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An examination of Supercritical fluid extraction from oilseeds

Hamid Nikoyeh

Inspector expert of the International Inspection Company Rahavard Sanat Atrak

*Author Email: Rahavardsanatatrak@gmail.com

Abstract: Supercritical fluid extraction is a novel technique for extracting compounds from solid and liquid tissues with sufficient purity. Oil extraction from oilseeds is one of the uses for the supercritical method. In this study, a supercritical extraction device with pressure tolerance of 400 bar was conceived and constructed. With this apparatus, oil was extracted from rapeseed and sesame oil seeds under various operating circumstances. 200 bar pressure, 308 degrees Kelvin temperature, 0.15 mm particle size, 5 g/min flow rate, and 45% bed porosity were the optimal working parameters for extraction. In 140 minutes, the best percentage of oil extraction from rapeseed was approximately 49% and from sesame oil seed approximately 61%. Two mass transfer models were compared to the experimental results obtained. The first model represented damaged and healthy cells, whereas the second model was a two-phase mathematical model whose parameters were found by numerically solving the model. The modeling results indicate that the seed and substrate model described the laboratory data more accurately than the broken and healthy cell model.

Keywords: oil seeds, supercritical extraction, mass transfer, sesame oil, mathematical model, supercritical flow.

Introduction

Today, the separation of materials from one another plays a significant role in numerous industries and has led to numerous advancements in the field of material separation. Today's technological advancement is largely attributable to separation techniques. Purification of raw materials or product purification compels researchers to look every day for improved separation problem-solving techniques. Increased energy costs for separation processes such as distillation, as well as environmental organizations' monitoring of the consumption of harmful solvents and the non-production of undesired byproducts, have prompted scientists to seek out novel separation procedures. In recent decades, supercritical fluid extraction (SFE) has been one of the approaches gaining the most attention. The great dissolving power of supercritical fluid is one of the primary reasons why researchers want to develop this technique. In comparison to other separation processes, such as distillation to extract temperature-sensitive compounds, this method reduces energy and time consumption, as well as the consumption of harmful solvents. In order for this technology to be considered in the pharmaceutical and food industries, etc. [1].

A supercritical fluid is any substance placed at a temperature and pressure above its critical temperature and pressure. When we approach the critical point on the gas and liquid balance curve, the density of the liquid falls as a result of thermal expansion, whilst the density of the gas increases due to the increase in pressure. At the critical point, the densities of both phases are identical, and the distinction between liquid and gas is eliminated. Critical temperature T and critical pressure P define this point. This region is referred to as the supercritical region. In this region, fluid characteristics are between those of gases and liquids. This characteristic has led to the

consideration of supercritical fluids for material extraction. Because the density of the supercritical fluid is one thousand times that of the gaseous state, the dissolving ability of supercritical fluids is greater than that of gases and close to that of liquids. In contrast, supercritical fluids have gas-like permeability and less viscosity than liquid solvents. These two parameters that regulate mass transfer accelerate the operation of supercritical fluids [2].

To produce optimal conditions in the supercritical process, either the dynamic-static approach or a mix of the two can be used. In the dynamic method, the sample is subjected to a constant flow of new supercritical fluid, the conditions and intensity of which are determined by the pressure and pressure reduction equipment. is obtained After a predetermined amount of time, the outlet valve is opened and the extracted fluid and materials are deposited into the extraction container. In this discipline, the most common procedure is a combination of the two; typically, static extraction is accompanied by a few minutes of dynamic extraction. This strategy is more effective than the two preceding ones. Before increasing the pressure, polarity modifiers can be added to the extraction cell with relative ease during stannic extraction, which has the benefit of consuming less fluid. It can also be used to measure the solubility of substances [3]. The dynamic method is more effective than the static method due to the enormous volume of gas moving through. With this approach, complete extraction is possible. A major downside of the dynamic approach is its high solvent consumption. In supercritical extraction, the analysis of the extracted sample is one of the factors that must be considered. During the extraction procedure, if the sample is captured inside the trap and then examined by any other equipment, the sample will be contaminated.

The SFE system is discontinuous, but if the sample is sent directly to the analyzer during extraction, the aforementioned procedure will be continuous. With the discontinuous system, just the sample extraction and collection conditions are managed, whereas in the continuous system, the chromatograph settings must also be regulated [4]. for samples with a high concentration, the discontinuous method is preferable because to the low fluid flow rate of the continuous method. In the continuous approach, it is possible to evaluate very small sample amounts since the extracted material can be completely transferred to the chromatograph machine.

In the discontinuous method, the acquired sample can be analyzed using any technique or even many instruments.

The discontinuous method, which is more engaging, is executed as follows.

