REDUCING PEROXIDE VALUE IN USED COOKING OIL USING AMPO AS AN ADSORBENT

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Article

REDUCING PEROXIDE VALUE IN USED COOKING OIL USING AMPO AS AN ADSORBENT

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Abstract
Cooking oil can only be used two or three times, more than that the oil is deemed unsuitable and can harm our health. The reason is, every time the cooking oil is used for frying, an oxidation reaction that is triggered by heat occured and causes the chemical chain bonds in cooking oil become unstable and forms hydroperoxides. The hydroperoxide found in used cooking oil increases the peroxide value. In which, the suitability of cooking oil is based on the peroxide value. In order to reduce the peroxide value in used cooking oil, in this study, Ampo is used as an adsorbent to adsorb the peroxide compound. The procedure used in this study includes a preliminary analysis of the used cooking oil to determine the initial peroxide value. After that, the Ampo adsorbent is activated with 1N Citric Acid to open the pores on the surface of the Ampo. Activated Ampo will then be mixed into 200 ml of used cooking oil with a magnetic stirrer with a stirring speed of 300 rpm and with a temperature of 50°C. The adsorption process was carried out by varying the mass variables of Ampo into 70, 75, 80, 85, and 90 grams, and stirring time of 30, 60, 90, 120, and 150 minutes. After the adsorption process is completed, the cooking oil that has been filtered by the Ampo adsorbent will be analyzed to get the final peroxide value. By knowing the peroxide value before and after the adsorption process, the adsorption isotherm can be calculated. The adsorption process of used cooking oil with Ampo adsorbent proved to reduce the peroxide value, in which stirring times and adsorbent masses were the influential variables in the study. The best result was obtained with 60 minutes stirring time and mass of 75 grams. This condition provided a peroxide value of 0.99 meq/kg, which means that there was a decrease in the peroxide value by 93%. This result is in accordance with the provisions of SNI 3741-2013, with a maximum peroxide value of 10 meq/kg. The maximum adsorption capacity in this study was obtained from the Freundlich equation of 14.6487 mg/gram.

Keywords: Adsorpsition, Ampo, Peroxide value, and Used cooking oil

1. Introduction

Cooking oil is an organic lipid which is often used for frying foods. Cooking oil that has undergone too much frying process will be referred to as used cooking oil and has the characteristics of brown coloured, rancid smell, and causes hoarse throat. Apart from the physical appearance, the cooking oil content itself undergoes several changes that have a negative impact on human health. The frying process causes oxidation, polymerization, and hydrolysis reactions. This study will focus on the oxidation reaction in cooking oil because the oxidation reaction causes the formation of cyclic peroxide and monomer groups which will increase the amount of peroxide contained in cooking oil [11]. The peroxide contained in used cooking oil can be used as one of the suitability parameters of used cooking oil because in general, the peroxide value is used for the measurement of unwanted reactions in food and oil ingredients [3]. The peroxide value according to SNI 01-3741-2013 has a maximum limit of 10 meq/kg [2]. For packaged cooking oil on the market the average peroxide value is 1-2 meq/kg. The increase of peroxide value in used cooking oil is bad for health because it can cause liver damage and cancer in the body [18]. Therefore, adsorbing the peroxide compound contained in used cooking oil can prolong the usability of used cooking oil. This study aims to reduce the peroxide value by using the adsorption method with Ampo as an adsorbent to adsorb peroxide compounds in used cooking oil.

The basic principle of adsorption is the implementation of mass transfer process, both the adsorption of molecules from liquids and gases to the surface of solid objects [6]. The adsorption that occurs in this study is physical adsorption, which is an adsorption caused by the activity of intermolecular forces that exist between the adsorbate and adsorbent [4]. For the selection of the adsorbent itself, according to [6] the adsorbent must have high abrasion resistance, small pore diameter, and high thermal stability. In addition, the reason for choosing Ampo as an adsorbent is because clay has a large surface area, physical and mechanical stability, and a regular layer structure. Moreover, the low costs required for processing are one of the advantages of using Ampo as an adsorbent.

