ISBN 979-587-621-1
PROCEEDINGS
SRIWIJAYA INTERNATIONAL CONFERENCE ON ENGINEERING, SCIENCE & TECHNOLOGY

# Integrated Adsorber Engineering on Waste Cooking Oil Purification

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**Abstract:** During the frying process, cooking oil oxidized and hydrolysed so triglycerides break down into other compounds like free fatty acid, peroxide acid and the water content is higher than the new cooking oil so it is not feasible to reuse. Hence, it is necessary to improve the quality of waste cooking oil through adsorbtion process. The present work aimed to decrease the amount of peroxide value, free fatty acid value, and acid value of waste cooking oil using integrated adsorber equipment through the medium of activated carbon made from coconut shell. The experiment were conducted at temperature 60ºC, 90ºC, and 120ºC. And also100 rpm, 200 rpm, and 300 rpm. The result of the study showed that the minimum percent of free fatty acid is 0.768% at a temperature of 120ºC and 300 rpm stirring speed, where as the percent free fatty acid before adsorption is 3.1744 %. The minimum acid value of the oil obtained at a temperature of 120ºC and the stirring speed of 200 rpm is equal to 1.9074, whereas the acid value before adsorption is 3.927. While the minimum peroxide value derived from temperature and stirring speed of 300 rpm at 60ºC is 5.38, whereas the peroxide value before adsorption is 20.59.

**Keywords:** Activated carbon; Adsorption; Purification; Waste Cooking Oil.

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## 1. INTRODUCTION

Cooking oil is one of the principal component in the food processing that act as a heat transfer medium on the surface of the material [1]. During the cooking process, the food is subjected into simultaneous heat and mass transfer, accompanied by numerous chemical reactions. When heated at high temperatures accompanied by contact with air, cooking oil will eventually degrade characterized by changing the color, odors, and the chemical structure as a result of oxidation, hydrolysis, polymerization, and browning reaction. [2-5] The reaction products of oil oxidation, such as peroxide, free radicals, aldehydes, ketones, hydroperoxide, polymers, oxidized monomers and various other oil oxidation products reported an adverse influence on health [6]. Cooking oil refining efforts required in order to reuse waste cooking oil for economic reasons which not harmful to human health.

Waste cooking oil treatment is generally done by adsorption [2-4]. Adsorption is process of separating certain components of the fluid to the surface of the solid substance that absorbs (adsorbent). The separation occurs because of differences in the molecular weight or porosity that causes some molecules bind tighter to the surface of the other molecule. As for the conditions of the an adsorption process, there is a substance which adsorbs (adsorbent), a substance adsorbed (adsorbate), and time of agitation until the adsorption run balanced [6].

In accordance with the type of bond that exists between adsorbed material molecules and adsorbent surface, adsorption process divided into two types, which are physical adsorption and chemical adsorption. Adsorption using activated carbon included into physical adsorption. Physical adsorption occurs due to the van der Waals interactions, ie, two or more particles in suspension, which has a different parameter, then merge into one so that the shape and the molecular weight of this combination increased [7].

A substance can be used as an adsorbent for separation purposes when it has selective adsorption, porous (having a large surface area per unit of mass and has a strong holding capacity to substances to be separated physically and chemically [8].

Activated carbon is a porous solid, consisting mostly of carbon-free element and each are covalently bonded. Thus, the surface of activated carbon is non polar [4]. Activated carbon not only contains carbon atoms but also contains small amounts of oxygen and hydrogen chemically bonded in the form of functional groups which are varied, for example carbonyl (CO), carboxyl (COO), phenols, lactones, and some ether groups. Based on physical properties, activated carbon has several characteristics, which include solid black, tasteless, odorless, hygroscopic, insoluble in water, acids, bases or organic solvents [8-10].

Activated carbon made through two stages: carbonation stage and activation stage. Carbonation is a process in the room without the presence of oxygen and other chemicals, while activation is required to change the results of carbonation into the adsorbent which has a large surface area [11]. Carbon is produced through the raw materials carbonization process, so most of the pores are still covered by hydrocarbons, tar, and other components, such as ash, water, nitrogen, and sulfur, so that the absorbency is still low. To improve absorption capacity of carbon, such materials can be converted into carbon through the activation process [8-12].

The activation process is a treatment of carbon which aims to enlarge the pore that is by breaking the bonds of hydrocarbons or oxidizing surface molecules that change the carbon nature, both physical and chemical, that surface area become larger and influence on the adsorption capacity [13,18].