- Collecting by means of Halal
- Collection using absorbents or a combination thereof • Collection in an empty container
- Cold collection

The solubility of the solute in the collecting solvent, the volume of the solvent, the residence time of the gas bubbles in the solvent, the size of the gas bubbles, and the temperature of the solvent all influence the collection efficiency when using a solvent. A collection jar with a narrower inner diameter provides a longer path for the bubbles, hence minimizing the amount of solvent required. A capillary tube with a smaller inner diameter can be employed as a flow limiter to lessen the intensity of the flow. Quick evacuation of the gas accelerates the rate of solvent evaporation from the collection vessel. Cooling the expanded gas reduces the temperature of the solvent, which reduces solvent evaporation and is required for extractions involving prolonged solvent addition durations. The use of solvent-assisted collection permits the chromatographic or other analytical examination of extracted chemicals. In the majority of instances, solvent-assisted collection has proven to be the most effective collection approach [5].

To reduce losses, larger collection containers are required to capture small amounts of soluble material. Additionally, these losses are decreased by filling the empty container with glass, metal, or wool.

With adsorbent-assisted collection, removing the adsorbed material requires an extra step. In the case of collection with the aid of an adsorbent, consideration should also be given to its removal and washing, in addition to its ability to absorb substances. To get a higher collection efficiency for a wider group of materials with varied polarities, it is preferable to utilize a variety of sorbents.

In cold collection, the vessel is typically chilled to the point where the fluid expands and is not cooled, and the extracted material settles to the vessel wall. Depending on whether the extracted material is separated from the fluid, the collecting temperature will vary. Due to crystal formation, some of the extracted components may be lost during cold collection, even if they have low volatility. Using a sealed cold liquid collector can alleviate this issue. With this cooling approach, it is required to give sufficient heat to the pressure relief valve and to prevent the formation of two phases and the clogging of the pressure relief valve.

In comparison to older techniques, supercritical fluid extraction of essential oils and natural chemicals from natural materials is one of the most relevant issues. Hence, numerous models have been presented to justify this procedure. In 1994, Sovova and his colleagues investigated the effect of different pressure, temperature, and solvent flow intensity parameters on the extraction of essential oil from cumin seeds, compared its analysis by supercritical fluid with water distillation, and analyzed the extraction using a mass transfer model [6]. In 1995, Sovova and his coworkers published a generalized plug flow model. This article [7] investigates the extraction of oil from black pepper seeds using supercritical fluid in a bed. These tests were conducted at temperatures of 30, 40, and 50 degrees Celsius and pressures of 150, 200, and 300 bar. The model offered explains the laboratory data. The description of this model can be found in the section on process modeling.

In 1997, Reverchon extracted oil from sunflower oil seeds using supercritical fluid at a pressure of 250 bar and a temperature of 40 °C in a semi-industrial pilot and a laboratory device, and based on the experimental data, he presented a model depicting the equilibrium between the solid and fluid phases that control the extraction process [8].

In 1998, Guderzania and his coworkers suggested a two-phase model consisting of a solid phase and a supercritical phase. This model relies on the equilibrium of mass transfer between the fluid and solid phases. Hence, axial dispersion is regarded as one of the most essential characteristics. In addition, this model takes penetration and film resistance into account. This model can be used to demonstrate how various operational conditions affect the extraction process [4]. In the year 2000, Reverchon and his coworkers extracted oil from flowers under various settings and studied the influence of each parameter on pressure, temperature, and particle size. The obtained laboratory data were utilized to validate the suggested model. The only desired parameter in this model is the internal mass transfer constant [10], which was derived from the mass transfer balance.

In 2000, Magoulas and his colleagues produced an experimental model and two mass balance models for extraction with supercritical fluid. The mass balance models consisted of a simple model and a generalized model. They provide an empirical link comparable to the one postulated by Mr. Naik. This model was developed using the Long-Muir isotherm [11]. In the year 2000, Reverchon and his colleagues extracted oregano oil using supercritical extraction and developed the provided mathematical model based on the mass transfer balance along the bed. They based their estimations on the removal of essential oils near the leaf's surface and the mass transfer resistance within the leaf's structure. Furthermore, axial scattering is accounted for in this model. The simulation results were also applied to other plant components, and the process's performance was analyzed on a broader scale [12].

In 2003, Gasper and his coworkers presented three models describing the supercritical extraction procedure. This model is developed based on the particle plane's form. The sole difference between two of these three solid phase models, the model (SP) with a single plate model and the model (SSP) with a simple plate model only, is the presence of film resistance. The third type is the single plate fluid phase model (FP/SSP). This model displays both the particle and fluid mass balances. All models demonstrate a strong fit with the laboratory data. The third model, however, provides the best fit [14].

Goto and his colleagues produced nutmeg oil from nutmeg seeds using supercritical fluid at varying pressures and temperatures in 2006. They studied the effect of separation process parameters such as temperature, pressure, carbon dioxide flow intensity, and particle size on the extraction rate of nutmeg oil and described the process using the broken and healthy cell model and the shrinking core model [15]. Samer Haman and his colleagues examined the extraction of oil from cardamom seeds using carbon dioxide and heptane fluid in 2007 and compared the quality of oil extracted using carbon dioxide and supercritical hetane using mass chromatography and liquid chromatography. did [16]. In 2008, Bernardo Gill and his colleagues extracted oil from pumpkin seeds and determined the fatty acid proportion. The percentage of fatty acids was compared between the oil extracted using hexane and ether and the oil extracted using the supercritical technique [17].

