OPTIMIZATION TIME AND MASS OF EGGSHELL ADSORBENT IN IMPROVING WASTE COOKING OIL QUALITY

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Abstract: Oxidation and hydrolysis damage to cooking oil can occur due to repeated use of cooking oil. Adsorption by utilizing eggshells, which contain high calcium carbonate and have a natural pore structure that can improve the quality of used cooking oil. This study aims to determine the physical properties and characteristics of the thermally activated chicken eggshell adsorbent and to determine the ability of the adsorbent to absorb acid and metal cadmium. The steps carried out in this study were the manufacture of adsorbents, quality tests of the adsorbents produced, and testing of adsorbents on used cooking oil samples based on acid numbers and decreasing levels of metal cadmium in the samples. The adsorption process of used cooking oil was carried out with variations in the mass of the adsorbent, namely 5, 7, 9, and 11 grams, and stirring time for 10, 20, 40, and 60 minutes. Based on the results of the study, it was found that the optimum conditions for decreasing the acid number with the use of an adsorbent mass of 11 grams and a stirring time of 60 minutes resulted in an acid number was 0.9974 mg NaOH/g with a decrease in the acid number of 76.54%, while for cadmium metal it decreased by 100% with adsorption capacity of 2.57124 mg/g.

Keywords: *Acid Number, Eggshells, Cadmium, Waste Cooking Oil*

INTRODUCTION

Cooking oil that is used repeatedly or better known as used cooking oil is a waste vegetable oil and is not good for health because during the frying process there is oil fat damage caused by contact with air, foodstuffs, and high heat. Several indicators of oil damage due to heating can be seen from changes in color to brownish, rancid odor, an increase in the free fatty acid content of about 4.71%, the water content of 0.5%, density of 0.8912 g/mL , and an increase in metal content [1][2][3]. The number of cadmium acids and metals is one of the indicators used to determine how much damage a cooking oil has. The number of milligram bases (NaOH/KOH) of 0.1 N used to neutralize free fatty acids contained in 1 gram of oil or fat is known as the acid number [4]. One type of heavy metal that is dangerous is Cadmium (Cd) metal because this metal is at high risk to blood vessels. Cadmium affects humans in the long term and can accumulate in the body, especially in the liver and kidneys [5][6]. Used cooking oil can be used as soap, cosmetics, or biodiesel by improving the quality of the used cooking oil. One of the efforts is to reduce the levels of acids and metals in used cooking oil using activated carbon as an adsorbent through the adsorption process. The adsorption process is the process of fluid absorption of a porous solid that has advantages in its work including being simple, environmentally friendly, economical, and does not provide side effects. Some factors that affect the rate of adsorption include the type of adsorbent, the type

of adsorbate, the surface area of the adsorbent, the concentration of adsorbate, pH, and temperature [7].

At the moment eggshells are available quite a lot but have not been utilized. In an effort to reduce environmental waste while increasing the economic value of eggshells, eggshells are used as adsorbents in increasing the shell of used cooking oil. The eggshell itself contains 10,000-20,000 pores containing 94% calcium carbonate which can be used as a polar absorbent [8][9].

Previous studies have examined the use of egg shells as adsorbents both for absorbing dyestuffs, and metal, and improving the quality of used cooking oil. Susanto used eggshells as adsorbents to reduce $Cr⁶⁺$ metal levels in electroplating waste by varying the size of the adsorbent and sampling time, obtaining the best absorption at a size of 80 mesh at a time of 40 minutes of 53.001% [10]. Adsorption of Cd metal from batik industrial liquid waste samples using variations in eggshell masses of 2, 4, 6, 8, and 10 g obtained absorption efficiency reaching 90.25% at a mass of 6 g [11]. Using a composite mixture of chicken eggshell and rice husks in a ratio of 1: 3 can adsorb methyl orange by 55.90% [8]. Variations in mass, pH, and time were carried out to determine the ability of the egg shell to absorb methyl orange solution, the best condition at a mass of 11 g with a contact time of 60 minutes at pH 1 obtained an adsorption efficiency of 41.46% [12]. The use of eggshells as adsorbents with a large mass and stirring time will improve the quality of the oil so

that it can achieve oil in accordance with the SNI standard 01-3741-2013 [9]. Eggshells can lower acid and peroxide numbers by 91% and 58% [1]. Purification of glycerol using eggshells weighing ratio of 15% by acidification method in a ratio of 1:0.6 resulted in glycerol levels of 67.22% [13].

