

Renewable Bleaching Alternatives (RBA) for Palm Oil Refining from Waste Materials

Muhammad Imran Ismail, Muhammad Hazim Hamidon, Mohd Zulhilmie Mohd Sofi,
Nur Shahirah Azmi

Faculty of Chemical Engineering, Universiti Teknologi MARA, Pasir Gudang, Masai, Johor, Malaysia

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ABSTRACT

Bleaching process is one of the crucial processes in palm oil refining. An adsorbent known as bleaching clay is widely used in the palm oil refining industry to adsorb the unwanted color pigments and a wide range of other impurities. A study was carried out to seek for adsorbents alternatives that possess similar characteristics to bleaching clay from renewable sources. Three different adsorbents which are peanut hulls from peanuts, press mud from sugar refinery waste and rice husks from the paddy process plant were chosen. These renewable bleaching alternatives from waste materials were chosen because of their characteristics, which are able to adsorb unwanted substances in crude palm oil. The analysis of free fatty acids (FFA), Peroxide value (PV), carotene value and color was carried out to analyze the characteristics. The characteristics of each alternative were examined and tested to analyze their bleaching efficiency. The conducted analysis has proven that press mud was able to adsorb the greatest value of FFA in crude palm oil, as well as reducing the color of crude palm oil to the desired color in bleached palm oil with bleaching clay. Crude palm oil bleached with press mud has the lowest value of FFA and color.

KEYWORDS: Peanut Hulls, Press Mud, Rice Husks, Refining, Bleaching, Palm Oil Refining.

INTRODUCTION

Refining is a process where moisture, insoluble impurities, free fatty acids (FFA) and oxidation product were kept at a minimum level. There are all together four processes in the palm oil refining process, which retains as much as possible the tocopherols and tocotrienols due to their antioxidant effect which can maintain the palm oil for a longer time. They are degumming, neutralization, bleaching and deodorization. Out of all four processes, bleaching is one of the processes that were examined due to its ability to reduce contaminants such as FFA, peroxide value (PV), carotene and color. It is the only process where adsorption of coloring pigments and other minor constituents as well as oxidation products was carried out [7]. Removal of these substances is essential in the refining of oil as it improves the quality of the oil, especially the stability and sensory of the palm oil [1]. The improvement in the color of the palm oil is due to the removal of organic compounds such as carotenoids especially beta-carotene and their derivatives, xanthophylls, chlorophyll, pheophytin, gossypol and their degradation products that give undesirable color to the oils [1].

Bleaching process is carried out by contacting the palm oils with an adsorbent in the absence of oxygen. Natural clay, acid-activated clay, carbon-activated clay and silicates are normally the adsorbents used for the bleaching process [12]. Activated carbon is one of the widely employed adsorbent because it has large surface area and also high adsorption capacity as well as surface reactivity [10]. However, the usage of carbon-activated clay is costly, but not effective [17]. Besides having a carbon-activated clay which is expensive and less efficient, continuous usage of bleaching clay also disturbs the ecosystem especially the habitats of flora and fauna. Therefore, the first objective of the experiment is to find other alternatives that can perform the bleaching process as efficient as the bleaching clay and without giving much impact to the environment.

Studies that were made have proposed peanut hulls, press mud and rice husks as other alternatives for bleaching clay. These three adsorbents were chosen due to their renewable sources and most importantly, they are waste materials. These alternatives have been proven to be good adsorbents in the industry. Peanut hulls and rice husks are good adsorbents for the removal of metals from wastewater [11,15], while press mud is good for extraction of heavy metals from soil [7]. It would be a great advantage to use carbonized peanut hulls, press mud and rice hulls as a partial or a full substitute to the bleaching clay. In the context of peanut hulls, they are plentiful, inexpensive and a renewable resource [3]. The carbonization process for peanut hulls is simple and cheap, while the spent carbonized ash is easily degradable and has little impact on the environment. The filter muds, which is a waste that is obtained from sugar industry was investigated and was compared with a commercial bleaching earth. As a waste that will be disposed, press mud can be converted into something useful,

which is as an alternative to bleaching clay. In addition, the disposal of press mud can cause huge environmental problem [1]. For rice husk, the recycling methodology of rice husks related to the adsorption of liquid and free fatty acids phases has been previously studied [8]. The adsorption properties of activated rice husks in adsorbing harmful heavy metals in water have also been reported [14]. Therefore, all the waste material has potential as bleaching alternatives due to the fact that the sources can be gained from the other suppliers. There is a significant relationship between the relationship flexibility with the competitiveness of the suppliers [18].

