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Free Fatty Acid Reduction in Used Frying Oil via Bio Adsorbent: A Short Review

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Used frying oil (UFO) is a byproduct of edible vegetable oil which is utilised as a medium to fry food. The disposal of UFO is a global and major environmental issue due to the straightforward dumping of the UFO into drains. Waste management issues are getting worse and endangering environmental preservation. Recycling UFOs could relieve disposal issues, but more importantly, it would improve the efficient usage of agricultural and food reserves. Most of the UFO's hazardous chemicals are oxidation products of free fatty acids (FFA), particularly polyunsaturated fatty acids. One of the main issues in the refinement of edible oil is the FFA concentration. It has also frequently been used to describe the oil's quality and suitability for food purposes. FFA is derived from triacylglycerol by cleavage of ester bonds due to the action of high temperature, oxygen and moisture. To reduce FFA, UFO is pre-treated by physical filtration, adsorption, heating, neutralisation, degumming, bleaching (whitening) and chemical treatment. In a base-catalysed system, it is preferable to have less than 1% of FFAs by weight of oil, or it will interact with the catalyst and produce soap. To enhance the quality of frying oil and UFO, numerous active filtering methods, or adsorption procedures have been researched. Bio-adsorption is economical, easily designable, environmentally friendly and is even an effective technique in the presence of competitive ions. Ions and molecules physically attach to or connect with surfaces during adsorption. The sorption capacity depends on the type of bio adsorbent and the nature of the oil. This review paper discusses the potential of four types of bio adsorbents which are coconut shell, palm kernel shell, rice husk, and bagasse in reducing FFA content in UFO. These materials possess a large surface area, which provides ample site for adsorption. The interaction and adsorption of FFAs are made easier by the presence of active sites such as hydroxyl (-OH) and carboxyl (-COOH) groups on the surface.

1. Introduction

Vegetable-based cooking oils (VBCO) are typically high in monounsaturated or polyunsaturated fats, which have a higher smoke point and are more stable at high temperatures than saturated fats. Recently, it has been used in a diverse range of non-food applications which led to food security issues. As an alternative, used frying oil (UFO), which is economical compared to edible VBCO, is preferred for non-food applications. Currently, UFO as precursors were used in the development of new greener products such as biodiesel (Susilowati et al., 2019), biopolymers (Enderus et al., 2017), lubricants (Paul et al., 2021), soaps (Medhiatika, 2021), candles (Liu et al., 2022) and material recovery (Matušinec et al., 2020). Production of biodiesel from UFO has emerged as the most efficient way of utilising UFO as it is more affordable and available compared to other biodiesel made from natural oil sources such as palm, soybean, corn, and coconut. Additionally, UFO-based biodiesel manufacturing uses less energy and releases the least greenhouse gases thus leading to minimal environmental effects. The production of large amounts of biodiesel is challenging as there is a need for high-quality oil with suitable fatty acid compositions, less free fatty acids (FFA), moisture, and contaminants (Liu et al., 2022). VBCO is commonly used for frying and is continuously heated to high degrees (above 180 °C). Under this condition, numerous polar molecules develop through oxidation, hydrolysis and decomposition reactions, altering the chemical, physical nutritional, and sensory aspects of the oil (Wiege et al., 2020). The hydrolysis reaction has resulted from the presence of moisture in the foods that are exposed to hot oil. During hydrolysis, the fatty acids such as di- and

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monoglycerides that are released from the triglycerides react with water to form FFA. The presence of FFA in UFO can be an indication of the oil's quality and freshness, and it can be further reacted to form aldehydes and ketones, which are signs that the cooking oil is rancid. Whereas oxidation is a result of oxygen interaction with hot oil (Alade et al., 2022). Oxidised monomeric, dimeric, and oligomeric triglycerides, as well as volatile substances, are the reactions of by-products. Correspondingly, the oil's double bonds break down during frying, its fatty acid composition changes thus increasing the amount of FFA and its saturation level. The transition can affect an oil's density, iodine value, viscosity, and other qualities (Gadhvi, 2019). UFO commonly have several characteristics such as high FFA, darker in colour, high in viscosity and produces a rancid odour. Since UFO are considered toxic after repeated use, the direct disposal to water severely harms the aquatic and marine life and it has been advised to be used only for non-food applications (Alade et al., 2022).

