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RESEARCH ARTICLE

The effect of the addition combination of chicken eggshell adsorbents (*Gallus gallus domesticus*) and siwalan fiber (*Borassus flabellifer*) on the adsorption process of used cooking oil

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OBJECTIVES Cooking oil is a daily necessity. Repeated use of oil with high heating temperatures (200-250 °C) can result in oxidation and polymerization processes in the oil. Cooking oil purification is carried out by the adsorption method, which utilizes cellulose content in coconut coir and calcium carbonate ($CaCO_3$) in eggshell to meet the National Standard of Indonesia (SNI) 3741:2013. METHODSIn this study, the adsorption process was carried out using siwalan coir adsorbents activated by 9% Zinc chloride ($ZnCl_2$) and eggshell adsorbents activated using a 600 °C furnace for 3 hours. This study was handled using a factorial 2^3 experimental design, and it was found that the adsorption spin time variable was the influential variable. RESULTS The results of this study were per SNI 3741:2013 with a Free Fatty Acid (FFA) concentration of 0.3049% and a peroxide value of 2 mekO_2/kg with a mass of adsorbent for siwalan coir and shells of 2: 10 gram, the optimum time was 69 minutes. CONCLUSIONS Analysis of the findings of the optimization sample, which has a specific gravity of 0.91 gram/cm^3, a clear yellow colour, and a moisture content of 0.083%.

KEYWORDS Adsorbent; Adsorption; Cooking Oil; Eggshell; Siwalan Coir

1. INTRODUCTION

In Indonesia, cooking oil is a very important product. The contribution of cooking oil to Indonesia's CPI (Consumer Price Index) has quite a big impact. Because cooking oil is a daily necessity (Alamsyah et al. 2017), it is estimated that the increase in cooking oil will significantly impact inflation (Nugroho and Salsabila 2022). Cooking oil content primarily comprises triglycerides derived from plant, animal, or synthetic fat (Kaltsum et al. 2016). Repeated use of oil by heating to high temperatures (200-250 °C) can result in oxidation and polymerization processes in the oil, if consumed, can lead to poisoning of the body and various diseases, including cancer, diarrhoea, digestive disorders, and accumulation of fat deposits in the veins. In addition, the disposal of used cooking oil into waterways can block river flows and disrupt aquatic ecosystems (Gusty et al. 2022).

Adsorption transfers certain components from one fluid phase (solution) to a solid adsorbent surface (Sahani and Inayah 2016). Separation occurs because the molecular weight or porosity differences cause these molecules to bond more strongly to the surface than others (Yustinah and Hartini 2011). Natural cellulose adsorbents can be used as a clarifier in oil separation, particularly for used cooking oil. Adsorption of used cooking oil to reduce acid numbers and peroxide levels to make the oil reusable (Julius Fernando Pakpahan et al. 2013). The composition of siwalan fibre is 89.2% cellulose, 5.4% air, 3.1% carbohydrates, and 2.3% water. Siwalan coir can make activated carbon, which can absorb dyes in used cooking oil due to its high cellulose content. According to Heriono and Rusmini (2015), siwalan coir is activated by the dehydrating chemical $ZnCl_2$, causing the carbon particles to enlarge and produce large pores, which are then used for colour refining in the batik wax textile industry. Meanwhile, because it contains 94% $CaCO_3$ and a trace of CaO, the shell of a chicken egg heated to a high temperature (600 °C) can be used as an adsorbent. Because $CaCO_3$ is a polar molecule and CaO has a hexagonal structure, eggshells can be used as a polar adsorbent (Fitriyana and Safitri 2015).

This study aims to determine the effectiveness of adding eggshell and siwalan coir adsorbents to reduce FFA (Free Fatty Acid) levels and peroxide value in used cooking oil so that consumption of cooking oil every day does not cause diarrhoea, fat deposition in blood vessels (arterosclerosis), cancer, and decreased digestibility fat (Ganesan et al. 2019; Idris

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TABLE 1. Cooking oil quality standards (Badan Standardisasi Nasional 2013).

No	Test Criteria	unit	Condition
1.	circumstances		
1.1	smell	-	normal
1.2	Colour	-	normal
2.	Moisture content and volatile matter	%(b/b)	maximum 0.15
3.	Acid number	Mg NaOH/g	maximum 0.6
4.	Peroxide number	Mek O_2/kg	maximum 10

et al. 2018). Moreover, the results of this study will get cooking oil that by Table 1 for cooking oil quality standards SNI 3741-2013. The process to be carried out is adsorption on used cooking oil using an eggshell adsorbent activated at 600 °C (Fitriyana and Safitri 2015). The addition of siwalan coir adsorbent, which has a high cellulose content, can be used to reduce dyes and acid numbers in used cooking oil to be a solution in research (Fitriyana and Safitri 2015), which still has a turbid colour in the adsorption oil and a decrease in acid number which is not optimal (Luthfian Ramadhan Silalahi et al. 2017).