The activated carbon pore size range from 10 to 250 A °. The pore size can be divided into three categories which are macropores, mesoporous and microporous. Macropore is defined as the pore size of activated charcoal which have diameters larger than 250 A° with a volume of 0.8 mL / g and a specific surface between 0.5 to 2 $m^2$ / g. Mesopores have diameters ranging between 50-250 A° with a volume of 0.1 mL / g and a specific surface of 20-70 $m^2$ / g. Microporous is the activated carbon porous with a diameter smaller than 50 A° and is divided into three parts:

a. Maxi Microporous
   Maxi micropores are pores with a pore diameter of 25-50 A°, that can be used to absorb the pigments of plants and very good for the adsorption of molasses.

b. Mesi microporous
   Mesi pore diameter is ranging between 15-25 A°, which is excellent for absorbing dye, especially methylene blue.

c. Mini microporous
   The pore diameter of mini micropores is smaller than 15 A°, and can be well used for the absorption of iodine and phenol [14].

Microporous plays an important role in the adsorption process because the total volume of microporous holes is greater than the total volume of macropores and mesopores. Macropores and mesopores only serves as transport pore (the road to the microporous). Pore size distribution is an important parameter in terms of the ability of activated carbon absorption of the molecule in varies size [17].

There are several factors that affect the activated carbon absorption, which is the properties of activated carbon, the properties of component absorbed, the properties of the solution and contact systems. Activated carbon absorption capacity is an accumulation or concentration of the components in the surface / interface in two phases. When the two phases interact with each other, it will form a distinct new phase with each previous phase. This is due to the attractive forces between molecules, ions or atoms in two phases. These attractive forces known as van der Waals. In certain circumstances, atoms, ions or molecules in the interfacial region experiencing imbalances style, so as to attract other molecules until the equilibrium of forces is reached [15,17].

This present study aims to investigate the effectiveness of the integrated adsorber by varying the temperature and stirring speed on the quality of the waste cooking oil in terms of the content of free fatty acid, peroxide value, and the acid value through the medium of activated carbon from coconut shell.

## 2. EXPERIMENTAL DETAILS

2.1. Materials

Integrated adsorber used in this research work based on function of temperature and stirring speed during the adsorption process.

Integrated adsorber consisting of the following equipment:

a) Material iron frames
b) Two filters in the form of a tube stainless steel vessel with 28 cm diameter and 20 cm of height equuiped with 60 and 100 mesh sieve.
c) Heating vessel in the form of stainless steel vessel with 28 cm diameter and 25 cm of height.
d) Heating coil
e) Thermocouples
f) Set the control panel
g) Agitator
h) Valve
i) Wheels

The equipments used for activation of activated carbon include:

a) Ring mill
b) Furnace
c) 100 mesh sieve and shaker

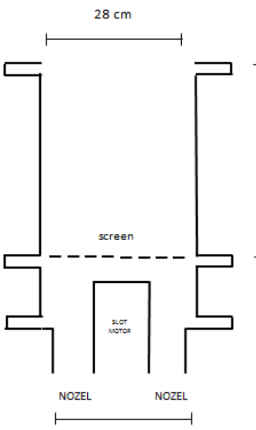
Materials used include:

a) Waste cooking oil brands Frytol
b) Activated carbon from coconut shell
c) Ethanol 95%
d) KOH 0.1M
e) NaOH 0.1M
f) Phenolphthalein indicator (pp)

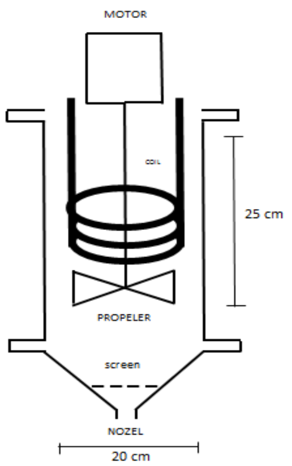
2.2 Preparation and Activation of Activated Carbon

Activated carbon is pulverized to 100 mesh using a ring mill. Sieve used in order to obtain activated carbon in the uniform size. Furthermore, activated carbon is activated physically in the furnace at a temperature of 800ºC for 3 hours.