Based on the background described, this study aims to determine the best contact time and mass of eggshell adsorbent so that it can improve the quality of used cooking oil by SNI standards 01- 3741-2013. Eggshell adsorbents are characterized using Fourier Transform Infra Red (FT-IR) to determine the main functional groups that play a role in the binding of metals to used cooking oil. Measurements of metals and acid numbers in used cooking oil were detected using atomic absorption spectrophotometry and titration. The development of this method is expected to produce an adsorbent that has a good enough adsorption capacity to improve the quality of used cooking oil so that it can be used as a useful product.

RESEARCH METHODS

The materials used in this study are eggshell as the main material for making adsorbents, used cooking oil as the object of study, Nitric acid pa (Merck) as a solvent in AAS measurements, Cadmium standards (SPEX CertiPrep) as standards in AAS measurements, Sodium hydroxide pa (Merck) and Phenolthalein as reagents in the determination of acid numbers in used cooking oil, iodine, amylum, and sodium thiosulfate pa (Merck) as reagents for the determination of iodine numbers in the eggshell adsorbent.

The tools used in this study are Fourier Transform Infra Red (FT-IR) (Perkin Elmer) used for the analysis of eggshell functional groups before and after being contacted with used cooking oil, Atomic Absorption Spectrometer (Perkin Elmer) is used for the analysis of cadmium in used cooking oil, a set of titration tools are used for the analysis of acid numbers in used cooking oil, furnaces, 60 mesh sieving and a set of glass tools for the production eggshell adsorbents.

Production eggshell adsorbents

The eggshell waste was washed several times until clean and then the membrane layer in the egg shell was separated from the shell and then dried at room temperature. After drying, the eggshell was crushed and mashed until it reaches the size of 60 mesh. The eggshell of a homogeneous size was heated at a temperature of 100° C for 3 hours and a temperature of 600° C for 2 hours to be activated. The shell of the activated egg was allowed to stand for 24 hours in the desiccator. Rendement of adsorbent was calculated. The characterization of adsorbents was carried out to ensure that the adsorbent produced is by SNI 06-3730-1995 including testing water content and ash content using gravimetric methods, Iod number was measured using the iodometric method and the functional group was measured using Fourier Transform Infra-Red (FTIR). Eggshell adsorbents were ready for use.

Determination of the best mass and contact time of the eggshell adsorbent against the quality of the acid number and the content of the Metal Cd in used cooking oil

100 mL of used cooking oil was contacted with eggshell adsorbents varying in mass by 5, 7, 9, and 11 g respectively during time variations of 10, 20, 40, and 60 minutes. The used cooking oil that has been contacted was then filtered to be separated between the filtrate and also the adsorbent. To see the effect of mass and contact time on the quality of the acid number in used cooking oil, the filtrate of the separation result was analyzed using acid-base titration with NaOH solution as the titrant, the acid number can be calculated using the following equation:

acid number=
$$
\frac{40 \times V_{\text{NaOH}} \times N_{\text{NaOH}}}{W}
$$

Description:

 V_{NaOH} = Volume of NaOH used (mL) N_{NaOH} = Normality of NaOH used for titration (N) $W = weight of the sample (g)$

while to determine the effect of mass and contact time on the Cd contained in the used cooking oil, the filtrate from the filtering results was analyzed using atomic absorption spectrophotometry at a maximum wavelength of 228.8 nm. Cd can be calculated with the following equation:

$$
Cd\;Content = \frac{C}{W}xV
$$

Description:

 $C =$ Concentration of sample of analysis results with (mg/L)

 $W =$ weight of the sample (kg)

 $V =$ The volume of the measuring flask (L)

RESULT AND DISCUSSION

The purpose of this study was to determine the influence of the mass and contact time of adsorbents on the quality of used cooking oil. The use of eggshells as adsorbents is because the eggshell contains a protein matrix and inorganic crystalline material $(CaCO₃, MgCO₃, and CaPO₄)$. $CaCO₃$ was the main component of the eggshell. $CaCO₃$ is a solid substance of white color, contains a maximum of 40% Ca, insoluble in water, but the solubility increases in alkaline conditions. $CaCO₃$ was polar so it was able to bind divalent metal ions (M^{2+}) [10] so it became the basis for selecting adsorbents for improving the quality of used cooking oil. Eggshell adsorbents were made through several stages, namely cleaning, grinding and sifting, and activation.

