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Recovery of Vegetable Oil from Spent Bleaching Earth: State-of-the-Art and Prospect for Process Intensification

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Vegetable oils undergo numerous refining steps in order to remove undesirable compounds and produce high quality, stable commercial products. Recovery of vegetable oil from spent bleaching earth is an area where ample opportunities exist for cleaner production and cost saving in the vegetable oil processing industry. Conventional oil extraction and refining processes, which involve multiple unit operations, have several disadvantages. These include complex separation steps, energy-intensive operations, the requirement for large amounts of water and hazardous chemicals and the potential of generating large quantities of wastes. Conventional oil extraction mostly uses hexane. High energy consumption, high temperature operation, the important portion of the nutritional oil components being lost and large amount of water during the process of refining result from the use of hexane. There is a dire need for the development of separation techniques that will facilitate recovery of vegetable oil from spent bleaching earth while sustaining the nutritional components naturally present in the vegetable oils and reducing the negative impact of oil processing on the environment. This paper reviews the state-of-the art technologies for recovery of vegetable oil from spent bleaching earth. It presents the development of the technologies chronologically and compares their relative merits from aspects of capital requirements, resource utilisation, cleaner production, sustainability and economy. The paper ends with a look at supercritical fluid extraction (SFE) as a potentially promising alternative recovery technology that could offer opportunities for process intensification; resulting in a simpler, cleaner and resource-efficient system for oil recovery from bleaching earth.

1. Introduction

High concentration of the bioactive lipid components has made vegetable oil received much attention due to the polyunsaturated fatty acids and pthytosterols that have shown some heath benefit. Fat and oil and their other lipids components are used in pharmaceuticals and other industries (Straccia et al., 2012). Spent bleaching earth are used to remove metals gums, oxidised products residual gum and colour from the oil. It engrosses about 0.5 % by weight of oil in the process. Spent bleaching earth (SBE) are usually discarded in landfills and because of the high cost of disposal it is necessary to use the material (Loh et al., 2013). Conventional or supercritical fluid extraction method can be used to extract oil from the spent bleaching earth with up to 30 % by weight of SBE (Loh et al., 2013). To lower the cost in oil processing industry, the residual oil in spent bleaching earth must be recovered and reused for application in the industry. Based on the worldwide production of more than 60 Mt of vegetable, it was appraised that about 600,000 t of SBE are exploited worldwide in the oil refining industry (Kheang et al., 2006). This paper reviews the state-of-the art technologies for recovery of vegetable oil from spent bleaching earth and discusses the common extraction technologies and also compares their relative advantages from aspects of capital requirements, resources utilisation, cleaner production sustainability and economy.

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2. State of the art Technologies in Spent Bleaching Earth extraction methods

Recovery of vegetable oil from spent bleaching earth requires suitable extraction processes. The major benefit for the extraction method is when it was used to recover the oil in shortest possible time with minimum solvent consumption. Short extraction time is needed because of the ability to minimise any potential degradation of the active components and reduced electricity consumption (Mohammad Azmin et al., 2016). The various extraction technologies to recover vegetable oils from spent bleaching earth discussed in this review are soxhlet extraction, membrane technology, subcritical water technology and supercritical fluid extraction (SFE) technology.

2.1 Soxhlet Extraction

Recovery of oil adsorbed in the spent bleaching earth (SBE) can be extracted by soxhlet extractor using hexane as the solvent (Huang and Chang, 2010).The process of extraction begins with a sample placed on a thimble holder filled with hexane from a distillation flask until the solvent extend to over flow level, a siphon releases the solute from the thimble holder moving the aliquot back into the distillation flask which then carries the extracted oil into the bulk liquid. Extraction process continue until all the oil had been extracted and the solvent is recycle through the sample in a continuous mode (Mohammad Azmin et al., 2016). The extracts obtained from the process were being filtered and the solvent was removed under reduced pressure and at defined temperature in a rotary evaporator.

