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Kinetic study of peanut seed oil extraction with supercritical CO_2

Estudo cinético da extração de óleo da semente de amendoim com CO_2 supercrítico

Estudio cinético de extracción de aceite de semilla de maní con CO_2 supercrítico

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Abstract

Due to the need to create new technologies for the production of biofuels, there are techniques to perform oil extraction from oilseeds, such as the supercritical extraction method using CO_2, where the seed used to carry out the study (peanut) is subjected to CO_2 at high pressures and temperatures, thus becoming a supercritical fluid and carrying out the process of extracting the oil from the seed to produce biodiesel more efficiently. There is another extraction technique in which organic solvents are used. Still, this method has some difficulties that end up causing damage to the environment as it is a process that consumes a significant amount of time and energy. Peanut is the world's fourth-most cultivated oilseed, with large plantations in the Americas, Africa, and Asia. Its planting is carried out to produce grains, oil, and bran. CO_2 has interesting physicochemical properties since it is an inert, non-polar, non-flammable, odorless, tasteless gas and has critical parameters and low value. It has a critical pressure of 72.01 bar and a critical temperature of 31.1 °C. The present work presents data from a kinetic study of the supercritical extraction of peanut oil under pressure and temperature conditions of 200 bar, 280 bar, 40 °C, and 60 °C. This project uses an experimental matrix to help carry out the experiments. At the end of the experiments, we obtained a yield of 30% in the supercritical method (80 min) and a percentage yield of 26% by Soxhlet using ethanol as a solvent for 480 minutes.
Keywords: Kinetic study; Biodiesel; Supercritical extraction; Supercritical fluid; CO_2; Peanut.

Resumo

Devido à necessidade de criar novas tecnologias para a produção de biocombustíveis, existem técnicas para realizar a extração de óleo de oleaginosas, como o método de extração supercrítica utilizando CO_2, onde a semente utilizada

para realizar o estudo (amendoim) é submetida a CO_2 em altas pressões e temperaturas, tornando-se um fluido supercrítico e realizando o processo de extração do óleo da semente para produzir biodiesel com mais eficiência. Existe outra técnica de extração na qual são usados solventes orgânicos. Ainda assim, esse método apresenta algumas dificuldades que acabam causando danos ao meio ambiente por ser um processo que consome uma quantidade significativa de tempo e energia. O amendoim é a quarta oleaginosa mais cultivada do mundo, com grandes plantações nas Américas, África e Ásia. Seu plantio é realizado para a produção de grãos, óleo e farelo. O CO_2 possui propriedades físico-químicas interessantes, pois é um gás inerte, não polar, não inflamável, inodoro, insípido e possui parâmetros críticos e baixo valor. Tem uma pressão crítica de 72,01 bar e uma temperatura crítica de 31,1 °C. O presente trabalho apresenta dados de um estudo cinético da extração supercrítica de óleo de amendoim sob condições de pressão e temperatura de 200 bar, 280 bar, 40 °C e 60 °C. Este projeto utiliza uma matriz experimental para ajudar a realizar os experimentos. Ao final dos experimentos, obteve-se um rendimento de 30% no método supercrítico (80 min) e um rendimento percentual de 26% no Soxhlet utilizando etanol como solvente por 480 minutos.
Palavras-chave: Amendoim; Extração supercrítica; Fluido supercrítico; CO_2; Estudo cinético.

Resumen
Debido a la necesidad de crear nuevas tecnologías para la producción de biocombustibles, existen técnicas para realizar la extracción de aceite a partir de semillas oleaginosas, como el método de extracción supercrítica mediante CO_2, donde la semilla utilizada para realizar el estudio (maní) se somete a CO_2 a altas presiones y temperaturas, convirtiéndose así en un fluido supercrítico y llevando a cabo el proceso de extracción del aceite de la semilla para producir biodiesel de manera más eficiente. Existe otra técnica de extracción en la que se utilizan disolventes orgánicos. Aún así, este método tiene algunas dificultades que terminan causando daños al medio ambiente ya que es un proceso que consume una cantidad importante de tiempo y energía. El maní es la cuarta semilla oleaginosa más cultivada del mundo, con grandes plantaciones en las Américas, África y Asia. Su siembra se realiza para producir granos, aceite y salvado. El CO_2 tiene propiedades fisicoquímicas interesantes ya que es un gas inerte, no polar, no inflamable, inodoro, insípido y tiene parámetros críticos y de bajo valor. Tiene una presión crítica de 72,01 bar y una temperatura crítica de 31,1 °C. El presente trabajo presenta datos de un estudio cinético de la extracción supercrítica de aceite de maní bajo condiciones de presión y temperatura de 200 bar, 280 bar, 40 °C y 60 °C. Este proyecto utiliza una matriz experimental para ayudar a llevar a cabo los experimentos. Al final de los experimentos se obtuvo un rendimiento del 30% en el método supercrítico (80 min) y un porcentaje de rendimiento del 26% por Soxhlet utilizando etanol como solvente durante 480 minutos.
Palabras clave: Maní; Extracción supercrítica; Fluido supercrítico; CO_2; Estudio cinético.