Cleaning was carried out by washing with and removing the membrane around the eggshell to be free from impurities and increasing the amount of calcium carbonate in the eggshell. Grinding and sifting of up to 60 mesh were carried out to increase the surface area on the eggshell. The adsorbent in the form of a powder has a smaller size than the shape of a solid. Solid substances would react faster when the surface is expanded by changing them to a small size so that the surface area between the collision plane and the reagent will be larger. The chicken egg shell in this study was changed in shape to chicken eggshell powder by passing a sieve at a size of 60 mesh. The size of this 60 mesh includes a small size which causes the number of pores given to be more and the contacts that occur are getting bigger so that the efficiency of the adsorption obtained will also be greater. Powder-shaped adsorbent media results in CaCO³ adsorption of used cooking oil samples will be faster and more abundant than solids.

A simple and commonly used way to obtain CaCO₃ was to treat it at high temperatures. Combustion using high temperatures was intended to remove other components of impurity compounds present in the shell of such chicken eggs. The eggshell was activated physically, which was heated in an oven at a temperature of 100° C and a furnace at 600° C, and the results are shown in figure 1

Figure 1. Physically activated egg shells (a) with an oven at a temperature of 100°C give a white color like the original color of the egg shell, a slightly pungent smell (at the time of heating), in the form of powder, smooth and dry while (b) with a furnace at a temperature of 600° C gives a blackish-gray color, odorless, in the form of a powder, denser and drier texture

In figure 1, it could be seen that the activation results at the two temperatures show different colors, smells, and shapes. At a temperature of 100° C, it shows the physical properties of the adsorbent were white, slightly pungently smelling, and fine and dry powder. The physical properties of white adsorbents indicate the high content of $CaCO₃$ has not been decomposed into CaO. Inorganic materials such as MgCO₃ and CaPO₄ have not burned to ash and organic matter has not burned to

carbon. This causes the adsorbent at a temperature of 110^0 C to be white.

The physical activation process was a thermal treatment of adsorbents that aims to enlarge pores, namely by breaking chemical bonds or oxidizing surface molecules so that the surface area increases in size and affects the adsorption power. Thermal treatment of this adsorbent consists of two parts, namely first it occurs that most of the organic matter burns, then there is a transformation of the egg shell into CaO. Activation at temperatures below 600° C does not occur calcination process so CaO didn't form. This study, with a temperature of 600° C, showed the physical properties of adsorbents in blackish gray color, odorless, and in the form of powders where the texture was denser and drier.

The heating process at a temperature of 600- 700° C causes a calcination process in which there is a release of carbon dioxide $(CO₂)$ and the formation of calcium oxide (CaO) which consists mainly of $CaCO₃$ and a small part of CaO [14] indicated in the following reaction:

$$
CaCO_3 \rightarrow CaO + CO_2
$$

The calcination reaction occurs at a pressure of 1 atm. If the pressure was greater than 1 atm, then a reversible reaction occurs where the $CO₂$ gas formed will react again with CaO and form $CaCO₃$ with the reaction:

$$
CaO + CO2 \rightarrow CaCO3
$$

Temperature and calcination time affect the quality of the resulting product. This was because, under certain calcination conditions, these two factors could cause a carbonation process, where $CO₂$ gas was reabsorbed on the Surface of CaO to reshape $CaCO₃$. Other components were $CaPO₄$ and $MgCO₃$, with a melting point of 100° C. At a temperature of 350° C, MgCO₃ was decomposed into MgO and CO₂ with the following reaction equation:

$$
MgCO_3 \rightarrow MgO + CO_2
$$

MgCO³ was a fine white powder. MgO was a white crystalline powder with a molecular weight of 40.304 g/mol.