Numerous quality control techniques have been created as guides for edible oil which addresses a particular subset of the wide range of oil degradation products. FFA value, total polar material (TPM), iodine value, peroxide value (PV), anisidine value, and thiobarbituric acid reactive substances are a few of these (Bazina and He, 2018). FFA measurement is a substantial indicator of oil damage caused by the oxidation of fatty acids and the breakdown of triacylglycerols (Fajriati et al., 2023). According to the Food and Agriculture Organization of the United Nations, the maximum FFA limit for edible VBCO is 0.1-0.5 % (Szabo et al., 2022). The indicator for the FFA content in the oil is expressed by the acid number which will be expressed as a percentage of the oil's weight and can be determined through chemical analysis methods, such as simple acid titration or gas spectroscopy (GC) (Bazina and He, 2018). For certain products, the UFO can be used directly without FFA reduction, but for applications such as biodiesel, the high FFA value can react with the catalyst used in the transesterification process, which is the chemical reaction that converts the UFO into biodiesel. This can result in the formation of soap, which can reduce the yield of biodiesel and cause other issues. Sosilawati (2019) stated that during the separation of biodiesel, oils with more than 1 % FFA will generate a challenging soap emulsion. Prior to transforming the UFO into biodiesel, it must be treated to meet the required criteria. There are several methods reported to lower the FFA content in UFO such as the usage of acid and base catalysts such as sulfuric acid and sodium hydroxide (NaOH), chemical refining by caustic soda neutralisation and physical refining based on steam distillation. This approach has some drawbacks, including corrosion, high cost, safety hazard and requires proper disposal (Gharby, 2022). The use of adsorbent as an alternative may be more desirable due to its environmentally friendly and lower safety hazards. The adsorption process is a typical technique used by VBCO manufacturers to remove FFA in crude oil. It can be described as the method by which the adsorbates adhere to the adsorbent, and it only occurs on the active site of the adsorbents where ions and molecules physically bind to the surface of the solid material. In this instance, the solid surface is the adsorbent and the substance that has accumulated at the contact is the adsorbate. Due to its low cost, this method typically has more economic benefits as compared to other technologies because this technique doesn't necessitate a sizable capital investment, thus the operational costs are cheap and flexible (Michalak et al., 2013). According to researchers, the predominant adsorbent materials practised in the treatment of residual oil are aluminum hydroxide, clay, magnesol, zeolite, silica gel and magnesium oxide. The limitations of the majority of commercial adsorbents are mostly brought on by their high cost and lack of renewable resources, which drives researchers to explore alternatives (Miskah et al., 2019). Adsorption's subclass of biosorption uses a biological matrix as the sorbent. Many different types of biomaterials found in nature have been used as biosorbents and have received more attention recently such as industrial waste biomaterials and agricultural waste materials which are available on a large scale (Ifa et al., 2022).

A procedure known as "biosorption" entails the fast and permanent bonding of ions from aqueous solutions to functional groups on the surface of biomass. It is crucial to select the suitable adsorbent as the adsorption process is influenced by variables which are surface functional groups, adsorbent surface area, particle, and pore size (Jariah et al., 2022). The process also requires the examination of parameters such as polarity, temperature, agitation speed, and starting concentration of adsorbate to achieve high yields (Jjagwe et al., 2021). High carbon content materials, such as activated carbon (AC) and biochar derived from biomass, are commonly used as adsorbents in various applications. Nevertheless, AC has been extensively used as a bio-adsorbent for the purification of contaminated oil for increasing oil quality (Yusof et al., 2021). In previous work, there is limited study in treatment for UFO using bio adsorbent because it was commercially used for wastewater treatment to remove toxic pollutants (Muhammad et al., 2023). Therefore, this work aims to review four types of bio adsorbents which are bagasse, rice husk, coconut, and palm shell, in terms of process and performance on reducing FFA content in UFO. In Malaysia, the selected bio adsorbents are preferred due to their abundant production of waste, making them readily available (Othman and Jafari, 2014) and inexpensive for cutting-edge applications. The practical aspects of using these bio adsorbents can lead to successful implementation in industries, promoting more sustainable waste management practices. Moreover, the consideration of their availability in Malaysia provides context-specific insights relevant to the country's waste management and bio diesel production efforts.