Ampo is a type of snack from Tuban, East Java, which is made of clay. Ampo producer is currently hard to find because there is less interest in Ampo although some people still believe in its efficacy as an alternative medicine. According to [20] Ampo has microbial contamination of 1.07x106 colonies/gr - 6.07x105 colonies/gr but negative for Coliform bacteria, Salmonella sp., and Staphylococcus Aureus. By utilizing Ampo as an adsorbent it is hoped that Ampo can be preserved because its benefits are not only for consumption.

According to [16] adsorption is influenced by several factors:

1. Adsorbent Particle Size: If the size of the adsorbent particles is smaller, the larger the adsorbent's surface area will cause a faster diffusion rate and more adsorbed substances.
2. Contact Time: An adsorption process requires sufficient contact time between the adsorbate and the adsorbent until adsorption equilibrium occurs.
3. Adsorbate Concentration: The more adsorbate being absorbed it will increase the adsorption rate.
4. Adsorbate Molecular Size: In adsorption, the size of the adsorbate molecule is expected to be smaller than the pore diameter of the adsorbent to create a strong attractive force which causes high adsorption rate.
5. Polarity of Substance: For non-polar adsorbents, the non-polar adsorbate molecules will be more powerful than polar molecules to be adsorbed.
6. Temperature: Temperature can increase the rate at which the material will be adsorbed. But too high of a temperature can cause desorption.
7. Mixing: If the stirring process is relatively slow, it is difficult for the adsorbent to penetrate the film layer between the adsorbent surface and the diffusion film, which is a limiting factor that reduces the absorption rate.

Based on the adsorption factors above, these factors can be used as a reference variable for this study. The fixed coditioan include Ampo adsorbent with a size of 60 mesh, 300 rpm stirring process, and a temperature of 50^{o}C. These variables are used as a fixed condition so that they can be a reference for comparison when varying other variable conditions to obtain the best result. The non-fixed variables are the variation of adsorbent mass and contact time between adsorbent and adsorbate. The selection of mass and time ranges for the non-fixed variables is based on the best mass and time results from previous studies. In a study conducted by [2] with Ca Bentonite adsorbent, the optimum adsorbent mass for reducing the peroxide value of 200 ml of used cooking oil was 80 grams. In another study conducted by [22] using bioadsorbent from palm oil empty bunches, founded that the optimum stirring time to reduce the peroxide value of 200 ml of used cooking oil was 150 minutes. By looking at these references, we are able to range the variables needed in the study with a different adsorbent which is Ampo.

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Langmuir Isotherm

Langmuir derived the theory of adsorption isotherms by using a simple model of a solid which adsorbs gases on its surface. This model defines that the maximum adsorption capacity occurs due to the presence of a monolayer of adsorbate on the adsorbent surface. Langmuir isotherm equation is mathematically expressed as

$$Xm/m = \frac{a.C}{1+b.c} \ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

$$m.c/Xm = 1/a + (b/a).C \ldots\ldots(2)$$

Where:
Xm = mass of adsorbate
m = mass of adsorbent (Ampo)
C =equilibrium concentration of substance in solution

By making the m.c/Xm curve against C, a linear equation will be obtained with an intercept of 1/a and slope (b/a), so that the values of a and b can be calculated, from the size of the values a and b that indicates the adsorption power.

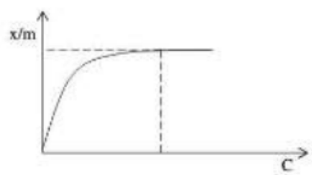

Fig. 1. Langmuir adsorption isotherm curve [7]

Freundlich Isotherm

Freundlich isotherm model is used for non-ideal adsorption and is a multilayer adsorption. The correlation between the amount of substance adsorbed and the concentration is shown in Equation 4 [13].

$$\frac{Xm}{m} = k.C^{1/n} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3)$$

$$\log\frac{Xm}{m} = \log K + \frac{1}{n}\log C \ldots\ldots\ldots(4)$$

Where:
Xm = mass of adsorbate
m = mass of adsorbent (Ampo)
C = equilibrium concentration of substance in solution

K and N are the adsorption constants which value depends on the type of adsorbent and the adsorption temperature. If a log curve (Xm/m) is made against log C, a linear equation will be obtained with a log k intercept and a slope of 1/n, so that the values of K and N can be calculated.