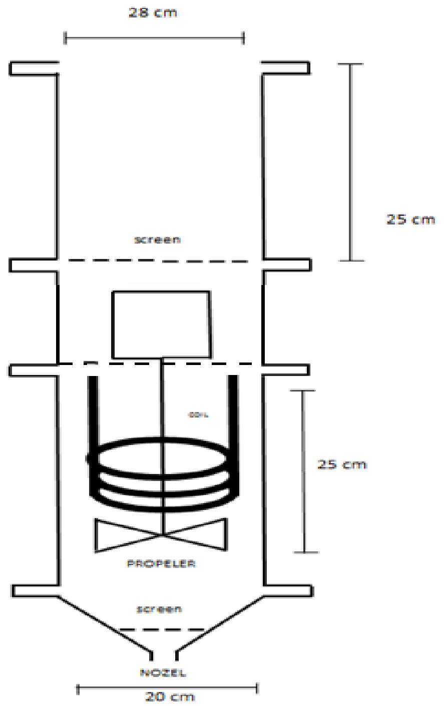
2.3. Integrated Adsorber Dimension



a. Mechanical Filter



b. Heating Vessel



c. Integrated Adsorber

2.4 Waste Cooking Oil Preparation

Cooking oil is heated at a temperature of 110ºC to vaporize water and volatile matter that still contained in cooking oil due to the frying process.

2.5 Adsorption Process

A total of 3 liters of waste cooking oil fed into an integrated adsorber and then heated up to 60, 90, and 120 ºC. 150 grams of activated carbon mixed into the oil while the oil is being stirred with a variation of 100, 200, and 300 rpm for 90 mins. Furthermore, activated carbon allowed to settle for 2x24 hours inside the equipment before leave the equipment through a valve which is attached at the bottom of the integrated adsorber.

## 3. RESULTS AND DISCUSSION

### 3.1. Integrated Adsorber Design

Cooking oil purification process is done by using an integrated adsorber. This equipment works on the principle of adsorption and influenced by several factors such as temperature, stirring speed and residence time so that the process will takes place optimally. There are three main processes in the integrated adsorber, which are pre-treatment, adsorption and filtration. Pre-treatment is done by using a double filter with sieve size each 60 and 100 mesh. The intention of using double filters is to separate large-sized impurities from waste cooking cooking oil. Once filtered mechanically, waste cooking oil is heated at a temperature of 110ºC for three hours to remove the water and unwanted volatile substances.

The adsorption process takes place on a heating vessel. There are are agitators, heating coils, activated carbon inlet valve, and outlet oil valve which is equipped with a 200 mesh sieve in the heating vessel. Activated carbon inlet valve serves as a place for activated carbon to enter into the heating vessel. The outlet valve is equipped with a filter size of 200 mesh, for separating cooking oil from the activated carbon. The heating coil is installed automatically with the control panel which can reach 300ºC. Temperature arragement aims to accelerate the binding reaction impurities during adsorption and activated carbon homogenization. Rising temperatures cause an increase in the speed collisions between particles which accelerate adsorption process. Agitator also installed automatically with the control panel by the range of 100-900 rpm. Stirring function is to disperse activated carbon in cooking oil. In addition, stirring also plays a huge role in homogenizing heat distribution in the adsorption process.

Activated carbon is derived from coconut shells and physically activated by heating at temperature of 800ºC for three hours [12]. Within 60 hours, the activated carbon even become saturated and no longer active due to contact with air and water vapor so that the reactivation process is required. The activation process aims to increase the porosity of the surface area per unit of carbon. Heating at high temperatures cause volatile matter and impurities detached from the carbon pores so that the activated carbon can be reused. One gram of activated carbon has approximately 500-1500 $m^2$ surface area, making it very effective in absorbing particles of very fine size of $10^{-2}$-$10^{-7}$ mm [18].

This present study aims to investigate the effectiveness of the equipment by varying the temperature and stirring speed in order to obtain optimum temperature and stirring speed on the adsorption process waste cooking oil using an integrated adsorber.

The settling time required by activated carbon in the integrated adsorber is 2x24 hours. Then, the filtering is done directly through the cooking oil outlet valve.

### 3.2. Cooking Oil Analysis After Adsorption

Waste cooking oil after adsorbed analyzed according to three parameters, which are the free fatty acids, acid value and peroxide value. Three parameters are obtained from the treatment temperature and stirring speed in the process of adsorption on adsorber purification using the integrated waste cooking oil.

a. Effect of Temperature and Stirring Speed against Percentage of Free Fatty Acid

Based on the results obtained, the percentage of free fatty acids ranged from 0.7680% to 1.28%, while the percentage of free fatty acids prior to the adsorption process is 3.1744%. The effect of temperature and stirring speed on the acid value can be seen in the picture below.