2. Bio adsorbents

2.1 Coconut shell

In tropical nations around the world, coconut is a crop that is easily cultivated (Chandana et al., 2020). Due to its high carbon content and porous composition, it offers a lot of adsorption-active sites and becomes a possible source of bio adsorbent. Components such as cellulose, hemicellulose, and lignin, are found in coconut shells. The lignocellulosic structure of coconut shells is destroyed during processing, producing a highly porous material with a substantial surface area (James and Yadav, 2021). Coconut shell charcoal is made by heating coconut shells in an oxygen-free environment to produce a substance rich in carbon. According to Bhatnagar et al. (2010), the carbonization of one tonne of coconut shells yields 300 kg of charcoal, which can then be transformed into 120 kg of coconut shell-activated carbon (CHAC). Correlated to CHAC, coconut shell charcoal often has smaller pores and less surface area, which may reduce its efficiency as a bio adsorbent. The technique of activation, which involves heating coconut shell charcoal in the presence of an activating agent such as steam, carbon dioxide, or chemicals, is used to create CHAC (Bhatnagar et al., 2010). With a huge surface area and high porosity produced by this technique, the material works well as an adsorbent for a variety of pollutants, including heavy metals, organic compounds, and other contaminants (James and Yadav, 2021). Findings by Sujiono et al. (2022) successfully fabricate high-quality microporous CHAC using variation chemical activation which are NaOH, phosphoric acid (H_3PO_4) and zinc chloride ($ZnCl_2$). A rich carbon CHAC was created by carbonising the coconut shell for three hours at 600 °C. The coconut shell was produced by granulating and sieved down to 200-mesh particle size. The granules were then activated using activating agents ($ZnCl_2$, NaOH or H_3PO_4) with a 1:4 sample-to-activator ratio, heated at 85 °C for two hours, and dried for three hours at 130 °C. The NaOH activator depicts the biggest value of surface area with 516 m^2g^{-1}. It has a significant carbon concentration, according to X-Ray diffraction analysis (XRD) data. Fourier Transform Infrared Spectroscopy (FTIR) analysis revealed that the activated carbon is indicated by the presence of the functional groups of C-O, –OH and C=O.

A crucial measurement for defining the FFA that is liberated throughout the hydrolysis process is the value of the acid number. A study by Khuzaimah and Eralita (2020) intends to purify UFO with CHAC in order to reduce the amount of acid, FFA and peroxide present in UFO. In this research, 10 g CHAC was mixed with 200 g UFO for 15 min using a mechanical stirrer and it was separated using Whatman filter paper number #1 and #42. The peroxide number was measured using the iodometric titration method, whereas the acid number was obtained using the acidimetric and alkalimetric titration methods. The results revealed that the use of CHAC lowers acid number by 34% and 29%, FFA content by 34 % and 37 % and peroxide number by 34 % and 62 % for the first and second frying respectively. Discovery by Fajriati et al. (2023) has successfully purified UFO used for cooking shredded chicken using CHAC. For the methodology, UFO was first filtered using Whatman # 41 filter paper, and then CHAC was constantly swirled with UFO for 120 min while utilising the 5 % (g/v) and 10 % (g/v) CHAC. The FFA shows a 28 % reduction of 5 % (g/v) and a 45 % reduction of 10% (g/v) as the outcome. According to these findings, 10 % CHAC is more efficient than 5 % CHAC at adsorbing FFA that is created from the decomposition of triglycerides in UFO. Oil compounds and CHAC have nonpolar surfaces, respectively. The hydrophobic groups in the oil engage with the CHAC due to their comparable polarity features, which starts the adsorption process that cleans the cooking oil from the shredded chicken. Differences in FFA levels and saturated fatty acid compounds are also confirmed by FTIR analysis where the greatest transmittance of 3,469 cm^{-1} is found in oil that has been purified using 10 % CHAC, indicating that the diglyceride ester molecule has a lower absorbance than spectra that have been purified using 5 % CHAC.