Materials used for experiments

The materials examined are rapeseed and sesame oil seeds procured from the Pakan Bazar Company in Isfahan. The solvents used were regular hexane and dichloromethane with a purity of 99, while Metabol and potassium hydroxide were purchased from Merck, Germany. Furthermore, pentadialonic acid was created as a fatty acid standard in the United States by Serva. 99% pure carbon dioxide gas is obtained from the ERISCHGAS firm.

Supercritical extraction device

To execute extraction with supercritical fluid, the following specifications were incorporated into a device.

Process design Due to the high pressures involved in the supercritical extraction process, safety factors must be carefully considered in the design and construction of the supercritical extraction device. The following factors should be taken into account while designing a supercritical unit:

Aspects pertinent to the laboratory

maximizing energy efficiency

Reduction of solvent pressure and its recovering ability

How to enter feed information and load it?

Laboratory space

The system was designed after theoretical studies and consideration of safety concerns and operational factors. First, the capacity and size of the device should be considered, followed by the preparation of the necessary equipment. Multiple containers of varying capacities were designed and manufactured for this machine, with each component designed separately to approximate semi-industrial conditions.

Components of the device are described.

Components necessary for the gas supply source and filter:

The carbon dioxide gas tank is supplied by ERISCH GAS at a pressure of 55 megapascals, and a filter is used at the tank's outlet.

Carbon dioxide gas liquefaction system

Since in the designed system, a pump is used to provide the required pressure, the input fluid must be liquid. The refrigeration cycle used in this project is similar to the normal refrigerator cycle, which includes four parts: compressor, condenser, evaporator, and expansion valve. This cooler has the ability to cool up to 20. The cycle temperature is controlled by a thermometer.

Yamp

In this project, a reciprocating pump model M36 made by Haskell pump company in the USA has been used. The pump consists of a cylinder and piston, which increases the pressure of carbon dioxide by changing the cross-sectional area. Before the pump, a one-way valve has been used to prevent the liquid from returning.

Extraction containers:

Extraction containers are stainless steel pressure tanks that can withstand at least 400 bar pressure. The material of the dishes is made of steel alloy with code L SS-316. A cap-type lid system with teflon washers is considered. It is possible to load with a special stainless steel plate. The extraction container is designed as a double wall. Hot water circulates around the container to bring the container to the desired temperature. In this device, four extraction sides with different diameters and heights are designed and built. The heating and cooling systems of the hot water cycle include heaters, water tank pumps, heating valves, connections and temperature control systems.

In the hot water tank, there is a cylindrical chamber and an electric heater with a power of 2000 watts is placed inside it. The hot water in the tank is controlled by a type 100 thermocouple. The pump used is a heating pump made in Germany and is able to circulate hot water in the system at three different speeds (flow rates) (flow rates). By adjusting the hot water temperature and the pump rotation speed, the temperature of the extraction and separation containers can be controlled. Water pipes are also made of galvanized iron with a size of half an inch.

Valves and fittings

The most important factor in choosing gas valves and connections is the safety of the device, which must be considered. Connections should be in such a way that it has the least leakage. Common valves and fittings in supercritical systems are mostly made of SS-304 SS-316 SS-L 316 alloys. In this project, all valves, elbows, three-way elbows, gas pipes and all fittings are of 316L type with pressure tolerance. 400bar is made.

Temperature and pressure control systems

The temperature control system is installed in different parts of the device, such as in the hot water containers, the inlet and outlet of the extraction containers, and on the pressure relief valve. To control the temperature in the water container, a 100-PT thermocouple is used, and the thermocouple used in the pressure relief valve K type and ASTM glass thermometer has been used to control the temperature at the inlet and outlet of containers.

To control the pressure of the system, two pressure gauges are used at the inlet and outlet of the extraction container. The pressure gauge at the entrance of the container is of the hand type and the second pressure gauge is of the digital type. All temperature and pressure control systems and water pumps are controlled using the control board.

pressure reducer

The pressure reducer used in the device consists of a pressure relief valve and a simple reducer that prevents it from freezing by using a 1000-watt element. With the help of this valve, you can set the desired flow. Separation containers are used to obtain the desired extracted essential oil after the pressure relief valve and reducing the pressure to one atmosphere. In this research, different types of collection containers were used, and depending on the type of experiment, each of these containers was used. After collecting the extracted materials in the collection container, the fluid released from the separator enters the flowmeter.

Description of the process

In this section, we will have an overview of the extraction process by the device.

First, the prepared oil seeds are ground, and the required mesh size sieve is separated using the set. According to the test, the necessary amount of weighed oil seed is mixed with the necessary amount of ethanol solvent.