The physical properties of blackish-gray adsorbents indicate that there was a small amount of $O₂$ gas (not airtight) around the furnace room so that an oxidation reaction with adsorbents occurs that produces blackish-gray inorganic ash. Organic matter undergoes a process of carbonization into carbon (black). The higher the temperature, the more $CaCO₃$ will slowly disappear, and $Ca(OH)₂$ appears. This is due to the increasing temperature, the more CaCO₃ decomposes into CaO. With the presence of moisture in the atmosphere, CaO is very sensitive to moisture and can absorb (absorb) water so that a hydrolysis reaction occurs and forms calcium hydroxide $(Ca(OH)₂)$, where the reaction equation was as follows:

$$
CaO + H_2O \rightarrow Ca(OH)_2
$$

After the chicken eggshell adsorbent is heated in the oven and furnace for 2 hours, then weighed the weight of the adsorbent sample after the activation process. The event that occurs during the calcination process of chicken eggshell adsorbent is that the release of free (H_2O) and bound (OH) water occurs around a temperature of 100° C and 300° C, a release of $CO₂$ gas occurs around a temperature of 600° C and at this stage a significant weight reduction occurs. Thermal activation of physics is the most favorable activation for the activation of chicken eggshell powder. The yield produced in the production of eggshell adsorbent amounted to 93.07% of the total eggshell of 1,529.7500 g.

After the adsorbent of the eggshell was prepared, characterization was carried out including testing the moisture content and ash content using the gravimetric method, Iod number used by the iodometric method, and determining the functional groups in the egg shell using infrared spectrophotometry. The results of the adsorbent characterization of eggshells are shown in Table 1.

Table 1. The results of the assessment of moisture content, ash content, and iod number in eggshell adsorbents

ausordents		
Assessment	Research	SNI 06-3730-1995
	result	
Moisture	0.15%	Maximum 15%
content		
Ash	6.69%	Maximum 10%
content		
iod number	842.6376	Minimum
	$m\varrho/\varrho$	750 mg/g

Based on Table 1, the results of the characterization of eggshell adsorbent have met the

requirements and quality of activated charcoal testing at SNI 06-3730-1995 so that it can be used. Characterization of moisture content was used to determine the hygroscopic ability of the adsorbent of the eggshell. A large amount of water content in an adsorbent could cause the surface area to become large so that the adsorption ability of the eggshell becomes smaller [11]. In the study, the water content was obtained by 0.15%, and the value was still far below the maximum limit of 15%, so it can be concluded that egg shells can be used as adsorbents. Testing the ash content of eggshells aims to determine the content of metal oxides in activated carbon. High ash content can reduce the quality of activated carbon because the higher the ash content, the more inorganic material content contained in the material will inhibit the adsorption process. In this study, an ash content value of 6.69% was produced, the value still meets the quality requirements for testing activated charcoal, which does not exceed 10%. In the iod number, a value of 842.6376 mg / g was produced, the value met the quality requirements for activated charcoal testing, which was at least 750 mg / g. The determination of iod number was aimed at determining the adsorption ability of adsorbates by eggshell adsorbents. The high value of iod number showed that the eggshell has a large surface area so that it is able to absorb a large and good amount of adsorbate. In addition, the large absorption of activated carbon to iodine illustrates the increasing number of microporous structures that are formed and gives an idea of the magnitude of the pore diameter that can be entered by molecules whose size is not larger.

The FT-IR analysis aims to determine the functional groups that are inside the eggshell. The resulting characteristic spectra appear in the area of $4000-450$ cm⁻¹, As for the comparison between the adsorbent of chicken egg shells and $CaCO₃$ which can be seen in figure 2. A comparison of functional groups found in chicken eggshells and $CaCO₃$ can be seen in Table 2.