In this extraction method, it is very vital to choose the most suitable solvent for extracting the targeted components because different solvent polarity will dissolve different compound. The boiling point of hexane is 65 ºC which make it used widely as a solvent. Conventional method used for the recovery of vegetable from SBE are shown in Figure 1. After the extraction, the oil-solvent is then separated between the oil from hexane which requires large amount of energy. In the refining step, the addition of caustic leads to saponification and the resulting soap can trap some oil, which is about 50 % of the free fatty acid content with total refining loss usually equal to the amount of FFA in the oil and hence heavy contaminated effluents are produced because large amounts of water and chemical are used.

2.2 Membrane Technology

The semipermeable barriers that separate two phase and prevent the transport of various substances in a specific way are called membranes (de Morais Coutinho et al., 2009). The main advantages of this separation process lie on the low energy consumption, greater separation efficiency, reduction in number of processing and the improved final product quality. The separation technique are very useful in the area of biotechnology, pharmaceutical and food industries (de Morais Coutinho et al., 2009).

The possibility of separation in minimisation of thermal damage, recycling solvents, low emission, the decrease in oil loses and lower energy are the major merits of the separation technique. The characteristics of the conventional vegetable oil refining process can be compared vividly with the refining with membrane in the area of loss of neutral oil, the need for large of water, loss of nutrient and other chemical reagent while the refining with membrane offer advantages in low energy consumption operation at mild temperature and also retain of nutrient and other desirable compounds (de Morais Coutinho et al., 2009).

Many researchers in Japan, Germany, America and India have proposed the feasibility of the membrane as an alternative processing and reliable method by considering the disadvantages of the conventional refining processes of vegetable oil (Reddy et al., 2001). Oxidation can be avoided due to the mild operating conditions which serves as major advantage of membrane based oil processing. Membrane technology can be applied to simplify processes, reduce energy consumption, and reduce wastewater production (Ghosh, 2007).

2.3 Subcritical Water Technology

The use of water instead of organic solvent is an alternative recovery method of palm oil from spent bleaching earth called Subcritical Water Technology(SWT) (Abdelmoez et al., 2015). The extraction of vegetable oil from spent bleaching earth using subcritical water technology method was performed in the laboratory whereby water was used as a solvent in a reactor tube (Fattah et al., 2014).

The reactor must be sealed and should not be filled up to avert the hydraulics pressure of the liquid from fracturing the vessel. The operating temperature of the extraction was between 180 to 280 °C and the pressure was obtained from the steam table for the saturated steam. Water was cooled down after the required reaction time and finally immersed in a cold-water bath.

The extracted product obtained was separated into the aqueous stage, solid and the oil phase. Simple centrifugation and vacuum-filtration processes was used to separate the three different phases and then petroleum ether was used to recover any oil traces from the aqueous phase. The recovered oil was weighed after the petroleum ether was evaporated in a furnace at 80 °C.