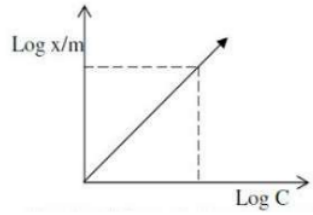

Fig. 2. Freundlich adsorption isotherm curve [7]

2. Methodology

The methodology used in this study is to use Ampo as an adsorbent to adsorb the adsorbate (the peroxide compound in used cooking oil) in an adsorption process. Peroxide adsorption can reduce the peroxide value in used cooking oil as a suitability parameter for reusing used cooking oil. This research was conducted at the Research Laboratory of the Department of Chemical Engineering UPN "Veteran" East Java. The study began in May until August 2020. The Ampo adsorbent was obtained from Trowulan Village, Tuban, East Java. First, the Ampo adsobernt was activated with organic acid, 1N Citric Acid, to open the pores on the surface of Ampo so that it can adsorb maximum adsorbate. Whereas in this study, the used cooking oil was obtained from one of the fried bread sellers at Pasar Pacuan Kuda, Surabaya. The adsorption process was carried out using a stirrer consisting of a magnetic stirrer and beaker glass with fixed conditions at a stirring speed of 300 rpm, a temperature of 50°C, and a volume of 200ml of used cooking oil. The variables used in this study were the Ampo mass of 70, 75, 80, 85, 90 grams and the stirring time of 30, 60, 90, 120, 150 minutes. The analysis of peroxide value was carried out at the Food Analysis Laboratory, Department of Food Technology, UPN "Veteran" East Java by using quantitative titrimetric chemical analysis methods. After the analysis, the adsorption isotherm can be calculated and therefore indicates the equilibrium between the concentration in the fluid phase and the concentration in the adsorbent particles at a certain temperature.

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Research Procedure

A. Material Preparation

The ingredients needed are Ampo, Used Cooking Oil, 1N Citric Acid, and Aquadest. Whereas the tools needed are a series of stirrers consisting of a magnetic stirrer and a 600 ml beaker glass. Other accompanying tools needed are Whatman filter paper, measuring cup, Erlenmeyer, spatula, and 60 mesh sieve.

B. Adsorbent Activation

First, Ampo is made by heating clay to a temperature of 100^{o}C for two hours using heat from a traditional furnace. After it dried, the Ampo is then crushed by using a mill until it reached a uniform particle size of 60 mesh. Ampo is then activated by mixing 200 grams of Ampo in a 400 ml 1N Citric Acid solution using a magnetc stirrer. The stirrer speed is set at 300 rpm with 2 hours activation period. Then the ampo is separated from the filtrate using Whatman filter paper. After that, the activated ampo is dried in the oven at 100^{o}C for about 2 hours until the ampo is completely dry. Finally, the Ampo is re-crushed until the size is uniform again with a particle size of 60 mesh.

C. Adsorbent Application in Used Cooking Oil

Used cooking oil was obtained from the fried bread seller at Pasar Pacuan Kuda, Surabaya and then analyzed to get the initial peroxide value to be compared to the final peroxide value after adsorption. The mass of the activated Ampo will be divided into five parts 70, 75, 80, 85, and 90 grams. Then, these variables are run under constant conditions where the Ampo adsorbent is mixed into 200 ml of the used cooking oil, stirred at 300 rpm using a series of stirring tools, and with a temperature of 50^{o}C. The stirring time is also varied into five parts, 30, 60, 90, 120, and 150 minutes. After the adsorption process, the used cooking oil will be separated from the Ampo adsorbent using Whatman filter paper.