Glass fillers with a diameter of 1 ml are prepared and its volume is measured with a graduated cylinder. Before loading, the extraction container should be cleaned with alcohol so that the residual oil from previous tests does not remain in it. First, the device is turned on and placed at the desired temperature so that the extraction

container and fillers reach the desired temperature. After stabilizing the temperature of the extraction container at the desired temperature, the oil seeds are loaded. Next, the gas released from the carbon dioxide capsule enters the refrigeration cycle, and after converting the gas into a liquid, it enters the pump. According to the pressure of the gas inside the capsule, the temperature of the refrigeration cycle is determined in order to prevent the freezing and blocking of the flow path in addition to creating liquid carbon dioxide. By entering the liquid into the pump and pumping it up to the desired pressure, the liquid carbon dioxide with high pressure enters the oscillating tank. The oscillating container is made similar to the extraction containers. In this vessel, in addition to preventing pressure fluctuations, the temperature of the fluid is also brought to the desired temperature by hot water, and after that, the supercritical fluid enters the extraction vessel by opening the valve. The feed is already loaded into the extraction container.

The outlet valve of the extraction vessel is closed and the fluid is placed in contact with the feed. A thermometer is used at the inlet and outlet of the extraction vessel to control the temperature of the water and the extraction vessel, which provides the possibility of controlling the temperature of the extraction vessel. A pressure gauge is used at the inlet and outlet of the container. By controlling the temperature and pressure, the desired conditions can be reached. After the static extraction time has passed, the outlet valve is opened and the dynamic extraction phase begins by adjusting the flow rate by the flow meter. At this stage, the way to break the pressure and get out of the critical area should also be taken into consideration. The extreme pressure drop of the fluid flow causes the transformation of suffocation or expansion - Joule-Thomson, due to the positive Joule-Thomson coefficient of Crane Dioxide, this causes a sharp decrease. The temperature in the place of the pressure relief valve, therefore, this place should be heated by a heater to prevent the formation of dry ice and the freezing of carbon dioxide and the blocking of the flow path.

For this purpose, thermal elements with temperature control system have been used. By breaking the pressure around the atmospheric pressure, the gas enters the separation side. When the gas passes through the U-shaped part of the container and cools down, the substances dissolved in the carbon dioxide are separated from it and settle, and it comes out of the container's stop valve. The gas is formed and exits from the upper valve of the vessel. After the time required for extraction in dynamic mode, the discharge phase begins. In this phase, the gas inlet valve is closed and the gas in the vessel is released. After the pressure inside the extraction vessel reaches Atmospheric pressure, its lid is opened and the material inside is emptied.

Oil analysis

Among all chromatography methods, the determination and detection of fatty acids by gas chromatography provides the most accurate results. Gas chromatography is a physical method that is used to separate, identify and measure components. To separate and separate fatty acids, they often use their methyl esters, which have a lower boiling point, to methylate the fatty acids in the oils obtained from The samples are first mixed with 0.01 g of the tested oil with 3 ml of normal hetane and 0.05 ml of 2 normal methanolic potassium hydroxide solution, and the solution is shaken for 20 minutes by an electric stirrer. After this time, glycerol settles and its upper layer is the methyl esters dissolved in henan, which are injected into the gas chromatograph to determine the type of fatty acids and their amount. The specifications of the used gas chromatograph are as follows.

First, 0.01 g of the tested oil is mixed with 3 ml of normal heptane and 0.05 ml of methanolic solution of 2 normal potassium hydroxide. The solution is stirred for 20 minutes by an electric stirrer. After this time, glycerol settles and its upper layer is the methyl esters dissolved in hetane, which are injected into the gas chromatograph to determine the type of fatty acids and their amount. The following are the specifications of the used gas chromatograph: Model 3400 G Varian carrier gas, helium, 30 psi pressure, polar 88 Cp Sil column with a length of 100 meters and a thickness of 0.1 um, injection part temperature of 250 degrees Celsius, hydrogen fuel and air oxidation flame ionization detector. The results of an analysis of fatty acids are provided.

Experiments involving models of damaged and healthy cells

rapeseed oil

Pressure effects

In three separate experiments, the effects of pressure were investigated at pressures of 20, 17, and 13 MPa. As shown in Figure 19, as the pressure increases, the density of carbon dioxide increases, and with the increase in density, solubility also increases, making the solvent more potent [19]. (1). Increasing the pressure from 13 MPa to 17 MPa increases oil extraction by 21.6% over the course of 140 minutes, while increasing the pressure from 17 MPa to 20 MPa increases oil extraction by 60.84 % over the course of 140 minutes. In the meantime, the increase in pressure from 13 MPa to 20 MPa increases oil extraction by 57.95 percent. These results demonstrate that increasing pressure has a substantial impact on the oil extraction from rapeseed. A further benefit of working at high pressure is the reduction in extraction time. As indicated in figure (1), at a pressure of 20 MPa, oil extraction reaches a certain percentage in around 45 minutes, while it takes 140 minutes at a pressure of 13 MPa. We save around 95 minutes of time, and we attain the extraction % at a pressure of 17 MPa in approximately 63 minutes, resulting in a total time savings of 77 minutes.

