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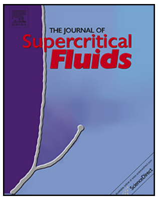

Kinetic modelling of supercritical carbon dioxide extraction of sage (*Salvia officinalis* L.) leaves and jatropha (*Jatropha curcas* L.) seeds

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ABSTRACT

In this study, the modelling of supercritical carbon dioxide extraction of sage leaves at different temperatures between the range of 40–60 °C and jatropha seeds at 50 °C are studied. These extraction processes are modelled using modified Reverchon–Sesti Osseo model equation at different temperature ranges. The influence of temperature on the total extract yield of sage are investigated. It is found that the modified Reverchon–Sesti Osseo model equation provides a good fit with respect to the experimental yield of total extracts of sage leaves and jatropha seeds over the entire range of experimental conditions explored.

1. Introduction

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There are various approaches used for the recovery of high value-added components from plants and biomass materials. The conventional procedures of extracting and separating active substances from plant materials such as steam distillation and extraction with organic solvents have some drawbacks. Due to the operation of steam distillation process at elevated temperatures, selectively extracted materials may tend to undergo thermal transformation or degradation, hence altering the structure and properties of the extracts. The use of organic solvents raises concern as they may be insufficiently selective and as a result, contamination of the product with undesired substances may occur [1,2]. Furthermore, extraction with organic solvents can hardly produce a solvent-free extract, leaving traces of solvent that are undesirable for organoleptic and health reasons. In view of these drawbacks, the extraction of organic compounds using supercritical fluid has gained much attention recently [3]. This technology has been employed in food processings and preservatives industries, medical and pharmaceutical fields, cosmetics products and many other applications extensively.

Supercritical fluid extraction (SFE) has been long established and employed to separate and extract materials of interest from a mixture of chemical compounds. SFE is widely used in the extraction of organic compounds from a variety of plant materials due to its efficiency and robustness in many applications. According to statistics, supercritical fluids have been mainly applied to the extraction of plant seeds and leaves, which contribute to about 45% among all the plant fractions (seeds, leaves, fruits, roots, flowers, rhizomes and bark) studied [4]. Previous researches have reported to effect of process parameters of the extraction of triglycerides from jatropha seeds using supercritical CO_2 [5,6]. Besides, studies on modelling of extraction of sage oil by supercritical CO_2 have also been reported [7–9]. In supercritical fluid extraction, carbon dioxide is the most widely used medium because of its great versatility and low critical parameters (31.1 °C, 7.4 MPa), non-flammability, non-toxicity, and more importantly, it is abundant and cheap [10]. Extraction procedures involving supercritical carbon dioxide (CO_2) are environmentally benign as this clean technology leaves no secondary products polluting the environment. Besides, CO_2 is chemically stable and shows great affinity to volatile (lipophilic) compounds and can be easily removed from any extract [11–15]. The unique properties of supercritical fluid such as high densities and diffusivities enhance the solubility, extraction and separation of organic compounds [10]. By changing the pressure and/or temperature above the critical point of CO_2, a pronounced change in density

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($d_c = 0.467$ g/ml) and dielectric constant (solvent power) can be achieved [16–18].

Many studies on kinetics of supercritical fluid extraction of various plant feedstocks have been widely conducted [4] in order to establish kinetic models that are extremely important for the upscaling of SFE processes into commercial industrial scales. In this context, research efforts such as continuous development of kinetic models and improvement of their robustness provide vital knowledge into developing larger scale processes. There are various kinetic models developed for the application in supercritical extraction [3]. Reverchon–Sesti Osseo model has been employed in several studies involving the extraction of basil oil, thyme, rosemary, marjoram and lavender [19–23]. In this study, the extraction of active substances (fixed oil) from sage leaves at 40, 50 and 60 °C and jatropha seeds at 50 °C are conducted using supercritical carbon dioxide. The kinetics of the extraction yield are modelled and compared using modified Reverchon–Sesti Osseo model.

2. Materials and methods

2.1. Supercritical fluid extractor

Supercritical fluid extraction with carbon dioxide in this study is carried out using a laboratory-scale high pressure extraction plant, HPEP (Nova Swiss, Effretikon, Switzerland). The extraction plant is composed of a diaphragm-type compressor (up to 100 MPa), an extractor with internal volume of 200 ml ($P_{\max} = 70$ MPa), a separator with an internal volume of 200 ml with a maximum pressure of 25 MPa and maximum CO_2 mass flow rate of approximately 0.095 kg/min.