METHODOLOGY

Three different samples collected were discovered to have the same method of preparation, although they are all from different sources [1, 7, 9]. The methods are divided into 4 steps which are sample preparation, carbonization process, bleaching experiment and analysis of the data.

Step 1: Sample Preparation

For sample preparation, most of the sample is readily available because our samples are waste taken from products such as peanut hulls, rice husks and press mud. Sample of peanut hulls is available from local shops nearby. The peanut hulls were washed well with distilled water, dried in an oven at 100 °C overnight, ground into powders and were sieved to pass a mesh of size screen of 100 µm. While rice husk rice collected at the paddy process plant which is situated in Kedah, Malaysia. The rice husk was grounded and then were dried at 105 °C for 3 hours in atmospheric air and the small fragments retained were removed using 30-mesh sieve. The press mud was taken from a sugar factory situated at Central Sugar Refinery in Selangor, Malaysia. For preparation of filter mud ash, fresh filter mud samples from sugarcane refining industry were air dried in the shade in an open field until the moisture content is less than 4 %. The dried filter mud samples were grounded into powders and were passed through a sieve of 200 mesh. The obtained powders were stored in sealed plastic bags.

Step 2: Carbonization Process

The carbonization for each sample is based on the past research articles, which conclude the different way of carbonization for each type. Dry ground hulls were placed in an aluminum crucible in a muffle furnace that was previously heated to the desired temperature. The desired temperature for the burning of peanut hulls is 700 °C for 60 minutes. The carbonized hulls were cooled and kept in desiccators until use. For rice husk sample preparation, the rice husks were carbonized in a stainless steel cylinder with external and bore diameters of 50 and 44 mm, respectively. Carbonization was carried out at 300-800 °C using an electric furnace. The furnace temperature was increased linearly from room temperature to the desired temperature [9]. The carbonized rice husks were kept for the bleaching experiment. The press mud samples were dried in an air atmosphere until the moisture content has reached to less than 4%. It is then heated in the muffle furnace for 2 hours at 800 °C for carbonization. The ash samples were then cooled in a desiccator at room temperature and are stored in an amber glass bottle[1].

Step 3: Bleaching Experiment

The bleaching experiments were conducted. A sample of 20 grams of crude palm oil was stirred and heated together with 0.4 grams of carbonized peanut hull samples in a rotary evaporator at 100 °C in a hot water bath under reduced pressure for 20 minutes. The oil was then filtered through a filter paper to remove carbonaceous adsorbent or bleaching earth. The bleaching process for rice husk was also conducted using the same procedure, similar to the bleaching experiment with rice husk as the characteristic of peanut hulls is almost the same as peanut hull in seed hulls family [9]. The bleaching experiments were carried out under vacuum in a two-necked flask equipped with a stirrer and contact thermometer for press mud. About 100 grams of neutralized oil were mixed with 2 grams of the ash samples in the two-necked flask. The bleaching process was carried out under a vacuum (9 mm Hg) at the constant temperature of 110 °C for 30 minutes. The bleached oil was then cooled under vacuum to 70 °C. It is then filtered through a filter paper to remove the adsorbent [1].

Step 4: Analysis

For the analysis, there are few types of impurities that we needed to analyze after the bleaching experiments. The FFA and peroxide value were determined by using the American Oil Chemists' Society (AOCS) method. For FFA analysis, all five oil samples containing bleached palm oil with peanut hulls, bleached palm oil with press mud, bleached palm oil with rice husk, bleached palm oil with bleaching clay and crude palm oil are placed into five conical flasks marked. These samples are then heated until melted on a hot plate surface model SB 300 to ease the process of mixing with chemicals. All 5 oil samples are weighed in analytical weighing balance of model GR-202. Each of 5 oil sample is added to 50 ml of Isopropyl Alcohol (IPA). Titration method is carried out using sodium hydroxide of 0.1019 molar concentrations. Each of the oil samples is titrated with sodium hydroxide while swirling the solution until the mixture of oil samples with IPA and sodium hydroxide turn to

light red. The amount of sodium hydroxide used for each oil sample is noted and recorded for calculations by using equation (1).