1. Introduction

The peanut (Arachis Hypogaea), also known as peanut, is an edible seed of a legume with excellent nutritional properties; its grains contain a high concentration of proteins and lipids, making them a good source of minerals like phosphorus, calcium, magnesium, and potassium (Fazelifar et al., 2021).

Peanuts are made up of approximately 70% seeds and 30% husk. Peanut seeds have a high content of lipids and protein and are a source of minerals such as calcium, potassium, magnesium, and phosphorus. Peanut oil has a rich composition of fatty acids, which places it among the most important sources of vegetable oil (Fogang et al., 2014).

Peanut is the world's fourth most cultivated oilseed, being planted on a large scale in the American, African, and Asian continents, where it is planted to produce grains, oil, and bran, among other things (Arya et al., 2016). China and India account for around 50% of the world's total peanut production (Arya et al., 2016).

There are several extracting oil from seeds to produce biodiesel and other biofuels (Arce et al., 2018; Papa Matar Ndiaye, 2004; Paraízo, A.; Junior, E.; Paraízo, 2005; Pinto et al., 2012; Serres et al., 2015). Among the oil extraction processes, there is the supercritical extraction method, in which a fluid in the supercritical state, such as CO_2, for example, is used, since its use does not leave residues after the extraction process is completed, where the only residues present will be those of the raw material used (Toledo et al., 2019).

The supercritical extraction method using CO_2 has several advantages: its extraction has a high purity content, does not use organic solvents, has a simple separation, has high selectivity for a particular compound in the solute, is a non-toxic solvent, and has a low critical temperature and pressure (31.1 °C and 72.01 bar) (Souza et al., 2008). Due to the concern about reducing the use of fuels derived from petroleum, so-called biofuels, such as biodiesel, have been developed (Araújo et al.,

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2012; Ferreira et al., 2011; Pinto et al., 2012; D. I. S. Silva et al., 2013).

In producing biodiesel, organic raw materials are used, such as peanuts, are used as oilseed to produce biodiesel (Toledo et al., 2019). One of the problems for producing biodiesel from peanuts is the extraction of their oil. However, it is possible to use the supercritical extraction method, which has significant efficiency for this purpose (Andersen & Gorbet, 2002; Toledo et al., 2019).

2. Methodology

Samples preparation

The peanuts were obtained from a supermarket in the Pontal do Paranapanema region. The matrices were dried in an oven at 60 °C for 72 h, crushed in a blender, and sieved. Carbon dioxide (99.9% by weight - liquid phase) was purchased from Air Liquid S.A. (Brazil), and ethanol (98% by weight) was obtained from Sigma-Aldrich.

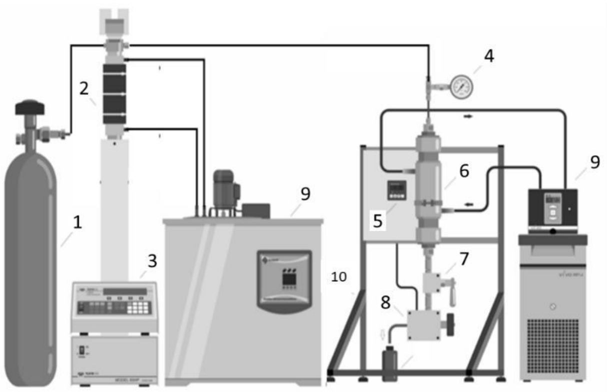
Soxhlet extraction

The organic solvent (ethanol) extractions were performed using a Soxhlet extractor according to AOAC method 920.39. A filter paper cartridge containing about 5 g of crushed pequi seeds was put into the extractor, coupled to a flask containing 150 mL of solvent for recycling over the sample. The material was subjected to extraction for eight hours. The organic solvent (ethanol) was removed from the extract using rotary vacuum evaporation at 323.15 K, and the total lipid content was determined gravimetrically using an analytical balance. Extractions were done three times, and the results are shown as the mean, standard deviation.