2.2. Supercritical extraction of sage (*Salvia officinalis* L.) leaves at various temperatures

Sage (*Salvia officinalis* L.) leaves are obtained from local communities of Trebinje, East Herzegovina. Sage leaves are air dried, milled and sieved using sieve set (Erweka Apparatebau GmbH, Germany). All extraction experiments are performed using samples with mean particle diameter of 0.44 mm. In a typical run, 60 g of sage leaves sample is loaded into the extractor at 10 MPa and temperatures of 40, 50 and 60 °C. The mass flow rate of carbon dioxide is set at 3.23×10^{-3} kg/min and the separator is set at 1.5 MPa and 25 °C. The yield of sage leaves extract is determined at different extraction times.

2.3. Supercritical extraction of jatropha (*Jatropha curcas* L.) seeds

Jatropha (*Jatropha curcas* L.) seeds used in this study are obtained from local communities of Perak, Malaysia. They are dried, milled and sieved to a mean particle diameter of 0.90 mm to be used in all extraction experiments. In a typical run, about 50 g of jatropha sample is extracted at 30 MPa and 50 °C, with CO_2 flow rate of 3.23×10^{-3} kg/min. Condition of the separator is set at 1.8 MPa and 25 °C. The yield of extraction at different extraction times is determined.

2.4. Modified Reverchon–Sesti Osseo model

The extraction of sage leaves and jatropha seeds with supercritical carbon dioxide is further modelled based on the integration of differential mass balances performed along the extraction bed. The model takes into account that the external mass transfer resistance was negligible and assumes that extraction was uniform along the extraction bed and accumulation of solute concentration in the fluid phase was neglected [23,24]. Modified Reverchon–Sesti Osseo model is developed from Reverchon–Sesti Osseo model as shown in Eq. (1) [21,22,25]:

$$Y = 100\left(1 - e^{(-k_p t)/((1-\varepsilon)V\rho/(W) + k_p t_i)}\right) \tag{1}$$

where Y, ε, V, ρ, k_p, t_i and t represent normalized extraction yield (%), bed porosity of plant material, extractor volume (m^3), fluid density (kg/m^3), volumetric partition coefficient of the extract between solid and fluid phase at equilibrium, internal diffusion time (s) and extraction time (s), respectively.

Assuming that $(1-\varepsilon)V\rho/(W) \ll k_p t_i$, the expression can be neglected and Eq. (1) is simplified into Eq. (2):

$$Y = 100\left(1 - e^{-t/(t_i)}\right) \tag{2}$$

Eq. (2) is known as the final form of Reverchon–Sesti Osseo model.

In order to avoid the complex evaluation of the internal diffusion time, t_i in Eq. (2), the equation is modified based on the assumption that for certain extraction system, t_i could be approximated as constant. This assumption allows one to assert the following Eq. (3):

$$-\frac{t}{t_i} = at + b = Z \tag{3}$$

where a and b are constant and correction term, respectively. Hence, combining with Eq. (3), Eq. (2) can be expressed as

$$Z = \ln\left(1 - \frac{Y}{100}\right) \tag{4}$$

Combining Eqs. (2) and (3), the modified Reverchon–Sesti Osseo model Eq. (5) is obtained as:

$$Y = 100\left(1 - e^{at+b}\right) \tag{5}$$

The modified Reverchon–Sesti Osseo model equation is used to compare with the experimental data (normalized extraction yield) obtained in the study. The normalized extraction yield can be determined by using Eq. (6):

$$Y = 100 \times \frac{y}{y_{\max}} \tag{6}$$

where y and $y_{\max}$ refers to extraction yield (g extract/100 g of sage leave or jatropha seed sample) and maximum extraction yield (g extract/100 g of sage leave or jatropha seed sample), respectively.

Based on experimental results, Z values are computed using Eq. (4) and based on the calculated values of Z coefficient, values of parameters a and b in Eq. (3) are determined numerically from the line of best fit. The errors are quantified by defnining average absolute relative deviation; $S_{x,y}$:

$$S_{x,y} = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(y_i - y_{\mathrm{mod},i})^2} \tag{7}$$

where n is the number of experimental points, y_i is the yield determined by experimental point i, and $y_{\mathrm{mod},i}$ is the yield obtained by the model at point i.

3. Results and discussion

3.1. Supercritical extraction of sage (*Salvia officinalis* L.) leaves at various temperatures

The extraction kinetics of supercritical carbon dioxide extraction of sage leaves for temperatures of 40, 50 and 60 °C are shown in Table 1. The normalized extraction yields and Z values are computed based on Eqs. (6) and (4), respectively.

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Table 1
Extraction yield of sage leaves with respect to extraction time.