$$\text{Free Fatty Acid Value} = (25.6 \times \text{Titration Value (TV)} \times N) / (\text{Mass}) \quad (1)$$

Equation (1) used to calculate the FFA content was given by the laboratory analysis assistant in company A. 25.6 is a fixed value in the equation, while TV represents the amount of sodium hydroxide titrated into the oil mixture. N represents the molar concentration of sodium hydroxide, which is fixed at 0.1019. Mass represents the mass of bleached palm oil and crude palm oil used in conducting the analysis for FFA. The amount of sodium thiosulphate used in titration for each oil mixture in the conical flask is observed and recorded to determine the calculation for peroxide value by using equation (2).

$$\text{Peroxide Value, PV} = (\text{Titration Value (TV)} \times N) / (\text{Mass}) \quad (2)$$

Equation (2) is a formula given by the laboratory analysis assistant in company A. It is used to calculate the PV in bleached palm oil as well as crude palm oil. TV represents the amount of sodium thiosulphate titrated into the oil samples, while N represents the molar concentration of sodium thiosulphate which is fixed at 10.25. Mass is the mass of oil used to conduct the analysis of PV. Five oil samples are heated until melted on a hot plate surface model SB 300 to ease the process of mixing with chemicals. The heated oil samples are then poured into five flat bottomed flasks and were weighed on a weighing balance with weight ranging from 0.25 to 0.27 grams. All five oil samples in the flat bottomed flasks are then added with isooctane chemical until the level of the solution reached to the level marked on the flat bottomed flask. Each of the oil sample is then poured into a UV container and is placed into the ultra violet (UV) spectrophotometer model U-1900. The carotene value in each sample is determined. Results shown by the machine are observed and recorded. For color analysis, all five oil samples containing bleached palm oil with peanut hulls, bleached palm oil with press mud, bleached palm oil with rice husks, bleached palm oil with bleaching clay, crude palm oil are placed into five conical flasks. The first sample containing bleached oil with peanut hulls was filled in a rectangular fused glass cell (one each of 1” and 51/4”). The glass cells then were placed inside Lovibond Tintometer equipment. It is arranged with two adjacent fields of view which seen through the viewing tube, so that the product in the sample field and a white reflective surface in the comparison field are the observed side by side that suitably illuminated.

FINDINGS AND DISCUSSION

Free Fatty Acid

Free fatty acid contents were determined. Two chemical solutions which are sodium hydroxide and IPA were used in the analysis process. Sodium hydroxide was titrated into each conical flask containing the oil samples and was swirled until the oil samples turn to red. A change to red color indicates that the reaction has stabilized and that it is the correct amount used in neutralizing the mixture containing isopropyl alcohol, sodium hydroxide and FFA in the conical flasks. The amount of sodium hydroxide titrated was observed.

Table 1: Results for FFA value

Sample	Mass (g)	Titration Volume, (TV) (ml)	Constant (N) (mol)	FFA Value (ml.mol/g)
Peanut hulls	4.75	7.3	0.1019	4.009
Press mud	4.45	3.8	0.1019	2.227
Rice husks	4.05	6.4	0.1019	4.122
Crude palm oil	5.10	8.0	0.1019	4.092
Bleaching clay	4.22	6.5	0.1019	4.018

FFA content is the most used criterion for determining the quality of palm oil, as it must not exceed 5% (expressed as palmitic acid) [5]. Fatty acids are generally present in oils as part of the triacylglycerol molecule [5]. The presence of free fatty acids in palm oil is an indication of the impairment of oil quality [6]. There are two types of fatty acids, which are trans-fatty acids and cis-fatty acids. Trans-fatty acids is the type of fats that lowers good cholesterol and increases the level of bad cholesterol in the body [4]. It is also harmful to heart health and cause cardiac death [16]. Based on the result shown, the bleached oil using press mud used the least amount of sodium hydroxide during the titration process which requires only 3.8 ml, followed by rice husks with 6.4 ml, bleached palm oil using bleaching clay with 6.5 ml, peanut hulls with 7.3 ml and finally crude palm oil with 8.0 ml. As inserted into the equation (1), higher amount of sodium hydroxide used resulted in a higher content of free fatty acids. As shown in the result, the least amount of FFA was proven on bleached oil using press mud with 2.227 ml.mol/g, followed by bleached oil with peanut hulls of 4.009 ml.mol/g, followed by crude palm oil with 4.018 ml.mol/g, followed by bleached oil using bleaching clay with 4.092 ml.mol/g and finally bleached oil using rice husks.