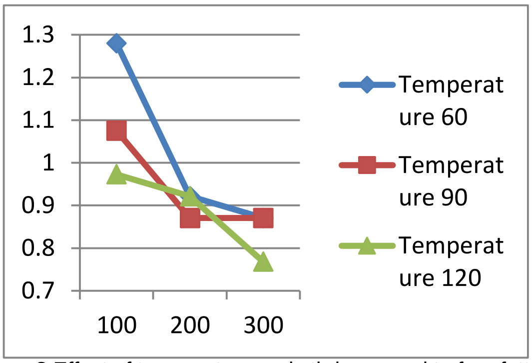



**Figure 2** Effect of temperature and stirring speed to free fatty acid

Figure 2 shows that the percent of free fatty acids tend to decrease with increasing temperature and stirring speed. Based on percent free fatty acids testing using a volumetric method with 0.1 M of NaOH, percent minimum of free fatty acids is 0.7680% at a temperature of 120ºC and stirring speed of 300 rpm.

Activated carbon adsorption occurs physically due to the van der Waals force between the solute and the surface of the absorbent media. Van der Waals force is the gravitational pull and repel between atoms, molecules, surface and between other molecules that caused a chemical bond between them. If the van der Waals force between the solute (FFA, impurities) and the surface of the absorbent media is greater than the van der Waals force between solvent and solute, the solute will be attracted by the surface of the absorbent media known as adsorption process.

Adsorption velocity is directly proportional to the temperature rise as shown in Figure 3. When the adsorption temperature is high, then the movement of the particles in the solution become faster and collide each other and break the bond between solute and solvent. This makes the van der Waals force between the solute and the solution is lower than the surface tension force and the media so that the solute dissolved substances (impurities) attracted and absorbed by the surface of activated carbon media. This is in line with the results of research that shows the percentage of free fatty acid minimum obtained at 120ºC which is the highest temperature variable in this study.

Stirring purpose is to distribute the activated carbon to all parts of the volume of solution so that the activated carbon has a uniform area and time contact against impurities in the solution. The percentage of free fatty acids is inversely proportional to the speed of stirring. The percentage of free fatty acids tend to decrease with the increase of stirring speed, but stirring with high velocity can cause the damage in adsorbent structure so that the adsorption process will become less than optimal. Based on this research, the minimum percent free fatty acids obtained in the stirring speed of 300 rpm. This shows that the stirring speed of 300 rpm is the optimum stirring speed to get the best results.

Free fatty acids are fatty acid from free acid groups which are not bound as triglycerides. Based on the research results, the higher the temperature and stirring speed on the adsorption process, the percentage of free fatty acids is getting smaller.

b. Effect of Temperature and Stirring Speed against Acid value

Based on the research results, the value of the acid value ranges between 1.7952 Meg / Kg to 2.805 Meg / Kg while the value of the acid value prior to the adsorption process is 3.927 Meg / Kg. The effect of temperature and stirring speed on the acid value can be seen in the picture below.

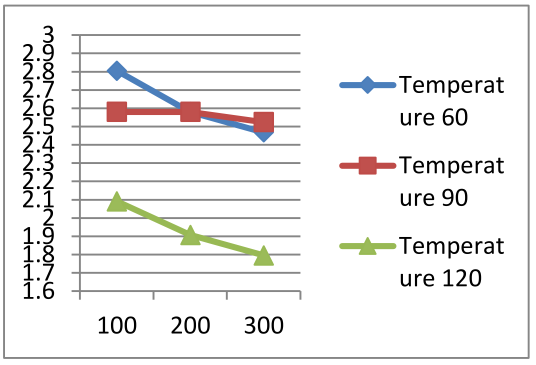



**Figure 3** Effect of temperature and stirring speed against acid value

Figure 3 shows that that the acid value tends to decrease with increasing temperature and stirring speed. This is based on the theoretical basis that the function of the amount of the acid value is directly proportional to the percent of free fatty acids. If the percentage of free fatty acid in cooking oil decreased, the acid value will also decreased. Slope in acid value caused by free fatty acid which are classified into saturated fatty acids (palmitic, stearic and myristic) adsorbed by the activated carbon in the adsorber. Saturated fatty acids are formed as a result of heating cooking oil at high temperatures when frying which can cause rancidity.

Analysis for acid value done in volumetric manner using KOH solution 0.1 M. The test results showed the minimum acid value of the cooking oil obtained at a temperature of 120ºC and stirring speed of 300 rpm is 1.7952.