Extraction time (min)	40 °C			50 °C			60 °C		
	Extraction yield, y (g/100 g sage)	Normalized yield, Y (%)	Z	Extraction yield, y (g/100 g sage	Normalized yield, Y (%)	Z	Extraction yield, y (g/100 g sage)	Normalized yield, Y (%)	Z
30	1.71	38.46	−0.49	1.44	47.84	−0.65	1.91	58.67	−0.88
90	2.63	59.23	−0.90	2.07	68.73	−1.16	1.72	53.00	−0.76
150	3.00	67.57	−1.13	2.19	72.64	−1.30	1.98	60.98	−0.94
210	3.28	73.78	−1.34	2.22	73.77	−1.34	2.22	68.43	−1.15
270	3.37	75.77	−1.42	2.47	81.97	−1.71	2.33	71.79	−1.27
390	3.83	86.21	−1.98	2.78	92.23	−2.56	2.72	83.68	−1.81
510	4.18	94.15	−2.84	2.90	96.22	−3.27	3.08	94.76	−2.95
600	4.44	100	–	3.01	100	–	3.25	100	–

Table 2
Values of a, b, R^2 and $S_{x,y}$ for extraction of sage leaves at different temperatures.

Temperature (°C)	a	b	R^2	$S_{x,y}$
40	−0.0045	−0.3823	0.9717	3.241
50	−0.0053	−0.4748	0.9688	3.883
60	−0.0042	−0.3926	0.8754	7.958

Based on Eq. (3), constant a and correction term b, with values of R^2 and average absolute relative deviation, $S_{x,y}$ are tabulated in Table 2.

After a and b values are estimated, the modified Reverchon–Sesti Osseo model (Eq. (5)) are used to compare with the experimental data. Fig. 1 shows the comparison of the model with experimental data at temperatures 40, 50 and 60 °C.

The results show that the yield of extraction increases with extraction time, with the highest extraction yield obtained at temperature of 40 °C with 4.44 g/100 g sage at extraction time of 600 min. As the extraction temperature increases from 40 to 60 °C, the maximum extract yield decreases from 4.44 g/100 g sage to 3.01 g/100 g sage and 3.25 g/100 g sage at 50 °C and 60 °C, respectively. This may be due to thermal degradation of the organic compounds at higher temperatures, hence decreasing the extract yield. Temperatures above 60 °C are not recommended for extraction of organic compounds from plant materials, as those compounds might be transformed at high temperatures [13,14,26]. Another possible reason that explains the decrease in extract yield is the decrease of solvent power of supercritical CO_2 at increased temperature [4]. The modified Reverchon–Sesti Osseo model showed a good agreement with the experimental data at the investigation temperatures of 40 and 50 °C, with standard error of regression of 3.241 and 3.883, respectively. As the temperature increased to 60 °C, the model is less successful in describing the kinetic data of the extraction, with error of 7.958, most probably due to the onset of thermal degradation of organic compounds at this temperature.

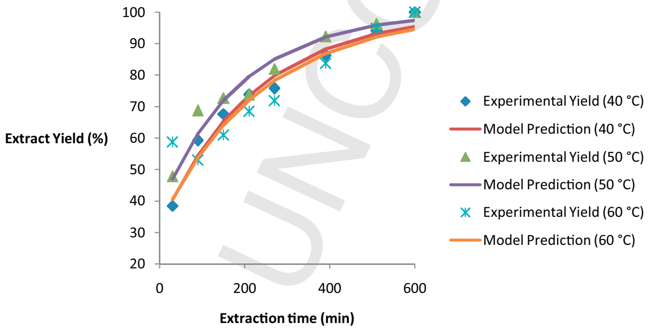

Fig. 1. Extraction of sage leaves: Comparison of experimental data with modified Reverchon–Sesti Osseo model at 40, 50 and 60 °C.

3.2. Supercritical extraction of jatropha (*Jatropha curcas L.*) seeds

In another study, the modified Reverchon–Sesti Osseo equation is also used to model the supercritical extraction of jatropha seed at pressure of 30 MPa and temperature of 50 °C. Table 3 shows the extract yield with respect to extraction time. The normalized extraction yields and Z values are computed based on Eqs. (6) and (4), respectively.

From Table 3, the Z values are plotted as a function of extraction time according to Eq. (3) and parameters a and b are obtained; $a = -0.1142$ and $b = 0.2689$. R^2 value of 0.9681 and calculated value of standard error of regression, $S_{x,y}$ of 5.607 showed that the modified Reverchon–Sesti Osseo model is able to give a good fit to the data obtained experimentally. The prediction of the model and the obtained experimental data is compared and shown in Fig. 2.

The yield of the extract is noticed to be fast for the first 12 h, as sharp increase in the extraction yield is observed. This is most probably caused by the convective mass transfer of extracted compounds from broken cell, which happens at a faster rate. As the extraction time is prolonged, the increase in extract yield is lesser, most probably due to slow diffusion of dissolved substances that

Table 3
Extraction yield of jatropha seeds with respect to extraction time at 50 °C.

