreasing oil yield at elevated temperature (>40ºC) also due to the degradation of oil in the seed. [39]

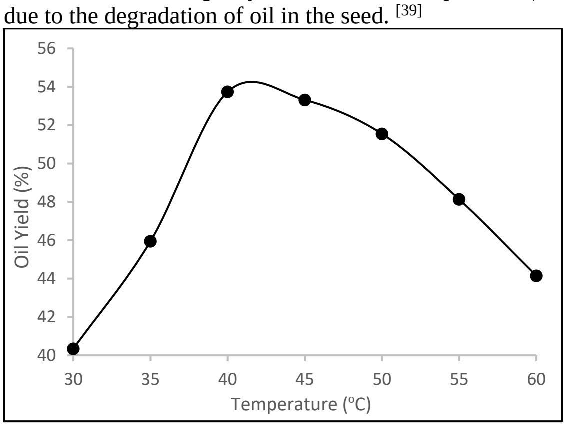

Fig. 4: Effect of extraction temperature on oil yield extracted from sunflower seeds (condition: extraction solvent ethyl acetate, extraction time 20 minutes, and L/S ratio 10 ml/g).

D. The Effects of Liquid to Solid Ratio

The oil yield from sunflower seeds using a different L/S ratio were determined (Figure 5). In the first phase (10 – 20 ml/g), the percentage of oil extracted was increase expeditiously from 52.36% to 54.51%. Afterwards, the percentage oil yield reduced slowly from 54.51% to 53.92% with the addition of L/S ratio (20 – 50 ml/g). According to Sayyar et al. (2009), the increasing L/S ratio up to optimum condition will heighten the percentage of oil yield. This is due to the higher concentration gradient between solid and solvent which can promote a greater mass transfer. [24] Based on the graph, a good mass transfer reached when 20 ml/g of solvent were used. Above 20 ml/g, the extraction yield is reduced because the transportation intensity of ultrasonic energy in the solution decrease as well as L/S increase. [40] Dissipation of ultrasonic energy also occurred by excessive mixture vibration that may be lead to the swelling of sunflower seeds. Therefore, the oil extracted is low. [41]

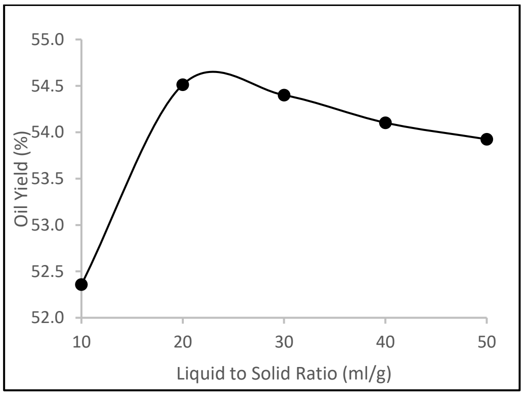

Fig. 5: Effect of liquid to solid ratio on oil yield extracted from sunflower seeds (condition: extraction solvent ethyl acetate, extraction time 20 minutes, extraction temperature 40ºC).

E. Kinetic Study

The experimental data (illustrated by symbols) and modelling data (illustrated by line) of oil extraction for three different temperatures is shown in Figure 6. It can be observed that oil yield increased quickly during the initial stage (5 – 20 min). The oil yield is the highest at 40ºC (43.57% - 54.42%) which followed by 45ºC (43.20% - 54.15%) and 35ºC (43% - 53.57%). During this stage, the rapid extraction and greater mass transfer occurred which called washing process. After 20 minutes, the slower process takes place in the second stage of the extraction that resulted the oil yield decrease to 53.92% (40ºC), 53.70% (45ºC), and 53.33% (35ºC). Second stage is called diffusion process due to the reduction of extraction rate and unsteady mass transfer. That is why the oil yield is decreased. [17,42]

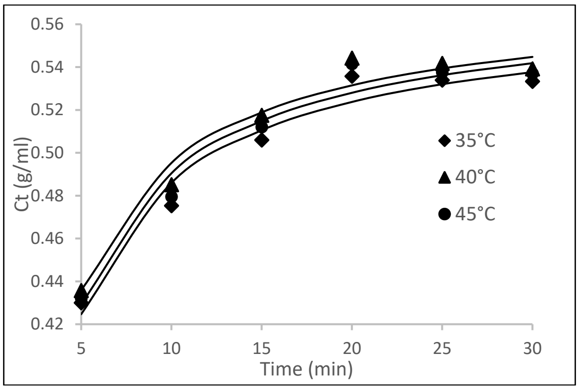

Fig. 6: Effect of different extraction temperature on kinetic of oil yield extraction from sunflower seeds (condition: extraction solvent ethyl acetate, extraction time 20 minutes, and L/S ratio 20 ml/g).

Table 1: The oil yields at equilibrium and mass transfer coefficients for different extraction temperature.

Extraction temperature (ºC)	C_s (g/mL)	k_t (mL g^{-1} min^{-1})	R^2	MRPD (%)
35	0.5679	1.0442	0.9959	5.2331
40	0.5734	1.1056	0.9924	9.5936
45	0.5718	1.0543	0.9793	7.7921

According to the Table 1, the coefficients of second order kinetic model with a different temperature were determined through the calculations from the experimental results. The modelling result showed the extraction temperature influenced extraction rate constant (k_t) and concentration of oil at saturation (C_s). From 35ºC to 40ºC, the constant C_s and k_t exemplified the increasing trend from 0.5679 g/mL to 0.5734 g/mL and 1.0442 mL g^{-1} min^{-1} to 1.1056 mLg^{-1} min^{-1} respectively. The increasing value of k_t and C_s due to the increasing of cavitational concentration that initiate the optimum bubble penetration. [21] At 45ºC, the decreasing value of C_s (0.5718 g/mL) and k_t (1.1056 mL g^{-1} min^{-1}) occurred. The reduction of k_t and C_s values at 45ºC due to the evaporation of solvent that may lead to depletion of oil produced. [38] In this study, the higher value of determination coefficient ($R^2 \geq 0.9793$) and MRPD value is between 5.2391% – 9.5936% for each temperatures indicates that the data from the experimental is fit to the modelling data. Therefore, the modelling data is acceptable to be used for the oil extraction from sunflower seeds. [17,43]