Supercritical CO_2 extraction

The experiments were conducted using a CO_2 cylinder, two thermostatically regulated baths, an Isco 500D syringe pump, a jacketed extraction vessel (1.91 cm in diameter and 16.8 cm in height), and an absolute pressure transducer (Smar LD301, Brazil) (see Figure 1).

Figure 1. Scheme of an experimental supercritical extraction unit: 1 - CO_2 cylinder, 2 - Syringe pump, 3 - Thermostatic bath, 4 - Pressure indicator, 5 - Temperature controller / indicator, 6 - Extractor, 7 - Valve, 8 - Needle-type valve attached to an aluminum jacket for heating. 9 - Thermostatic bath. 10 - Aluminum structure.



Source: Authors.

Additional information about the instrument and approach used in the experiment may be found in past studies (Garciaet al., 2012; Gonçalves et al., 2013; Lemos et al., 2012; Rogério Favareto, n.d.; M. O. Silva et al., 2016).

The extractor was uniformly fed with 1 g of material (peanut in shell), and the remaining space of the extraction cell was filled with glass beads (inert bed). Thus, the CO_2 supplied to the extractor initially passed through the inert bed and then through the ground seed. After reaching the desired temperature for extraction, the pump and the extractor were pressurized simultaneously. After operating pressure was reached, the system was allowed to stand for 30 minutes to reach equilibrium and ensure that the solvent was saturated at the beginning of the extraction. The extractions were carried out for pressures of 200, 240, 280 bar, temperatures of 40 °C, 50 °C, 60 °C and CO_2 flow rate of 2.0 $mL.min^{-1}$, from a 2^2 factorial design with triplicate at the central point, as shown in Table 1. The flow rate of 2.0 $mL.min^{-1}$ of solvent was controlled by a micrometric valve (Parker Autoclave Engineers, USA) maintained at 90 ºC. The total extract was collected in glass vials and weighed in six initial cycles of 5 min and five cycles of 10 min (total 90 min). The extraction yield was determined gravimetrically by an analytical balance and calculated as a ratio between the total mass extracted and the initial mass of the seeds in the extractor (dry basis).

Table 1. Factors and levels for 2^2 factorial design with center point.

Factors	Symbols	Units	Levels		
			-1	0	+1
Temperature	T	ºC	40	50	60
Pressure	P	bar	200	240	280

Source: Authors.

Statistical analysis

The data was subjected to analysis of variance (ANOVA) at 5% significance, followed by the Tukey test, and the main effects and interactions were computed using Design expert, Software, version 12 (Stat-Ease, 2008). Statistica Software version 8.0 (StatSoft, 2008) was used to compute the main effects and interactions, which examined the influence of independent factors on response.

3. Results and Discussion

The experiments were carried out with shelled peanuts. The experimental conditions and the total yield for extraction by supercritical CO_2 (2.0 $mL.min^{-1}$) and by organic solvent (ethanol) are presented in Table 2. The yields of the supercritical extraction experiments varied between 27% and 36% and the extractions performed using the conventional method (by organic solvent) reached 26% of lipid material.

Table 2. Experimental conditions employed and yields obtained for the extraction of burrito metabolites from supercritical carbon dioxide.