D. Peroxide Number Analysis

The peroxide value analysis of used cooking oil was carried out before and after the Ampo's adsorption process. The analysis is used to obtain the initial and final peroxide values as the basis for calculating the peroxide value reduction percentage and calculating the adsorption isotherm. The peroxide value analysis was carried out at the Food Analysis Laboratory, Department of Food Technology, UPN "Vetean" East Java using the titrimetric qualitative chemical analysis method.

1. Weigh 0.3 - 5 grams of sample in a 300 ml Erlenmeyer.
2. Add 25 ml of chloro-acetate while shaking vigorously and add 1 g of solid KI.
3. Cover immediately and shake for about 5 minutes, leave in a dark place for 30 minutes.
4. Add 75 ml of distilled water and shake vigorously.
5. Add starch solution as an indicator.
6. Titer with standard solution of 0.02N $Na_2S_2O_3$ in duplicate
7. Do a blank determination.

Peroxide Value (mg/kg) =

$$\frac{(V\ sample - V\ blank)\ x\ N\ titran}{Sample\ mass\ (gr)}\ \mathbf{x\ 1000}$$

Where:

meq/kg	: Peroxide Value
ml $Na_2S_2O_3$	: Volume of the $Na_2S_2O_3$ titran
N $Na_2S_2O_3$	: Normality of the $Na_2S_2O_3$ solution

[1]

3. Results

The study results of reducing the peroxide value in used cooking oil by using Ampo adsorbent are divided into two, first is the percentage decrease of the peroxide value in the used cooking oil and second is the adsorption isotherm equation to determine the maximum adsorption capacity.

Percent Decrease in Peroxide Value

The peroxide value data is obtained from the titrimetric method test at the Food Analysis Laboratory, Department of Food Technology, UPN "Veteran" Jawa Timur. Based on the analysis, the following is the analysis results graph of the reduction in peroxide values in this study.

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Table 1. Results from the Analysis of Peroxide Value and Percentage Decreases in Peroxide Value of Used Cooking Oil.

Stir Time (Minutes)	Mass of Ampo (Grams)	Peroxide Value (meq/kg)	Percent Decrease of Peroxide Value
30	70	3,99	71%
	75	2,99	79%
	80	1,99	86%
	85	2,99	79%
	90	3,98	71%
60	70	4,99	64%
	75	0,99	93%
	80	1,49	89%
	85	1,99	86%
	90	5,97	57%
90	70	5,99	57%
	75	5,97	57%
	80	1,99	86%
	85	4,48	68%
	90	6,97	50%
120	70	6,98	50%
	75	7,93	43%
	80	7,98	43%
	85	8,47	39%
	90	8,95	36%
150	70	8,99	36%
	75	8,99	36%
	80	8,49	39%
	85	7,98	43%
	90	8,98	36%

The reduction percentage of the peroxide number is obtained using this formula

$$\% \textbf{ percent decrease in peroxide value} = \frac{\textbf{initial peroxide value}}{\textbf{final peroxide value}} \textbf{ x 100 \%}$$

The initial analysis of peroxide value was obtained from used cooking oil that had not undergone an adsorption process, it was 13.94 meq/kg. This indicates that this cooking oil cannot be reused because the value of peroxides contained has exceeded the predetermined quality standards. The high peroxide value in used cooking oil is caused by an oxidation reaction that occurs as a result of the repeated heating process found in the frying process. This oxidation reaction forms peroxide compounds that destroy the benefits of cooking oil. In addition, the presence of high peroxide compounds in used cooking oil causes the used cooking oil to have a blackish brown color and rancid smell.