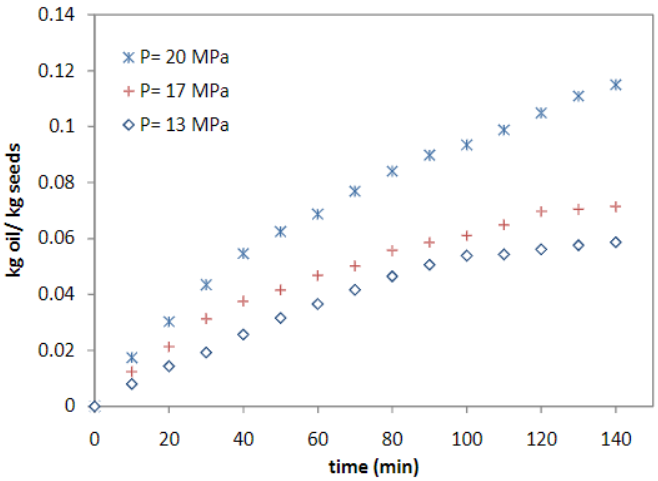

Figure 1: A comparison of the pressures required to extract oil from rapeseed oil seeds.
Temperature 308 degrees Celsius; flow rate 5 grams per minute

Temperature's affect

In the experiment, the impact of temperature on the extraction rate was investigated. Temperature can have two distinct effects on extraction. As the temperature rises, the density of the solvent falls while the oil's vapor pressure increases. Reducing density has a detrimental effect on extraction, while raising vapor pressure has a favorable effect [19]. Depending on the settings of the test, each of these two parameters may be effective. At pressures greater than 35 MPa, the effect of steam pressure becomes more apparent, and the extraction percentage can increase with increasing temperature [20,21]. However, in this investigation, increasing the temperature had the reverse impact on the percentage of extraction since the experimental circumstances were less than 20 MPa. As shown in Figure (2), the extraction % falls as the temperature rises; from 308 to 318 degrees Kelvin, the extraction percentage decreases by 37.26 degrees, and from 308 to 328 degrees Kelvin, the extraction percentage decreases by 39.4 degrees.

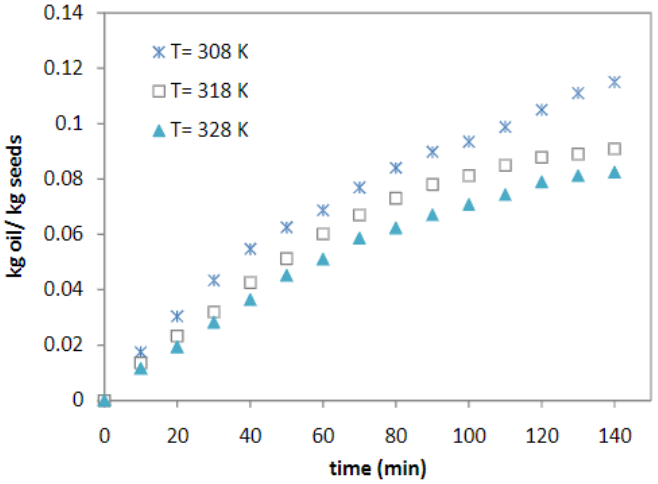

Figure 2: Temperature-based comparison of oil extraction from rapeseed oil seeds
Flow rate of 5 g/min at 20 MPa pressure.

Impact of current strength

The effect of current intensity in tests 1, 6, and 7 (Table 3(2)) on the oil extraction % was explored, and its effect on the extraction percentage is depicted in Figure (3). The extraction percentage increases as the current intensity increases. This increase is a result of the increasing differential in concentration between the solvent and particle. [22] Because the concentration of the solvent on the surface is large, this factor has little influence at the beginning of the graph. But, when the concentration of the solvent on the surface decreases, its effect becomes apparent. Over time, this element demonstrates its effect. Obviously, the intensity of the current can also have the opposite effect on the extraction rate. If the current intensity is too high, the solvent will not have sufficient time to dissolve the solvent in the extraction vessel, resulting in a significant amount of solvent consumption. As a

result, the extraction process should result in optimal conditions [23]. Certainly, it should be mentioned that the amount of solvent consumed has also increased, but according to figure (4), the same amount of solvent may now be used with less. Raising the strength of the flow enhances the extraction rate.

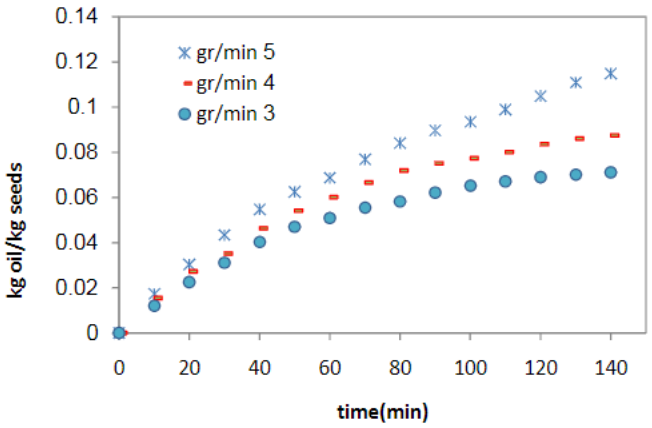

Figure 3: Comparison diagram of oil extraction from rapeseed oil seed in the intensity of different currents with regard to time at 20 MPa pressure.