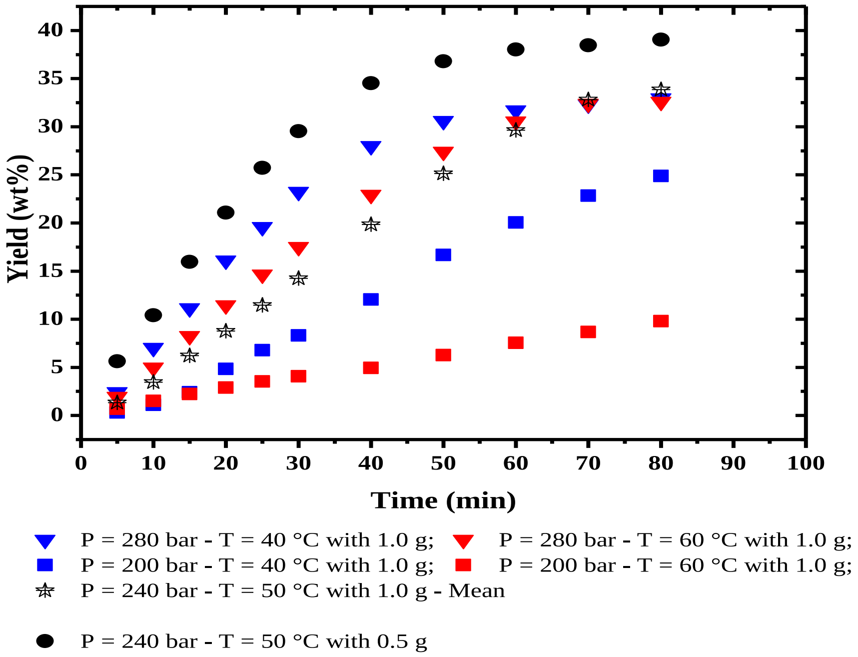
Experiment	Temperature (°C)	Pressure (bar)	Time (min)	Oil Yield (wt%)
1	40	280	80	36.2
2	60	280	80	34.6
3	40	200	80	28.9
4	60	200	80	27.3
5-7	50	240	80	31.85 ± 0.3 [a]
8-10	50	Atmospheric	480	26.1% ± 0.2 [a]

[a] Mean ± standard deviation (n =3). Source: Authors.

There are two stages of mass transfer and three distinct extraction periods in supercritical extractions. In the first period, easy access to oil extraction takes place. This step depends on the solubility in the fluid phase and is characterized by a linear curve with a slope close to the solubility value of the oil in the solvent. Then there is a decrease in the extraction rate, followed by the extraction of the barely accessible oil, which is controlled by an internal diffusion mechanism. Later, the extraction curve becomes almost linear in the third period, with an extraction rate much lower than in the first period.

In general, most of the lipid extracts were removed within the first 50 minutes. After this period, the amount of extract withdrawn is significantly reduced. In Figure 2, it can be seen that there was a change in the slope of the extraction kinetic curves. Changes in slope occur due to variations in the convective and diffusive mass transfer mechanisms. The convection mechanism has a more significant influence in the fluid phase than the diffusion mechanism due to the speed of the mass transfer process. With the gradual removal of the lipid material, a discontinuity effect begins in the surface layer. At the start of this split, the extraction rate drops because of the diffusion mechanism.

Figure 2. Experimental and modeled kinetic curves for peanut seed extractions by supercritical CO_2.



Source: Authors.

It was verified in the experiments that the time of 80 minutes was not enough to extract all the lipid content of the peanut seed. The kinetic curves did not present a maximum plateau, and, consequently, it was not possible to reach the stability curve. This is due to the low flow of CO_2 used in the extraction. The low flow of CO_2 chosen for application in the extraction experiments is a consequence of the dimensional limitations of the experimental apparatus. Therefore, an experiment was done with 0.5 g (half the amount used in the experimental matrix) to see if time affected extractions.

As shown in Figure 2, it can be seen that the extraction using half of the peanut seed mass was faster than the other extractions. Thus, the extraction time (or CO_2 flow rate), related to the seed mass inside the extractor, is a significant variable. Thus, since the mass of the lipid matrix within the extractor vessel is invariable, the flow rate and extraction time must be carefully considered.

Extraction yield can be shown by looking at the yield response surface graph, as shown in Figures 3 and 4. The response surface graph shows that pressure and temperature play a significant role in extraction yields.

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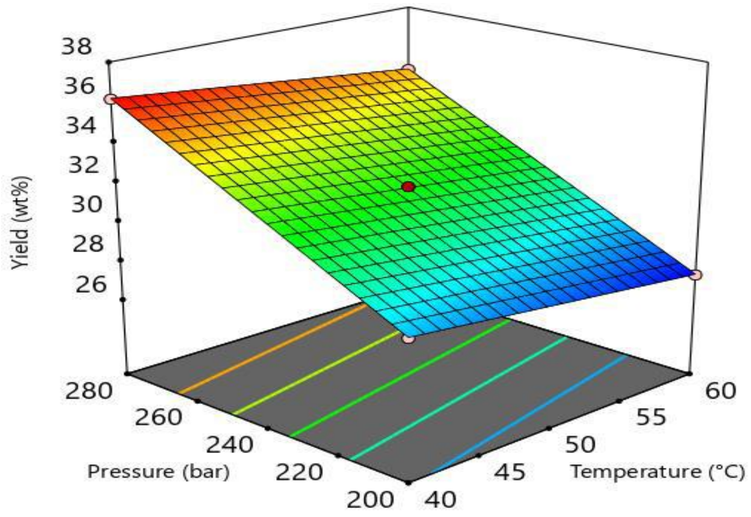
Figure 3. Response surface referring to peanut oil extraction yield as a function of temperature and pressure levels with constant flow rate of 2.0 $mL.min^{-1}$.