Extraction time (h)	Extraction yield, y (g/100 g jatropha)	Normalized yield, Y (%)	Z
2	2.73	14.04	−0.15
3	3.93	20.21	−0.23
4	4.84	24.88	−0.29
5	5.96	30.64	−0.37
6	7.17	36.86	−0.46
7	8.66	44.52	−0.59
8	9.40	48.33	−0.66
9	10.03	51.57	−0.73
10	10.74	55.22	−0.80
11	11.54	59.33	−0.90
12	12.40	63.75	−1.01
13	13.14	67.56	−1.13
14	13.69	70.39	−1.22
15	14.22	73.11	−1.31
16	14.79	76.04	−1.43
17	15.27	78.51	−1.54
18	15.73	80.87	−1.65
19	16.10	82.78	−1.76
20	16.60	85.35	−1.92
21	16.91	86.94	−2.04
23	17.70	91.00	−2.41
25	18.22	93.68	−2.76
27	18.73	96.30	−3.30
29	19.45	100	–

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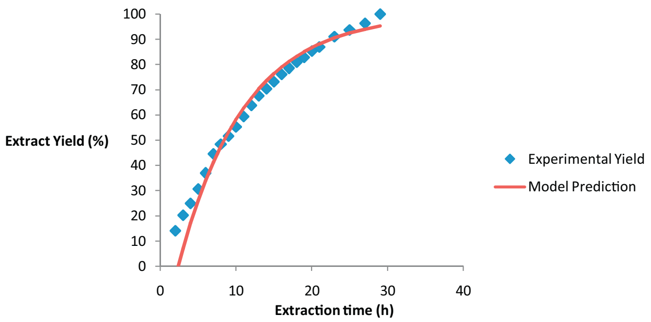

Fig. 2. Extraction of jatropha seeds: Comparison of experimental data with modified Reverchon–Sesti Osseo model at 50 °C.

are bound to the matrix structure of the plant cells. This trend is also observed in other work [11].

Compared to the modelling of sage leaves extraction, modified Reverchon–Sesti Osseo model is seen to be less successful in modelling the extraction extraction of oil from seed, as seen from the larger error obtained in extraction of jatropha seeds. Similar observation is also reported in literature [24,27].

3.3. *Feasibility and limitation of the model*

Generally, the modified Reverchon–Sesti Osseo model used in this study is able to give relatively good agreement with the obtained experimental data. However, it is important to note that the modified Reverchon–Sesti Osseo model predicts a solute yield that is different from zero at the beginning of the extraction process. This non-zero extract yield may be due to washing of superficial solute from broken surface cells where the easily accessible solute particles available on the external surface of the sample matrix gets extracted (positive deviation) [28]. Another possible explanation for non-zero extract yield may be due to slow extraction and diffusion mechanisms before reaching the equilibrium state (negative deviation) [28]. These deviations are dependent on the cell structure of the natural material [28].

From Figs. 1 and 2, it is observed that the residuals of the obtained kinetic data and model's prediction are negative at the beginning and end, and slightly positive in between. The negative residuals at the beginning may be due to the initial constant extraction rate (CER) regime where the easily accessible solute on the external surface of the sample is being washed out [4,28]. The slightly positive residuals may be due to the external mass transfer resistance which is neglected in the model [23,24], hence causing the experimental yields to be slightly lower than the model's predictions. Towards the end of the plots in Figs. 1 and 2, negative residuals are observed most probably because the extraction process has entered the regime of falling extraction rate (FER), which deviates from description of the modified Reverchon–Sesti Osseo model [4,28].

The modified Reverchon–Sesti Osseo model is able to produce relatively good agreement with the kinetic data obtained within the range of experimental durations explored in this study. However, it is essential to examine the validity and limitation of this model beyond the experimental duration (10 h for sage leaves and 29 h for jatropha seeds) in our future work.

4. Conclusions

In conclusion, extraction of sage (*S. officinalis* L.) leaves and jatropha (*J. curcas* L.) seeds using supercritical carbon dioxide as solvent under various conditions are investigated. The influence of temperature on the extraction yield of sage at 40, 50 and 60 °C and 10 MPa are studied and the highest yield of 4.45 g/100 g of sage extract is obtained at 40 °C. Besides, the extraction kinetics of the process is found to be well modelled by the modified Reverchon–Sesti Osseo model. In another study involving the extraction of jatropha seeds using supercritical carbon dioxide at 50 °C and 30 MPa, the extraction kinetics of the process is also found to be in good agreement with the modified Reverchon–Sesti Osseo model. The modified Reverchon–Sesti Osseo model, which is simplified from its original form has given a good representation of the kinetics of supercritical carbon dioxide extraction of sage leaves and jatropha seeds within the experimental conditions and durations explored in this study. In future, its application may be tested and extended on the supercritical extraction of other bio-materials to further explore its robustness and limitations.

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