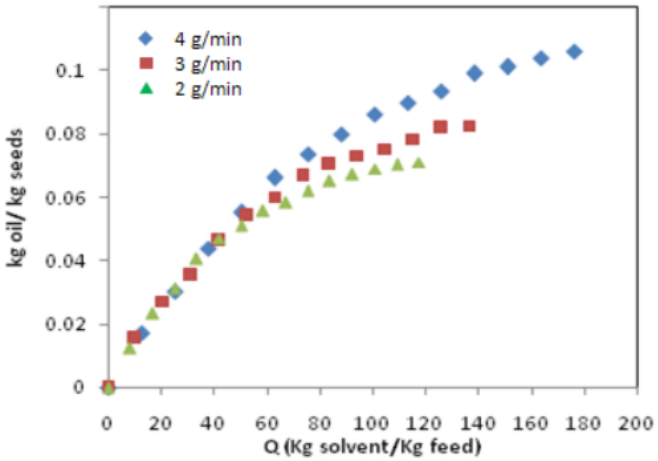

Figure 4: Comparison diagram of oil extraction from rapeseed oil seeds at a pressure of 20 MPa and a temperature of 308 K for different current intensities.

Consequences of particle size

By grinding the particles, the particle's contact surface with the solvent is increased. Grinding damages the cell wall of the grain; as a result, the soluble components are more easily brought to the particle's surface and are more accessible to the solvent. After grinding, the penetration path within the solid matrix becomes shorter, the internal resistance of the solid matrix reduces, and the solute penetrates through the cell wall with relative ease. The influence of particle size on oil extraction rate was explored in three separate studies (7 and 16). As shown in Figure (4), by decreasing the particle size from 0.6 to 0.3 mm, the extraction percentage increases by 27.15 percent in 140 minutes, however by decreasing the particle size from 0.6 to 0.156104 mm, the extraction percentage increases at the conclusion of the test. In general, the proportion of oil extracted rises as the particle size decreases, assuming there are no difficulties with the test conditions. Because lowering the particle size can allow the oilseed to stick together, causing the phenomenon called bed channeling.

Sesame Pressure effect

As mentioned in the section on the influence of pressure on the extraction of oil from rapeseed, increasing the pressure can increase the density of the solvent, and this can increase the solubility of carbon dioxide. [19] As it is anticipated that increasing pressure will improve sesame oil extraction, the effect of pressure was tested in three separate experiments (3, 102) in Table (3-1). By increasing the pressure from 13 MPa to 17 MPa, the percentage of oil extracted increases by 67.49 percentage points, and by increasing the pressure from 13 MPa to 20 MPa, the percentage of oil extracted increases by almost 102%. Obviously, from an economic standpoint and

for the implementation of industrial plans, the cost of producing the device must be addressed, which rises dramatically as the device's pressure increases.

Temperature's affect

Experiments on the effect of temperature on the extraction of oil from sesame seeds are presented in this section (1, 4, and 5). Owing to the fact that these experiments were conducted at a pressure of 20 MPa and, at this pressure, the influence of density is greater than the pressure of the soluble vapor, increasing the temperature decreases the oil extraction rate. Increasing the temperature from 308 to 318 K decreases the extraction percentage by 27.31 percentage points throughout the test period, while raising the temperature from 308 to 328 K decreases the oil extraction % by 53.44 percentage points within 140 minutes. Obviously, when high pressures are employed, the effect of steam pressure can also be utilized by increasing the temperature.

Impact of current strength

As the intensity of the current increases, so does the extraction rate. Obviously, the current intensity might also have the reverse effect on the extraction %. As previously stated, if the current intensity is too high, the solvent will not have sufficient time to dissolve the solute in the extraction vessel. As a result, the extraction process should result in optimal conditions. Increasing the strength of flow from 3 to 4 g/min increases the percentage of extraction by 46.7% during a period of 140 minutes, while increasing the intensity of flow to 5 g/min increases the percentage of extraction by 29.2%.

Laboratory results for the model of seed and substrate

rapeseed oil

Pressure effect

At four different pressures, the effect of pressure on the percentage of oil extracted from oilseed rape was examined. A rise in pressure can cause the density of the solvent to increase. This can boost the oil's solubility in carbon dioxide. As anticipated, the pressure required to extract oil from rapeseed oil seed increased as well. Increasing the pressure from 12 MPa to 15 MPa raises the extraction percentage by 25% after 140 minutes, while raising the pressure from 12 MPa to 18 MPa increases the extraction percentage. The oil increases by 62.8%, and the extraction rate rises to 75.94% when the pressure climbs to 20 MPa. These data demonstrate that an increase in pressure has a significant impact on the extraction rate.

Temperature's affect

Three studies (1, 4 and 5) in Table (3-4) evaluated the influence of temperature on the extraction of oil from rapeseed, and the increase in temperature at a pressure of 20 MPa had the opposite effect on the percentage of oil extraction. At this pressure, the density effect is more prominent than the vapor pressure of a soluble substance. At the conclusion of 140 minutes, the percentage of extraction drops by 73.55 degrees with an increase in temperature from 313 degrees to 323 degrees Kelvin, and by 73.55 degrees with an increase in temperature from 313 degrees to 333 degrees. Oil extraction decreases by 77.46 percent. Naturally, if high pressures are applied, the influence of steam pressure can be used to improve the percentage of extraction by increasing the temperature [20].