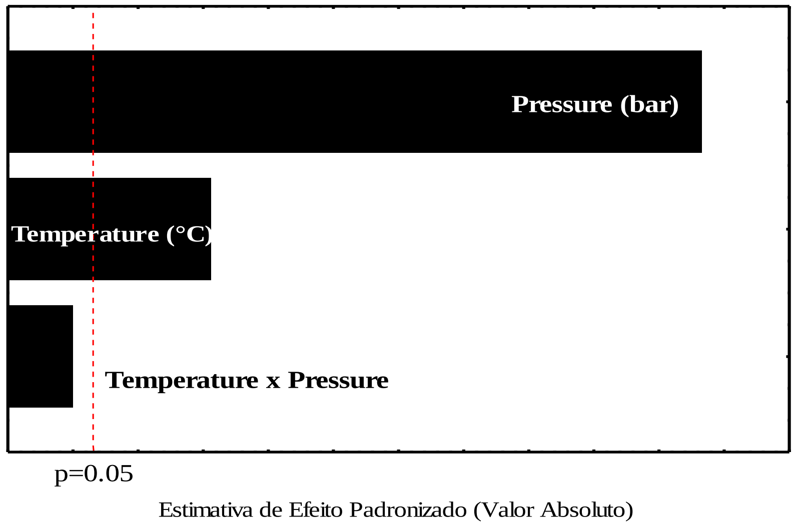


Source: Authors.

The statistical analysis of the experimental results, as shown in the Pareto graph, Figure 4, indicated that the two variables, temperature (T) and pressure (P) were significant (p-value < 0.05) for the yield.

Pressure is a major influencing factor in peanut seed extractions. Temperature, although extremely important, was not the factor that significantly altered the extraction yields. Pressure reduces the distance between molecules and makes CO_2 and the sample more likely to interact, which can help with convective mass transfer. In general, the yields obtained at the two temperatures studied were similar, and the energy spent on extractions at 60 °C does not lead to more extracts, which means that this is not a good thing.

Figure 4. Pareto Chart: Estimation of the linear effects of the variables.



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These variations in income can be verified through a statistical analysis (see Table 3). The analysis indicated a significant linear model for the temperature and pressure factors (see Equation 9), where T corresponds to temperature, P corresponds to pressure.

$$Yield\ (\%) = 31.79 - 0.8000\ T + 3.65\ P \qquad \text{Eq. (2)}$$

Table 3. Analysis of variance data for extracts obtained using 2^2 factorial design for carbon dioxide extractions.

Source	Sum of Squares	Degrees of freedom	Mean Square	F-value	p-value	R^2
Peanut Seed						
Model	55.85	3	18.617	3257.91	< 0.0001	0.9997
T	2.56	1	2.56	448	0.0002	
P	53.29	1	53.29	9325.75	< 0.0001	
T.P	0	1	0	0	1	
Pure Error	0.0000	2	0			
Cor Total	55.87	6				
T = Temperature; P = Pressure.						

Source: Authors.

4. Conclusion

The supercritical extraction using CO_2 from peanut seeds indicated that pressure is the most relevant factor for obtaining the highest oil yields. The best extraction yields occurred at 240 and 280 bar pressures, regardless of the temperature used. The temperature is a factor of minor influence in the extraction. In this way, it is verified that the energy expenditure of the extraction with the elevation of temperatures is not necessary. Furthermore, it was observed that the time and flow of CO_2 extraction are determinant variables for obtaining the total lipid content. By comparing the yields obtained between the conventional extraction (with ethanol) and the supercritical (with CO_2), it is verified that the supercritical extraction presents a significant superiority since the conventional extraction reached 26% and the CO_2 extraction reached values above 30%. Although the difference in yield is not significant, it is observed that the extraction time used for the two methods is significant. While the conventional method required 480 min to reach a 26% yield, the supercritical extraction achieved yields more significant than 30% in 80 min.

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