Figure 2. FTIR results in comparison between chicken eggshell adsorbent (left) and $CaCO₃$ (right)

Figure 2 and table 2 showed that the peaks of wavelength numbers at 875 and 876 cm^{-1} due to $C=C$ indicate the presence of $CaCO₃$ compounds [15]. While the peak at the wavelength number 646 $cm⁻¹$ was the vibration of Mg-O. The absence of an Mg-O group formed on the FTIR spectra indicated that the sample used was pure, already free from impurities Mg. Relatively low wavelength numbers at 3572 cm-1 correspond to O-H bonds corresponding to low concentrations of $Ca(OH)_2$ in the sample. The wavelength numbers at 1436, 1457, 876, and 713 cm⁻¹ correspond to three different modes of extension of the C-O bond of the $CaCO₃$ ion. The wavelength numbers at 2875 cm⁻¹ and 2925 cm-1 correspond to the C=O of the carbonate ion. The wavelength numbers in 1796 cm^{-1} were also related to the carbonate C=O bond.

The next stage was adsorption between the sample in the form of used cooking oil and eggshell adsorbent which was varied in mass and contact time, the parameters to be tested in improving the quality of used cooking oil were the acid number and metal content Cd. The results of the adsorption between the eggshell and used cooking oil could be shown by characterization from FT-IR comparing the eggshell before and after being contacted with used cooking oil shown in figure 3.

Figure 3. FTIR results of chicken eggshell adsorbent before (red) and after treatment with used cooking oil samples (blue)

A comparison of the functional groups found in chicken egg shells before and after treatment with used cooking oil samples can be seen in Table 3.

Table 3. Comparison functional groups generated FT-IR on chicken eggshell adsorbent before and after treatment with used cooking oil samples

In Table 3. There was a comparison between the functional groups in the eggshell before and after it was contacted with used cooking oil. The eggshell before it was contacted provides functional groups including Ca-O, O-H, and $CO3²$. An intense peak of eggshell particles is observed at 1436 cm⁻¹, which was strongly associated with the presence of carbonate minerals in the eggshell. The peak observed at 3405 cm-1 could be attributed to the presence of an alcoholic hydroxyl group (-OH) and an acidic hydrogen group (-OH). There were also two peaks that could be observed at 713 and 877 cm-1, which indicate the presence of calcium carbonate (CaCO3). Meanwhile, after the adsorption process of chicken eggshells with common oil, there were visible functional groups, namely the carbonyl group $(C = 0)$ in used cooking oil has a strong absorption at 1750 cm^{-1} which binds to the adsorbent of chicken egg shells, while for the C-O group, it occurs at 1165 cm-1 . Strong absorption in the area $2854-2925$ cm⁻¹ was an absorption for C-H, in the alkene group (-CH=CH-) which indicates the presence of unsaturated fatty acids, this was reinforced by absorption at 1457 cm^{-1} which is the absorption for CH2. Meanwhile, after the adsorption process of chicken eggshells with common oil, there were differences in functional groups that were visible, namely the carbonyl group $(C = 0)$ in used cooking oil has a strong absorption at 1750 cm-1 Absorption at 3406 cm^{-1} were a typical absorption for the OH group from glycerol. This mechanism occurs at a time when the adsorbate molecule was trapped inside the adsorbent. Adsorption occurs due to the presence of van der walls (intermolecular

force) forces caused by the oscillation of electron clouds from adjacent atoms or molecules, resulting in a weak attraction. Van der walls force occurs between adsorbate molecules and active surfaces in the adsorbent pores.

1. Effect of mass and contact time of eggshell adsorbent on decreasing acid number of used cooking oil

The maximum allowable acid number is 0.6 mg NaOH/g. Before processing the measured acid number level of 4.2516 mg NaOH / g. acid number levels in used cooking oil decreased with the adsorption process by varying the adsorbent mass by 5, 7, 9, and 11 g as well as variations in contact time at 10, 20, 40, and 60 minutes. The result of the decrease in the acid number can be seen in Table 4.

Table 4. The result of a decrease in the acid number (mg NaOH/g) in used cooking oil with the adsorption of egg shells varied in mass and contact time

The efficiency of decreasing the acid number through the adsorption of the eggshell varied in mass and contact time as shown in figure 4.

Figure 4.The efficiency of reducing the acid number in used cooking oil adsorbed by eggshells based on adsorbent mass and contact time.