Peroxide Value

Acetic acid mixture with chloroform in a ratio of 3:1 is mixed into each conical flask containing bleached palm oil using peanut hulls, press mud, rice husks and bleaching clay. The other one is a crude palm oil which has not yet undergone any bleaching process. A 5 ml solution of potassium iodide was added into each of the oil mixture and is left in a dark ambient for a minute before proceeding to the next step. About 2ml of starch is added into the solution which causes the reaction to occur and caused the color of the solution to turn black. Titration process was carried out in each conical flask which mixture that has turned black color. The amount of sodium thiosulphate used in the titration method is observed as the amount of sodium thiosulphate used is important in the calculation of peroxide value. The amount of sodium thiosulphate used for titration is the least in bleached palm oil using bleaching clay with 0.4 ml, followed by crude palm oil with 0.8 ml, followed by peanut hulls with 1.7 ml, followed by press mud with 1.8 ml, and finally bleached oil using rice husks with 2.0 ml. The higher amount of sodium thiosulphate during the titration process indicates higher peroxide value in the oil sample based on the equation (2). As reflected by the amount of sodium thiosulphate used, the peroxide value shows the highest amount of bleached oil using peanut hulls with 7.478 ml.mol/g, followed by rice husks with 6.327 ml.mol/g, press mud with 5.458 ml.mol/g, crude palm oil with 2.246 ml.mol/g and finally bleached palm oil using bleaching clay which is 1.054 ml.mol/g.

Table 2: Results for PV

Sample	Mass(g)	Titration Volume (TV) (ml)	Constant, (N) (mol)	Peroxide Value (ml.mol/g)
Peanut hulls	2.33	1.7	10.25	7.479
Press mud	3.38	1.8	10.25	5.459
Rice husks	3.24	2.0	10.25	6.327
Bleaching clay	3.89	0.4	10.25	1.054
Crude palm oil	3.66	0.8	10.25	2.246

Peroxide value is measure of oxidation or rancidity of crude palm oil [13]. Over exposure of palm oil towards oxygen will lower down the oil quality. Therefore, a high peroxide value should be avoided as it only brings adverse effect to the oil constituents. The dark or black color contained in palm oil is also affected from oxidation [13]. This is proven when the cooking oil turns black after time when it is used repeatedly during frying. Besides rancidity and dark color, high peroxide value is also associated with the bad odor as the oil is being exposed to oxygen during frying [13].

The result of peroxide value shown by bleached oil from other adsorbents which are peanut hulls, press mud and rice husks did not reach our expectations. This is due to the oil samples that were heated in excess. Peroxide value is very sensitive towards heat and excess temperature. During the laboratory analysis that was carried out in company B, the oil samples were accidentally heated in excess. The analysis process in determining the peroxide value was carried out manually without any automatic controller or sensor to control the temperature of the hot surface. Therefore, the peroxide value has been disturbed and the results shown increased excessively to an unexpected level. Despite the accident that occurred during the analysis process of peroxide, it was shown in the results that press mud adsorbs the highest amount of peroxide in the oil sample, leaving behind only 5.458 ml.mol/g amount of peroxide in the oil sample compared to peanut hulls and rice husks, each with 7.478 ml.mol/g and 6.327 ml.mol/g. Therefore, the best alternative to substitute bleaching clay in reducing the peroxide value in crude palm oil is press mud.