2.2 Palm Kernel Shell

The waste generated from Malaysia's palm oil industry, known as palm kernel shell (PKS), can reach 4 million tonnes annually. Interestingly, it is able to be transformed into a suitable material for the manufacturing of premium activated charcoal because it is high in carbon content, porosity, density, surface area and low ash content. The activation of charcoal at high temperatures with an activator has led to higher carbon adsorption and char produced during carbonization shows surface functional groups comprising aromatic rings, ketones, and quinones. The material's surface area is considerably increased and its ability to absorb more pollutants and impurities is enhanced by the network of microscopic pores and channels that are created by this activation process throughout the material (Itam et al., 2016). Cross-links may be encouraged to form as a result of chemical activation agents, creating a stable, nonvolatile matrix. Chemical activation has some benefits over physical activation, including lower activation temperatures and larger global yields, which translate into reduced prices (Dlamini et al., 2020). The research by Hidayu et al. (2019) produces palm kernel shell-activated carbon (PKSAC) by chemical activation for crude palm oil application. The activation of PKS carbon was chemically processed using $ZnCl_2$ at an impregnation ratio of 1:1 mass basis for 24 h and carbonization was done at 500 °C for 30, 60, and 90 min using a muffle furnace. It dictates that activation activities raise the carbon content from 48.70 wt% to 58.43 wt% and continue to do so as the activation duration increases from 30 to 90 min. $ZnCl_2$ tends to boost the pore growth in AC and the amount of active site will likewise increase dramatically with this high carbon concentration.

The by-product of burning palm kernel shells is known as palm kernel shell ash (PKSA) which is an inorganic substance made up of metal oxides and minerals. With various levels of potassium (K_2O), calcium (CaO), and other trace components, silica (SiO_2) makes up the majority of PKSA. In order to reduce the FFA, PV and water content in UFO, a study by Novita et al. (2020) optimised PKSA weight variations, which were then followed by the best contact time variations. Changes in PKSA weight were 5 g, 10 g, 15 g, and 20 g, while changes in contact time were 1, 2, and 4 weeks. The result dictates that 20 g PKSA with 2 weeks of contact with UFO showed the best benefits. Under these circumstances, UFO moisture content, FFA, and PV were reduced by 81.21 %, 59.01 %, and 61.40 %, respectively. The existence of silanol clusters on silica surfaces is connected to the effectiveness of PKSA in decreasing the FFA levels in UFO. Through the silanol group, oxygen from the fatty acid carbonyl group can bind to the surface of silica. The bond that forms is a hydrogen bond between the hydrogen atom in the silanol group and the oxygen atom in the fatty acid carbonyl group. analysis was conducted to identify the amount of SiO_2 that serves as an adsorbent. At a wavelength of 2900 cm^{-1}, silanol (Si-OH) groups were found in PKSA and the water molecule adsorption mechanism in the siloxane group is indicated by the silanol group (Novita et al., 2020).

The finding by Susanto et al. (2022) revealed that the PKSAC at 5 wt% addition in crude palm oil with the condition of high temperature at 80 °C was capable of offering the least FFA value (3.86 wt%). The quantity of FFA in the crude palm oil tends to decrease with increasing H_3PO_4 chemical concentrations (1%, 3% and 5%) used to activate PKS and transform it into PKSAC. PKSAC can neutralise the FFA molecules, and this also might be due to the cellulose component in PKSAC. Additionally, it has larger pores and surface area due to chemical activation with H_3PO_4 where FFA molecules on the surface might be bound into it (Susanto et al., 2022).

2.3 Rice Husk

Rice husk, which has a 30 % to 50 % organic carbon content, is a waste product produced from paddy grain. The "rice husks" which are tough protective coverings on rice grains are stripped from the grains during the milling procedure. Cellulose makes up to 32 % of the chemical composition of rice husks on average, followed by hemicellulose at 20 %, lignin at 21 %, and other organic components like protein and fat at 20 % (Singh, 2018). The presence of carboxylic, hydroxyl and amine functional groups, together with the silanol groups (Si-OH) can enhance the adsorption operation. In general, soluble organic compounds can be extracted from rice husk through the pre-treatment process by applying a variety of modifying agents, such as base and acid solutions. For instance, base solutions would remove inorganic substances like silica and carbonate against the surface of rice husk and improve the adsorption properties. Additionally, burning rice husk could result in 20 % of ash. The produced ash retains the skeleton of the cellular structure and is expected to include greater than 95 % of silica functionality and has a high porosity and huge surface area (Daffalla et al., 2020).