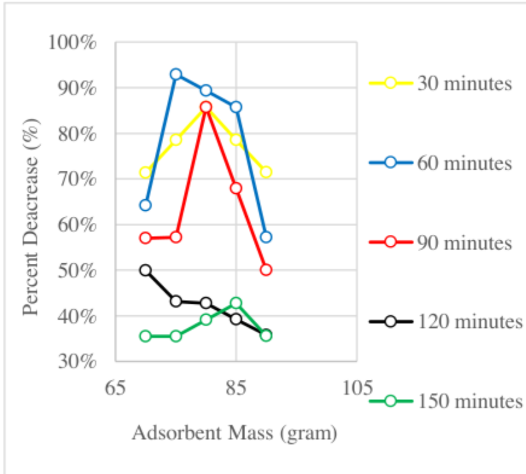

Fig. 3. Graph of Ampo Adsorbent Mass vs Percent Decrease in Peroxide Value

The graph above shows the reduction percentage of the peroxide value by using ampo adsorbent at mass variables of 70, 75, 80, 85, 90 grams and time 30, 60, 90, 120, 150 minutes. The peroxide value of used cooking oil before adsorption is 13,94 meq/kg. In this study, the best conditions for adsorption are 60 minutes stirring time with 75 grams of ampo adsorbent. These conditions are still classified in the average range of previous studies with the same method.

However, the effectiveness of Ampo in reducing the peroxide value in used cooking oil can be said to be superior to other adsorbents from previous studies. The stirring time by using activated charcoal requires 120 minutes. So, with a 60minutes stirring time Ampo requires shorter time to reduce the peroxide value.

Ampo, which already has a large surface area, physical and mechanical stability, regular layer structure provides the advantages to reduce peroxide value quickly. Moreover, activation process to open Ampo's pores is proven to be effective in adsorbing peroxide compounds contained in used cooking oil. The reduction of

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the peroxide value with Ampo as an adsorbent occurs due to a difference in potential energy between the peroxide compunds as the adsorbat and Ampo as the adsorbent which is commonly referred to as the intermolecular force or Van Der Waals force. The interaction between those two particles can be categorized as physical adsorption.

The study resulted a peroxide value of 0,99 meq/kg, which means that there is a decrease in the peroxide value by 93%. These results are in accordance with SNI 3741-2013 the maximum peroxide value of 10 meq/kg. It can be concluded that the adsorbent mass and stirring time affect the reduction of the peroxide value in cooking oil, these results are also in accordance with the theory of the factors that influence the adsorption process. By showing a low peroxide value, the used cooking oil is suitable for reuse without worrying about the bad effects when consumed. This result also shows that there is a molecular movement occured on the surface of the Ampo therefore concludes an increase of peroxide mass transfer to the adsorbent with longer contact time. This adsorption process takes place at a temperature of 50°C. Heating affects a better adsorption of peroxide compounds on the Ampo's surface. This indicates that Ampo has a tendency of increased capacity when heated. The contact between the Ampo particles and the peroxide compounds in the cooking oil will accelerate the adsorption between the two substances. The heating process also evaporates dissolved oxygen therefore reducing the oxygen levels present in the oil.

Calculation of Langmuir and Freundlich isotherms

The calculation of the adsorption isotherm for the peroxide adsorption process with ampo as an adsorbent is carried out by using the Langmuir and Freundlich equations. Langmuir equation calculations are done using the following equation.

$$Ce/(x/m) = 1/ab + 1/a\ Ce$$

Meanwhile, the Freundlich equation calculation is using the following.

$$Log\ (x/m) = \log k + 1/n \log Ce$$

Ce = Peroxide concentration in the cooking oil after adsorption.

x/m = Peroxide mass adsorbed per gram of adsorbent ampo.

b = Langmuir number.

a & k = Maximum adsorption capacity (mg/gram)

The values of a and k indicate the capacity of the ampo's peroxide adsorption, the greater the Langmuir Isotherm equation value and the Freundlich Isotherm equation value means the greater the adsorption capacity. The value of 1/ab and log k are influenced by temperature therefore affects the adsorption rate. To determine the Langmuir and Fruendlich isotherm equation, the values of x/m, Ce/(x/m), log Ce/(x/m) and log Ce are calculated. From the table above, graph mapping is carried out using Excel by plotting the value of Ce/(x/m) versus Ce to get the Langmuir equation and plotting log (x/m) versus log Ce to get the Freundlich equation. Mapping results with graphs are shown in the image below.