Carotene Value

Carotene content in the samples was to be determined. All five samples containing oil samples of bleached palm oil using peanut hulls, press mud, rice husks and bleaching earth as well as crude palm oil were placed into five different flat bottomed flasks with line indicator. An isooctane chemical is used during the analysis and is poured into each flat-bottomed flask until it reached the calibrated line. Isooctane is a fast absorbing chemical. Therefore, gloves must be worn all the time to prevent contact with skin. Isooctane was also chosen because it is not carcinogenic and does not cause cancer compared to other alternative chemical such as n-hexane. Each sample is inserted into a UV container and is placed into the UV spectrophotometer to determine the carotene value.

Table 3: Results for carotene value

Sample	Carotene Value
Peanut hulls	2.165
Press mud	2.356
Rice husks	2.218
Bleaching clay	1.717
Crude palm oil	2.083

Crude palm oil is refined in the refining process to remove contaminants such as FFA and peroxide that causes it to be dark in color. Although it is refined to reduce all levels of contaminants, the refining process carried out must still retain 80% of the carotenes present in the crude palm oil [2]. This is because oil from the

palm fruit is a rich natural source of provitamin A carotenoids and can be used as an alternative for vitamin A supply [2]. Therefore, carotene content in palm oil should be retained to maintain the good benefits in palm oil. The result in the table reveals that press mud is the most efficient in retaining the carotene value. There was a significant difference between press mud, rice husk and peanut hulls. Press mud shows the most efficient result, followed by rice husks and peanut hulls. The bleached palm oil using press mud shows the most positive result as it is able to retain a higher value of carotene, which is 2.356, followed by rice husks of 2.218 and peanut hulls of 2.165.

Although the value of carotene in bleached palm oil has to be maintained at 80 %, the results shown in the table has exceeded the carotene content in crude palm oil itself. This happens because carotene content in palm oil is very sensitive to heat and excess temperature. While conducting the analysis in company B, the equipment was conducted manually without any controller or sensor to detect if excess heat was supplied to the oil sample. This unexpected high increment of carotene value in oil samples bleached with adsorbent alternatives is therefore due to the excess heat supplied. However, despite the incident that occurred, press mud shows its ability of retaining the highest value of carotene. Therefore, it was proven that if the incident did not occur, press mud could be the best alternative in substituting bleaching clay as an adsorbent in the carotene value.

Color

Based on the tables above, the color that has been changed from crude palm oil using Lovibond Tintometer Model E to observe and measure the sample color content. We measured the color changes in based on red and yellow color only and do not include blue as its do not involve in our product nor analysis procedure. The reduction color by using press mud marked the lowest reading of color reduction which is 18R and 18Y, followed by rice husk 20R and 20Y and peanut hull 22R and 22Y. Peanut hulls does not show any different color changes from the crude palm oil as the readings remain constant. It also proves that press mud is the most suitable bleaching earth for color reduction compared to the others as the reading of the bleached palm oil using bleaching clay is almost the same as the press mud which is 17R and 16Y. Based on the result shown in Table 4, press mud is the best alternative for color reduction compared to peanut hulls and rice husks.

Table 4: Overall results

Sample	FFA (ml.mol/g)	P (ml.mol/g)	Carotene	Colour
Peanut hulls	4.009	7.479	2.165	22R+22Y
Press mud	2.227	5.459	2.356	18R+18Y
Rice husks	4.122	6.327	2.218	20R+20Y
Bleaching clay	4.092	1.054	1.717	17R+16Y
Crude palm oil	4.018	2.246	2.083	22R+22Y

CONCLUSION AND RECOMMENDATIONS

The first objective of reducing the environmental impact due to trimming of caves and hills for bleaching clay was accomplished. By using peanut hulls, press mud and rice husks which are waste materials from peanuts, sugar refining waste and paddy process industries, there is a no need for a disposal area to dispose these waste materials. Instead, of being solely waste materials, these substances are converted into something useful to the palm oil refining industry. Therefore, there is a no need for a huge disposal area for disposing these waste materials. Other than that, the objective of seeking other alternatives which possesses similar characteristics as bleaching clay was a success. Out of all three alternatives which are peanut hulls, press mud and rice husks, press mud possesses the most similar characteristics to bleaching clay. Due to its characteristics being similar to bleaching clay, it is therefore able to adsorb the contaminants found in crude palm oil as much as bleaching clay does. Therefore, it came to a conclusion that press mud is the best alternative to substitute bleaching clay as an adsorbent in the bleaching process.

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