Consequences of particle size

The influence of particle size on the oil extraction % was studied for three distinct particle sizes: 3,15, 0.6, and 0. Reducing the particle size from 0.6 to 0.3 mm increases the extraction percentage by 33.52 percent in 140 minutes, while reducing the particle size from 0.6 to 0.15 mm raises the extraction percentage by 107.4 percent. With decreasing particle size, the solute becomes more accessible to the solvent. Following grinding, the penetration path and internal resistance of the solid matrix are diminished, and the solvent can be easily extracted. These data indicate that the effect of particle size on the extraction % can be significant.

Impact of current strength

Rapeseed oil was extracted by conducting experiments on the influence of current intensity. By raising the strength of the flow, the new fluid is able to come into greater contact with the particles, hence boosting the extraction rate. By raising the flow rate from 2 to 3 grams per minute in 140 minutes, the extraction rate increases by 15.53 percent. This percentage increases by 38.08 percent when the flow intensity increases to 4 g/min.

Optical microscopy image analysis

Using a vegetable electron microscopy, the surface of rapeseed and sesame seed particles and the effect of texture on oil extraction from oilseeds were examined. As seen in the diagram, the surface of the seed is extremely abrasive, making it difficult to extract the oil. To extract rapeseed oil more efficiently, we must heat milled rapeseed seeds at 90 degrees Celsius for 30 minutes, or until the oil becomes liquid. The surface of the particle is readily accessible to the supercritical fluid, as is the oil. Increase the oil extraction percentage [25.6.7.24]. In the

case of sesame seeds, it is no longer necessary to heat the grain, and grinding the grain makes it easy to apply oil to the grain's surface.

Oil extraction modeling from rapeseed

The effect of test conditions on the seed and substrate model's parameters

When pressure and temperature increase, the mass transfer coefficient (ke) of a film drops. Hence, reducing pressure has a bigger impact on the increase in film mass transfer coefficient than temperature increase. The mass transfer rate rises by increasing the pressure at a constant temperature or reducing the temperature at a constant pressure [9,21,27]. In general, a rise in pressure or a reduction in temperature causes the effective diffusion coefficient (Date) to fall and the axial dispersion coefficient to increase (D). While an increase in pressure decreases the diffusion coefficient, it increases the solubility of compounds that are soluble. In experiments 1, 9, and 10, particle size effects were explored. When the particle size rises, so does the penetration path, and the fluid must have the capacity and time to accomplish penetration; hence, the resistance to penetration increases as well. When particle size diminishes, the film mass transfer coefficient rises, the effective diffusion coefficient remains almost constant, and the axial dispersion coefficient falls. In this study, when the intensity of the flow rose, the extraction rate increased, and the effective diffusion coefficient remained nearly constant. When the intensity of the current increases, the film layer surrounding the grain grows thicker and the mass transfer resistance reduces. Moreover, both the film diffusion coefficient and the axial diffusion coefficient rise. Increasing the strength of the flow improves extraction because the flow is driven out of its natural (free) state and into a forced state. In general, the seed and substrate model for extracting oil from rapeseed was in good accord with laboratory data.

Oil extraction modeling from sesame oil seeds

The effect of test conditions on the seed and substrate model's parameters

In the instance of oil extraction from sesame seeds, the effective diffusion coefficient drops as pressure increases and increases as temperature rises. This effect is also observed for the film's mass transfer coefficient, which rises with increasing temperature and falling pressure. In the instance of the axial dispersion coefficient, a rise in pressure and a reduction in temperature result in an increase in the axial dispersion coefficient. The effective diffusion coefficient remains unchanged when flow strength increases, although the mass transfer coefficient of the film increases. This rise may be caused by the decrease in thickness of the film around the particle, which increases the film's mass transfer coefficient, which is inversely proportional to film thickness. Because approximately fifty percent of sesame is constituted of oil, it is challenging to separate the powdered particles into very small sizes. As a result, the effect of particle size was not explored in the instance of sesame, and the porosity of the substrate was investigated as an additional test parameter. By increasing the substrate's porosity, more fluid can come into touch with the particles, which has a significant impact on improving the extraction rate. be an intermediary Due to the presence of additional fluid in the bed, the constant effective penetration coefficient, film mass transfer coefficient, and axial dispersion coefficient rose as bed porosity increased.