Based on figure 4, it could be seen that the maximum amount of acid adsorbed by the adsorbent was at a mass of 11 grams and a contact time of 60 minutes, which was 76.54% with the value of the acid number being 0.9974 mg KOH/ g. This acid number has not appropriated the SNI 3741-2013 standard, so it requires even more adsorbent mass. Optimum conditions occur within 60 minutes. Too

long a contact time will reduce the effectiveness of the adsorbent to absorb because it is already in a saturated condition so desorption occurs that could raise the content that has been absorbed again. The more adsorbent mass, the higher the adsorption efficiency [16][17]. More amounts of adsorbent results in higher surface area from active groups of carboxyls, sulfates, and amines from mucopolysaccharide acid proteins in the shell of chicken eggs to bind to used cooking oil. Free fatty acids that had polar carboxyl, so could be bound with adsorbents of chicken egg shells that are polar A large number of fried foods and how often the oil is used for frying affects the deterioration of the oil, more often and more the amount of food that is fried, the more damaged the oil, is because during the heating process the oil undergoes various reactions such as chemical reactions of hydrolysis which results in the formation of free fatty acids The amount of free fatty acids in the oil is indicated by the number of acid numbers, higher the acid number, the higher the free fatty acids that cause damage to the oil [18].

Contact time was the time egg shell powder gave to adsorb using cooking oil samples. The longer the contact time between the adsorbent and the adsorbate, the more ions can be adsorped. The length of the given contact time can cause the diffusion and attachment of the adsorbed solute molecules to take place more. Based on statistical testing with two-way ANOVA it was found that the mass and time of adsorption with egg shells reduced the number of acids in used cooking oil.

2. Effect of Mass and Contact Time of Eggshell Adsorbent on Decreasing Cd Metal in Used Cooking Oil

The maximum allowable cd metal content is 0.2 mg/kg referring to (SNI) 3741:2013. In this study, the initial concentration of Cd(II) metal solution contained in the used cooking oil sample was 0.5143 mg/kg. The results of measuring cd metal content are shown in Figure 5.

Contact Time (minutes)

Figure 5. Effect of mass and contact time of eggshell adsorbent on decreasing Cd Metal in used cooking oil

Based on figure 5, it can be seen that the amount of adsorbed Cd (II) metal increases as the adsorbent mass increases and the length of the stirring time. From 10 minutes to 60 minutes which shows the amount of adsorbent mass and the length of stirring time is directly proportional to the amount of metal Cd(II) adsorbed. The results of the FTIR analysis showed that the eggshell has organic compounds with a hydroxyl group (O–H). The mechanism of ion exchange in adsorbents and metal ions Cd(II) can be shown in Figure 6.

Figure 6. Mechanism of ion exchange in adsorbents and metal ions Cd (II) in used cooking oil

This ion exchange mechanism occurs when the carboxylic groups (COOH) in amino acids are deprotonated due to the presence of hydroxide ions (OH⁻), so that the carboxylic group turns into a negatively charged (COO-) which is highly reactive to bind to Cd^{2+} [19] [20]. The absorption of Cd^{2+} metal that is maximally absorbed with adsorbents is at a mass of 11 grams and a stirring time of 60 minutes, which is 100% with a cadmium metal content value of -0.1440 mg/kg. This cadmium metal has appropriate the standards of SNI 3741- 2013, but provides negative results starting from the absorption of the adsorbent mass of 5 grams with a stirring time of 10 minutes that by using less adsorbent mass, the adsorbent of this chicken eggshell has been able to reduce the cadmium metal content in the used cooking oil sample.

CONCLUSIONS

Based on the results of the research that has been carried out, it can be concluded that the optimum result of activated carbon reduces the level of acid and cadmium metal, obtained in the addition of chicken eggshell adsorbent mass at 11 grams with a stirring time of 60 minutes, at the acid number by 0.9974 mg NaOH/g with a decrease in acid number by 76.54%, while for cadmium metal it decreased by 100%. eggshell adsorption capacity at optimum conditions of 2.57124 mg/g. The mass and timing of the adsorbent stirring of the eggshell affect the improvement of the quality of the used cooking oil.

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