The acid value is a number that indicates the amount (mg) of KOH required to neutralize 1 gram of chemical compounds. Figures show the acid content of free fatty acids contained in the cooking oil. The content of free fatty acids in the cooking oil resulted in an increased number of acid and rancid oil [16]. Based on the research results, the higher the temperature and stirring speed on the adsorption process, the acid value is getting smaller.

c. Effect of Temperature and Stirring Speed against Peroxide value

Based on the research results obtained by peroxide value ranging between 5.83 Meg / Kg to 20.32 Meg / Kg while the value of the number of peroxide prior to the adsorption process is 20.59 Meg / Kg. The effect of temperature and stirring speed on the peroxide value can be seen in the picture below.

Figure 4 shows that the peroxide value tends to decrease with the increase of stirring speed and temperature decrease. Minimum peroxide value obtained from the temperature and stirring speed 60ºC and 300 rpm is 5.38. The downward trend in the most optimum peroxide value is at a temperature of 90ºC.

Peroxide value is the number of milliequivalents of active oxygen contained in 1000 grams of oil or fat. Peroxide is an important indicator that signifies oil damage due to oxidation which takes place when there is contact between the oxygen and cooking oil. In principle, any oxygen and heat the fat or oil will undergo oxidation reaction. Oxygen molecules bound to the double bonds of the unsaturated fatty acids. The double bonds of unsaturated fatty acids undergo the oxidation process to form short chain fatty acids, aldehydes and ketones which can cause odor and unpleasant taste. Fatty acid oxidation involves the formation of free radicals which consists of three basic processes, which are initiation, propagation and termination. In the early stages of the reaction, hydrogen released from unsaturated fatty acids homolitically to form alkyl radicals which occur because of the initiator in the form of a heat, active oxygen, metal or light. In normal circumstances alkyl radical rapidly reacts with oxygen to form peroxy radicals. These peroxy radicals react further with unsaturated fatty acids to form hydrogen perokside and alkyl radicals, then alkyl radicals formed react with oxygen. Thus, the autooxydation reaction or free radical chain reactions that produce hydrogen peroxide continuously occurs. Excessive temperatures will cause higher oxidation rate, so the peroxide value tends to increase. It underlies minimum peroxide value obtained by the treatment of low temperature is 60ºC.

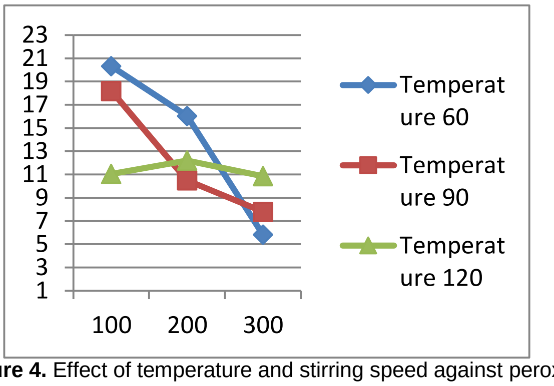



**Figure 4.** Effect of temperature and stirring speed against peroxide value

Stirring speed with higer velocity can cause the hydrogen peroxide that has been absorbed by the activated carbon detached so that the purified cooking oil still contained high amount of hydroperoxide. In addition, the higher stirring speed can ruin the structure of activated carbon and cause disturbance to adsorption process. Based on this research, the minimum peroxide value obtained from the treatment of mixing speed of 300 rpm. This shows that the stirring speed of 300 rpm is not a critical point that could result in damage to the structure of the adsorbent and the release of hydroperoxide from the surface of activated carbon.

Peroxide value is the amount of oxidized fatty acids that are determined by the amount of iodine ($I_2$) which is formed from the reaction of peroxides in the cooking oil with iodine ions ($I^-$) proportional to peroxide concentration samples [16]. Based on the research results, the higher the temperature in the adsorption process, the greater the peroxide value and the higher the speed of stirring the peroxide value is getting smaller.

## 4. CONCLUSIONS

a) Integrated adsorber designed based on a function of temperature and stirring speed on the adsorption process.
b) The higher the temperature and stirring speed, the smaller the percent of free fatty acids. The optimum temperature and stirring speed for percent free fatty acids are 120ºC and 300 rpm with the 0.7680% of free fatty acids.
c) The higher the temperature and stirring speed, the smaller the acid value. The optimum temperature and stirring speed to a decrease in acid value is 120ºC and 300 rpm with acid values of 1.7952 Meg / Kg.
d) The higher the temperature and stirring speed, the smaller the peroxide value, the optimum temperature and stirring speed for peroxide value is 60ºC and 300 rpm with a peroxide value of 5.38 Meg / Kg.

## 5. ACKNOWLEDGMENTS

The author is grateful to the Research Institution of Sriwijaya University for financially supporting this research no. 591/UN9.3.1/LT/2016.

ISBN 979-587-621-1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