Next, the experimental data (demonstrated by symbols) and modelling data (demonstrated by line) of oil extraction with different L/S ratio is shown in Figure 7. During the initial stage (washing process), the oil yield increase for all the L/S ratios. The oil yield is the highest at 20 mL/g (43.57% - 54.42%) which followed by 30 mL/g (43.53% - 54.14%) and 10 mL/g (43.37% - 53.66%). Later, after 20 minutes, the slower process takes place in the second stage (diffusion process) of the extraction that resulted the oil yield decrease to 53.92% (20 mL/g), 53.72% (30 mL/g), and 53.39% (10 mL/g). [17,42]

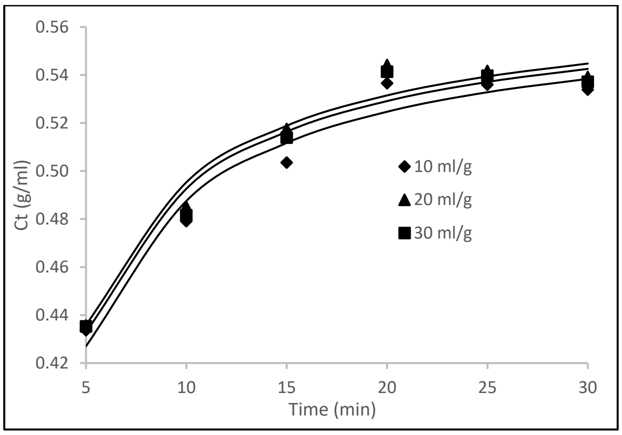

Fig. 7: Effect of different extraction temperature on kinetic of oil yield extraction from sunflower seeds (condition: extraction solvent ethyl acetate, extraction time 20 minutes, and L/S ratio 20 ml/g).

Table 2: The oil yields at equilibrium and mass transfer coefficients for different extraction ratio.

Liquid to solid ratio (g/mL)	Cs (g/mL)	k (mL g^{-1} min^{-1})	R^2	MRPD (%)
10	0.5680	1.0664	0.9929	2.0692
20	0.5734	1.1056	0.9924	9.5936
30	0.5715	1.0925	0.9779	8.1044

Based on the Table 2, the coefficients of second order kinetic model with a different L/S ratios were obtained. The modelling result showed the L/S ratio effected extraction rate constant (k_t) and concentration of oil at saturation (C_s). From 10 mL/g to 20 mL/g, the constant C_s and k_t increased from 0.5680 g/mL to 0.5734 g/mL and 1.0664 mL g^{-1} min^{-1} to 1.1056 mLg^{-1} min^{-1} respectively. This is due to the higher L/S ratio will fasten the reaction rate. [24] However, the increased of L/S ratio from 20 mL/g to 30 mL/g resulted the decreasing C_s (0.5715 g/mL) and k_t (1.0925 mL g^{-1} min^{-1}) values. The reduction of k_t and C_s value at greater L/S ratio (30 ml/g) due to the of ultrasonic energy in the solution decrease when L/S ratio increase. [40] The value of determination coefficient ($R^2 \geq 0.9779$) and MRPD is between 2.0692% – 9.5936% gained from this study proved that modelling data is acceptable and can be applied for sunflower seeds oil extraction. [17,43]

F. Thermodynamics Study

Figure 8 demonstrated the plot of ln (k_t) versus 1/T.

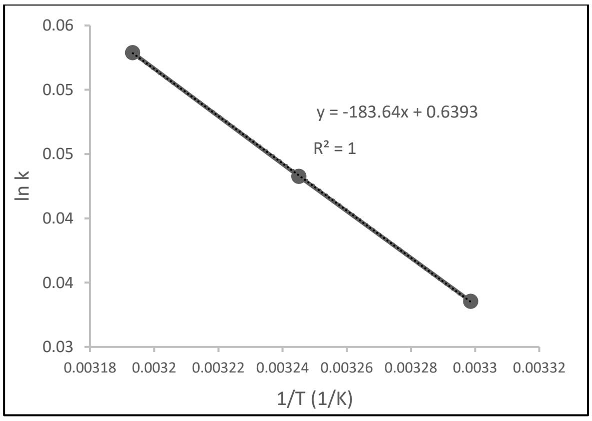

Fig. 8: Linear relationship between extraction constant, ln (k) and temperature for oil extraction from sunflower seed.

According to Equation 7, the value of temperature independent factor (A) and activation energy (E) obtained from graph were 1.8952 mL g^{-1} min^{-1} and 1526.78 J mol^{-1}. As a conclusion, the oil extraction from sunflower seeds oil is an endothermic process due to the positive value of activation energy. [24]

IV. CONCLUSION

In this study, the optimal condition for oil extraction from sunflower seeds were determined through one factor at a time (OFAT). As a resulted, ethyl acetate was the best extraction solvent, optimal extraction time was 20 minutes, extraction temperature was 40ºC, and L/S ratio was 20mg/L. Next, the evaluation of second order kinetic model is accepted with the higher determination coefficient ($R^2 \geq 0.9779$) and the lower MRPD value (< 10%). In thermodynamics study, the activation energy of the system was 1526.78 J mol^{-1}.

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