The silicon content of rice husk is around 5.02 %. Silicon reacts with oxygen to generate silicon dioxide (SiO_2), a substance that exhibits adsorption capabilities by binding with the rest to produce silanol groups. A successful study conducted by Schneider et al. (2019) has used the batch method to examine the ability of the adsorbents to extract FFA from UFO while comparing various temperatures, adsorbent mass, and agitation settings using activated carbon rice husk (ACRH) and rice husk. From the result, at a temperature of 22.4 °C, speed of 169.64 rpm, and 3.39 g of adsorbent mass, the highest result for ACRH for acidity reduction was 63 %. The removal rate was the same when employing rice husk with 63 % reduction, at a temperature of 22.4 °C, speed of 80.36 rpm, and 1.61 g of adsorbent, but in less time. Because of the shorter processing durations, using rice husk as an adsorbent material to treat UFO provides an alternative to save expenses. The analysis of rice husk revealed the presence of silica functionality that may have a favourable effect on adsorption. Even with values less than those for ACRH, the results connected to the material's surface properties and the alkalinity of active sites were comparable in terms of surface area and pore volume. Lastly, research by Manique et al., (2012) aims to investigate the purification of biodiesel from UFO using rice husk ash (RHA) at concentrations of 1 %, 2 %, 3 %, 4 %, and 5 % (w/w) and compare it to two other purification techniques: the conventional acid solution (1 % aqueous H_3PO_4) and the commercial adsorbent Magnesol 1 % (w/w). The RHA demonstrated outstanding results for removing contaminants from biodiesel at a concentration of 4 %. The outcomes were comparable to those that Magnesol or an acid solution had shown. Its remarkable capacity for adsorption can be explained by the high concentration of silica in its composition and the existence of meso- and macropores.

2.4 Bagasse

Bagasse is the fibrous residue from sugarcane. It consists of dry, pulpy fibres that are left after the sugarcane stalks are crushed and squeezed to extract the juice from the sugarcane. Bagasse contains carbon material which makes it suitable to be used as a bio-adsorbent, although it is not frequently and readily used candidate as a substitute for an organic FFA reducing agent (MacCarthy et al., 2021). Hydrothermal carbonization (HTC) and steam activation at 180-240 °C and the steam activation temperature of 700-900 °C at 1 h were used to create AC from sugarcane bagasse. According to the findings by Congsomjit and Areeprasert (2020), the dry-basis AC yield ranged from 10.4 to 27.1 %. The product has a maximum Brunauer-Emmett-Teller (BET) surface area of 390 m^2/g. The overall pore volume of the AC was 0.1-0.25 cm^3 /g, with an average pore size of 2.2 to 2.5 nm. The FFA content in palm kernel oil is regarded as a key quality predictor as the larger level of FFA can be damaging to the oil's flavour, colour, stability, and safety. In order to minimise the FFA content in crude palm kernel oil, MacCarthy et al. (2021) studied the feasibility of employing bagasse as a bio adsorbent by varying the weights; 1 g, 1.5 g, 2 g, and 2.5 g. The % FFA content in both the raw and bagasse-treated palm kernel oils was measured using an American oil chemists' Society (AOCS) standard test. The result showed that the crude palm kernel oil's FFA concentration was reduced by 74 %, 77 %, 79 %, and 82 % for 1 g, 1.5 g, 2 g, and 2.5 g using bagasse adsorbent, respectively. This study reveals that the use of bagasse as a bio adsorbent is able to bring down the higher levels of FFA in crude palm kernel oil to an acceptable level.

Rahayu et al. (2018) conducted a comparison study between bagasse, coconut husk and pineapple dregs in different particle sizes and immersion times. The amount of FFA in the pure frying oil was examined and fixed at 0.27 % and when the oil was used for three repetitive fryings, the FFA increased up to 7.1 %. All adsorbents were activated with 0.1 N $KMnO_4$ for 24 h and 10 g AC powder bio adsorbent was immersed in 150g of UFO. Bagasse dictated the lowest FFA (0.24 %) chased by coconut husks (0.28 %), and pineapple dregs (0.34 %). The outcome also discovered that the longest immersion and smaller particle, which was 72 h immersion and 236 μ respectively, reduces the FFA value. It was concluded in the study that bagasse is the most effective way to reduce FFA levels in UFO.

3. Conclusions and Future Directions

The use of these bio adsorbents offers an eco-friendly and cost-effective solution for reducing FFA in UFO. However, the production is crucial because the high yield is only achievable if the adsorbent has a high number of active sites, porosity, and surface area. Future research could look into different bio-adsorbents to expand the choices for lowering the FFA level of UFO.

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