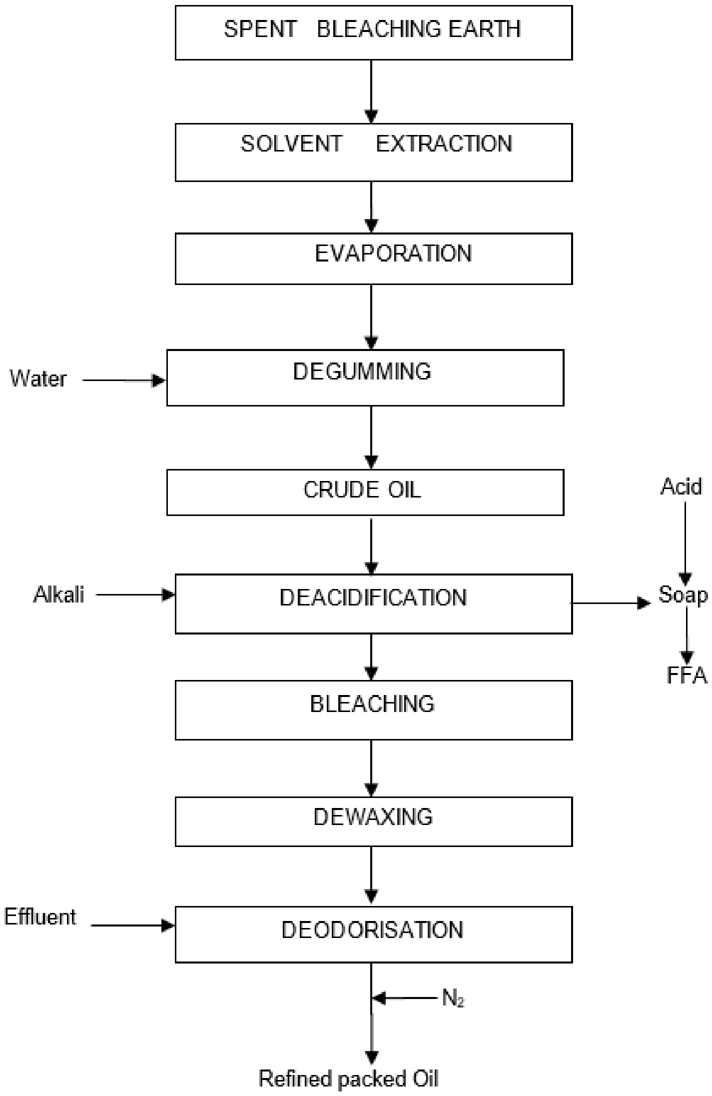

Figure 1: Conventional method for recovery of vegetable oil from SBE (Cheryan, 2005)

2.4 Supercritical Fluid Extraction (SFE)

Supercritical fluid extraction (SFE) is one of the extraction methods used in the recovery of vegetable oil from spent bleaching earth. Extraction of spent bleaching earth via supercritical fluid technology is a clean and environmental friendly technology, utilising non-hazardous carbon dioxide (SC-CO_2) as solvent for extraction of oil (Herrero et al., 2010). Overall good quality of oil can be recovered by this method (Kheang et al., 2006).
Katiyar and Khanam (2014) reveal that higher yields and better quality of oil can be achieved with supercritical fluid extraction. Supercritical fluids (SCF) have been used in different fields such as the food, pharmaceutical, chemical, and fuel industries due to the advantages of supercritical fluids, such as the absence of toxic residue in the final product (Pereira and Meireles, 2009). In super critical extraction process, the mobile phase is subjected to temperature and pressure near or above critical point at the supercritical process for the purpose of improving the mobile phase solvating power (Sovilj, 2010). Exclusion of organic solvent reduce the problem of their storage in the lipidologist laboratory serves as major advantages of using supercritical fluid extraction (Sairam et al., 2012). The critical temperature and pressure of carbon dioxide at supercritical state are 31.1 °C and 73.8 bar. At this state, they have high diffusion coefficients and low viscosities, and, possess

high solvating power properties with characteristics of liquids. These account for supercritical CO_2 as a good fluid solvent (Mohammad Azmin et al., 2016).
The penetration of carbon dioxide into the solid material was facilitated by the increased diffusivity of solvent and decreased viscosities which lead to an increased mass transfer and reduced extraction times in SFE (Mohammad Azmin et al., 2016). Another factor that determine the effectiveness of the supercritical fluid extraction is the solvating power that changes with temperature and pressure. The recovery of oil from SBE using supercritical fluid extraction method can be achieved only by optimising the experimental conditions. The extraction of oil from spent bleaching earth was conducted at 82.7 MPa and 80 °C in which the solubility in SC-CO_2 is maximised. Table 1 shows the SFE experiment on the recovery of vegetable oil from spent bleaching clay. Such conditions have been shown to yield vegetable oil at a slightly lower cost than oil obtained from the conventional liquid-extraction process (King et al., 1992). Due to channelling of extraction fluid through the sorbent bed as well as compaction of the clay bed under high pressure, the particle size of bleaching clays can also inhibit complete extraction of the oil from the SBE.

Table 1: SFE Experimental results on Spent Bleaching Earth (King et al., 1992)

Run	Clay type	Extractor	% Oil Extracted
1	Neutral	Pilot	15.2
2	Neutral	Pilot	19.2
3	Neutral	Pilot	26.8
4	Neutral	Pilot	25.5
5	Neutral	Lab	32.1
6	Neutral	Lab	30.7
7	Neutral	Pilot	30.9
8	Acid	Lab	34.1
9	Acid	Pilot	32.6
10	Neutral	Pilot	26.5