Results of modeling oil extraction from rapeseed

The effect of test conditions on broken and healthy cell model parameters

As stated previously, the extraction diagram in the model of damaged and healthy cells is composed of three sections. The first portion of the diagram is dependent on the fluid's solubility, which is represented by the model parameter y, whose value increases as the pressure increases. This condition is caused by an increase in fluid density. The value of y has more than doubled as a result of the rise in pressure from 13 MPa to 20 MPa. Raising the solubility of rapeseed oil in the supercritical fluid allows the driving force to develop in the supercritical fluid, hence accelerating the initial stage of extraction. With an increase in pressure, the mass transfer coefficient in the fluid phase and oil penetration into the fluid decrease, resulting in a decrease in extraction speed; however, the effect of the driving force on the extraction percentage is greater than the increase in mass transfer resistance in the initial stage of extraction. With an increase in pressure, the mass transfer coefficient in the solid phase increases relative to the mass transfer coefficient in the fluid phase. This may be due to the disintegration of the cell wall of the oil seeds, resulting in a decrease in the mass transfer resistance in the fluid. As a result, extraction can be carried out more effectively in its last phase, as the effect is now manifest. According to the data derived from the model of broken and healthy cells, the extraction percentage reduces as the temperature rises, since increasing the temperature increases the mass transfer coefficient in the fluid phase and decreases the mass transfer resistance in the fluid phase. This can have a favorable influence on the oil extraction rate, but the inverse effect of temperature on fluid density and solubility neutralizes this effect. Extraction also increases as current intensity increases. With an increase in flow intensity, the value of the mass transfer coefficient in the fluid and solid phase increases, and the increase in these two values causes a decrease in the mass transfer resistances in the fluid and solid phase, as well as an increase in the extraction percentage; however, it has no effect on the solubility of the fluid and its value remains unchanged. By grinding the seeds, the visible surface of the particles increases and the walls of the cells carrying the oil are shattered, making them more accessible to the fluid; lowering the particle size reduces

the path of penetration and improves oil extraction. When particle size drops, so does particle quantity. A reduction in particle size has no effect on the fluid's solubility. In general, particle grinding has altered the structure of particles and diminished their mass transfer resistance. By decreasing particle size, the fluid's mass transfer coefficient has increased due to the increased surface area. Obviously, when lowering the particle size, consideration must be given to the cost of grinding and the pulping of the particles in the bed into small sizes so as not to induce the phenomena of fluid flow channelization. [27]

Good agreement existed between the results of the damaged and healthy cells model and the laboratory results, and particle grinding improved extraction. In the initial phase, oil was taken from broken cells, and in the last phase, oil was extracted via fluid penetration into healthy cells. In the initial phases, the extraction of the solubility component exerts its influence, whereas the penetration phenomena exert its influence in the final stages. In general, it is true that the extraction percentage rises as pressure and flow intensity increase. Moreover, the reduction in temperature and particle size boosts the extraction rate.

Conclusion

In this study, a supercritical extraction apparatus with pressure resistance of 400 bar was designed and constructed. With this apparatus, oil was extracted from rapeseed and sesame oil seeds under various operating circumstances.

At increasing pressure, the density of the supercritical fluid increases and approaches the density of a liquid; hence, the solubility of the fluid increases substantially. In this study, oil extraction from rapeseed and sesame seeds rose with increasing pressure, and increasing pressure was one of the criteria with the highest influence on the extraction percentage.

In this investigation, a rise in temperature had the opposite effect on the extraction rate. With an increase in temperature, the density of the fluid decreased, resulting in a loss in solubility. The influence of the vapor pressure of the soluble component was unable to compensate for this shortfall, and the extraction percentage reduced. The influence of temperature variations on extraction % was less than that of pressure variations.

In the extraction of oil from rapeseed, the influence of particle size was also explored, however due to the high oil content of sesame seed, it cannot be separated by a specific particle size during grinding, thus the effect of bed porosity was investigated. The change in particle size affects the porosity of the substrate, and raising the porosity can assist in enhancing the extraction percentage. In the extraction of oil from rapeseed, particle size had a substantial effect on the percentage of oil extracted, comparable to the effect of pressure on the percentage of oil extracted. As the particle size increases, the fluid has a more difficult time penetrating the particle, and as the particle size decreases, the extraction percentage increases.

The intensity of the current can also affect the extraction rate. By increasing the intensity of the flow, more fresh fluid may come into contact with the sample, and by increasing the speed, oil extraction can be increased by decreasing the film resistance. In this investigation, raising the strength of the current assisted in increasing the extraction rate. Obviously, its impact was not as significant as that of pressure and particle size.

The optimal operating parameters for extraction were 200 bar pressure, 308 degrees Kelvin temperature, 0.15 mm particle size, 5 g/min flow rate, and 45% bed porosity. In 140 minutes, the best oil extraction percentage from rapeseed was approximately 49% and from sesame oil seed it was 61%. In order to examine the supercritical extraction of oil from rapeseed and sesame seeds, a dynamic temporal simulation of the process was performed. Time reduces the concentration of fatty acids in the solid phase. The concentration of fatty acids in the fluid phase increases throughout the length of the bed, according to the obtained results. Two mass transfer models were compared to the experimental results obtained. were determined The modeling results indicate that the seed and substrate model was superior than the broken and healthy cell model in representing the laboratory data, and its error percentage was determined.

Suggestions

Experimenting with oil extraction from oilseeds at higher pressures

Using gas and liquid chromatography to investigate the quality and quantity of oil components at varied pressures, temperatures, and flow intensities.

Comparative economic analysis of oil extraction from oilseeds by supercritical and conventional processes

Capacity expansion on an industrial scale is investigated by evaluating the oil quality of various oilseeds and comparing it to standard methods.

Enhancing the settings of the developed device and developing it for further nanomaterial extraction and nanomaterial production activities

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