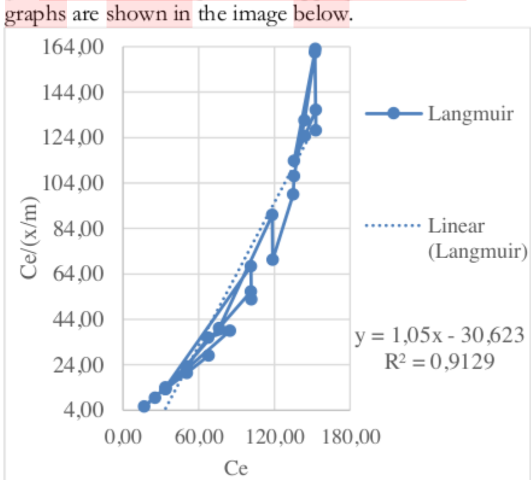

Fig. 4. Langmuir Isotherm Adsorption Equation of Ce vs Ce/(x/m)

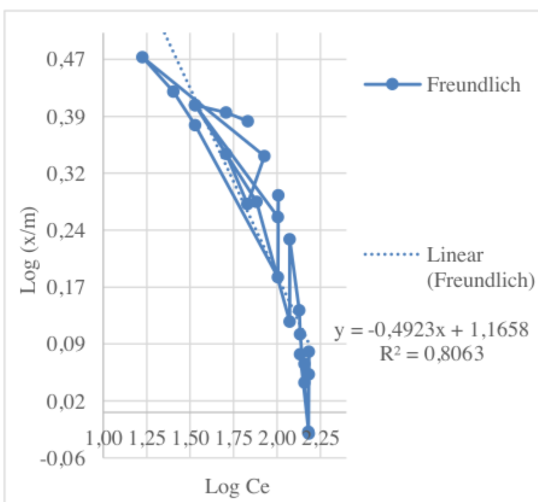

Fig. 5. Freundlich Adsorption Isotherm equation of log Ce vs log (x/m)

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The graph of the log (x/m) versus log Ce correlation produced the linear equation from Langmuir isotherm of (Figure-4) y = 1.05x - 30.623 and R^2 = 0.9129. Meanwhile, the Freundlich isotherm (Figure-5) produced a linear equation of y = -0,4923x + 1,1658 and R^2 = 0,8063. The Freundlich equation model states that there is more than one surface layer, and the sides are heterogeneous, so there is a difference in bond energies on each side. In the Langmuir equation model, adsorption occurs on each side and is homogeneous. From these statements, it shows that the Langmuir and Freundlich isotherm equations are applicable in the adsorption process of peroxide compounds in used cooking oil.

After obtaining the linear equation of the two adsorption isotherm models, they are used to find the constant values. The constant values of the equations above are shown in Table-2 below.

Table 2. Langmuir and Freundlich Isotherm Constants values

Isoterm	Constant	Value
Langmuir	a	0,9524
	b	0,0343
Freundlich	k	14,6487
	n	2,0313

To determine the maximum adsorption capacity that occurs in the adsorption process using an Ampo adsorbent, Langmuir and Freundlich isotherm equation can be used. In this study, the determination of the adsorption capacity of peroxide compounds in used cooking oil using Ampo is more suitable with the Freundlich equation. The maximum peroxide compounds adsorbed by using Ampo in used cooking oil is 14.6487 mg/gram.

4. Conclusion

Based on the results of the study it can be concluded that:

1. The best result in this study was obtained with a stirring time of 60 minutes and with an ampo adsorbent mass of 75 grams. This condition provided a peroxide value of 0,99 meq/kg, which means that there was a decrease in the peroxide value by 93%. This result is in accordance with SNI 3741-2013 with a maximum peroxide value of 10 meq/kg.
2. This study indicates that the maximum adsorption capacity using Freundlich isotherm equation is 14,6487 mg/gram.
3. The peroxide adsorption equation with Ampo has qualified the Langmuir equation with R^2 = 0,9129 and the Freundlich equation with R^2 = 0,8063

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