3. Comparison of Extraction Technologies

The extraction processes discussed in the previous section reveals that SFE is a well-established technique for the extraction and separation of both important oils and its derivatives for food, pharmaceutical, and cosmetic. SFE processes also result in high-quality oils with commercially viable compositions, as compared to the products obtained using conventional processes (Mohammad Azmin et al., 2016). The density, diffusivity, dielectric constant, and viscosity can be easily controlled by varying the pressure or temperature without crossing the phase boundaries because of the physicochemical properties of the supercritical extraction fluid.
Supercritical CO_2 has been applied for the extraction and isolation of multiple valuable compounds from natural products over the past decade (Wüst Zibetti et al., 2013). CO_2 is more environmentally friendly than most extraction techniques that employ organic and liquid solvents. Table 2 shows comparison of solvents, efficiency, operating conditions on the recovery of different vegetable oils from spent bleaching earth (SBE). From the comparison of the extraction methods in Table 2, it shows that supercritical fluid extraction method is the preferable separation technique because of increase in extraction yield, low temperature operation and production of superior extract materials.
Subcritical water technology seemed to be the greener alternative method for different vegetable oil extraction. The short extraction time and the elimination of using solvent were the major benefit of using such technology in comparison to the extraction method using hexane as a solvent and membrane technology.
The super critical fluid extraction technology is really needed as advanced method which can provide fast, reliable, clean and cheap methods. The SFE process has a number of advantages over conventional extraction. These are low temperature operation, pollution free operation, inert solvent, selective separation and fractionation of tailor-made end-product, and extraction of high-value product or new product with improved functional or nutritional characteristics.
Membrane technology can be used to recover solvent and also membrane can be stored in organic solvents for several weeks without changing the initial flux, but that the combination of organic solvent and elevated pressures gives rise to considerable changes in membrane performance (Snape and Nakajima, 1996). It was also revealed that development of membrane processing techniques for vegetable oils also reduce energy consumption, minimise degradation and maximise anti-oxidant status compare to the conventional extraction methods (Lindley, 1998). Another important advantage of supercritical fluid extraction methods over the use of conventional extraction methods is the ability to recover heavy metals from solid matrices and liquids.

Complexing agents used in conventional solvent extraction can also be used in SFE complexation of metal Ions (Sairam et al, 2012).

Table 2: Comparison of solvents, efficiency, and operating conditions on the recovery of different vegetable oils using Soxhlet (Hexane Solvent) and Supercritical Fluid Extraction (CO_2 Solvent)

S/N	Sample	Solvent	Efficiency (%)	Pressure (MPa)	Temp (K)	Reference
1	Soybeans	Hexane	90.0	0.29	333	(Huang and Chang, 2010)
		CO_2	34.4	7.46		(King et al.,1992)
2	Palm Oil	Hexane	30.0			(Kheang et al., 2006)
		CO_2	90.2	24.00	323	(Ooiet al., 996)
3	H/seed Oil	CO_2	50.0	62.50	343	(Revenchon et al., 2000)
4	Palm Kernel	CO_2	50.0	48.30	353.3	(Zaidul et al., 2007)
5	Cheery seed	CO_2	90.0	45.70	319	(Stracciaet al., 2012)
6	Stamineous oil	CO_2	95.0	10.24	353	(Pouralinazar et al., 2012)
7	Oil seed	CO_2	90.0	30.00	313	(Nunez and Delvalle, 2014)

4. Conclusion

Extraction technologies for the recovery of vegetable oil from spent bleaching earth described in this review are suitable for solid-liquid extraction. Soxhlet extraction is one of the common methods which is effective, but can be time-consuming and also uses a large amount of solvent. Soxhlet extraction not only requires high operating cost but also causes additional environmental problems. More advanced extraction methods such as supercritical fluid extractions (SFE), membrane technology have been used as alternatives to perform faster extraction and obtain higher quality yield. With the expectation of preserving the greater part of these nutrients, SFE processes are carried out at low temperatures and with the elimination of a substantial number of unit operations as compared to conventional processes. SFE processes operated at higher temperatures and/or pressures can greatly decrease the extraction time. These specialised extraction techniques have found limited applications as they require higher capital investment for the complex equipment. There is ongoing work on the search for potential method that could result in higher extraction yield, less solvent and energy requirement, faster extraction time and environmentally friendly process.

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