2. RESEARCH METHODOLOGY

2.1 Materials and Tools

The equipment used includes a furnace; oven; burette; desiccator; sieve; measuring cup; hotplate; beaker glass; magnetic stirrer; filter paper; analytical balance; aluminium foil; Oswald viscometer; pycnometer; crucible porcelain. The materials needed are: aquadest; egg shells; siwalan choir; 9% $ZnCl_2$; used cooking oil five times for frying at a temperature of 250 °C (Febrianto et al. 2020); Sodium hydroxide granular crystalline solid which is diluted to a 0.05 N solution; Sodium thiosulfate anhydrous | Merck 1.06512 with a concentration of 0.1 N; Chloro-form for analysis EMSURE ACS, ISO, Reag. Ph Eur; Technical acetic acid 98%; Potassium iodide for analysis | Merck 1.05043; Starch soluble | Merck 1.01252.

2.2 Procedures

2.2.1 Synthesis of adsorbent

In this research, the adsorption process was carried out using siwalan coir adsorbents and egg shells. The siwalan coir adsorbents themselves were made starting by removing the water content by drying in the sun and followed by baking at 200 °C for 2 hours and grinding, then activated (50 grams of siwalan carbon: 500 mL $ZnCl_2$ 9%) for 16 hours after that carbonized siwalan coir at 105 °C for 2 hours. Meanwhile, eggshell activation was carried out using a furnace at 600 °C for 3 hours and then pulverized into powder to manufacture eggshell adsorbents.

2.2.2 Adsorption process

This study's adsorption and analysis process used a factorial design, as shown in Table 2, using 3 test variables two levels = 2^3 with 8 grams and 10 grams of eggshell adsorbents, 1.5 and 2 grams of siwalan coir adsorbents. From these variables, an adsorption process was carried out by mixing the two adsorbents in 100 mL of used cooking oil, and then adsorption was carried out with a stirring time of 60 and 75 minutes.

2.2.3 Analysis techniques

The analysis carried out in this study was the acid number or free fatty acid (FFA) to see the amount of free fatty acids in the oil (Parida Hutapea et al. 2021), a peroxide value test used to determine how much damage has occurred to an oil or grease (Yulia et al. 2017), test water content, colour, density, and viscosity concerning the 3741-2013 test standard.

TABLE 2. Data analysis.

Run	Dependent Variable			Interaction			
	m_1	m_2	w	m_1w	m_2w	m_1m_2	m_1m_2w
1.	-	-	-	+	+	+	-
2.	+	-	-	-	+	-	+
3.	-	+	-	+	-	-	+
4.	+	+	-	-	-	+	-
5.	-	-	+	-	-	+	+
6.	+	-	+	+	-	-	-
7.	-	+	+	-	+	-	-
8.	+	+	+	+	+	+	+

Siwalan coir adsorbent weight (gram) (m_1), where:
- = 1.5 gram
+ = 2 gram
Chicken eggshell adsorbent weight (gram) (m_2), where:
- = 8 gram
+ = 10 gram
Adsorption play time (minute) (w), where:
- = 60 minute
+ = 75 minute

(a) (b)

FIGURE 1. (a) Siwalan coir adsorbent (b) eggshell adsorbent.

FFA analysis was conducted by weighing 10 grams of sample dissolved in 50 mL of warm ethanol and adding phenolphthalein, then titrating with 0.05 N Sodium Hydroxide until pink. Peroxide number analysis by homogenizing 5 grams of a sample with 50 mL of the acetic acid solution, adding 0.5 mL of saturated potassium iodide and 30 mL of distilled water, then titrating with 0.1 N $Na_2S_2O_3$ solution until the yellow colour almost disappears.

Analysis of the water content by heating the porcelain crucible and the lid in the oven at (130 ± 1) °C for 30 minutes and then letting it stand in the desiccator for 20 minutes after that, weighing (W_0), inserting a 5-gram sample (W_1), repeating the baking and weighing stages until a constant weight (W_2) is obtained. As for the colour and odour tests, an analysis was carried out using three panellists. Density testing using a pycnometer and viscosity using an Ostwald viscometer.

3. RESULTS AND DISCUSSION

3.1 Adsorption of used cooking oil with eggshell and siwalan coir adsorbents

This research on used cooking oil adsorption was carried out to improve the cooking oil quality by adsorbing eggshell and siwalan coir adsorbents. This research was processed using a level 2^3 factorial design experiment with different adsorption conditions, this experimental design was carried out to determine the effects on the process variables used, and the optimum conditions obtained were more precise because they included interaction factors. In this study, three process variables were used, namely the weight of the siwalan coir adsorbent (m_1), the weight of the chicken eggshell adsorbent (m_2), and the adsorption spin time (w).

In Table 3, run 6 (2 grams siwalan choir adsorbent, 8 grams eggshell adsorbent, with 75 minute adsorption play time) and 7 (1.5 grams siwalan choir adsorbent, 10 grams eggshell adsorbent, with 75 minute adsorption play time) still have levels of FFA and peroxide number and high water content and not by SNI 3741-2013. So with an adsorption time of 75 minutes, it is not good to use because the adsorbent particles which have absorbed dirt will break again and cause turbidity; this also happened in the research of Fitriyana and Safitri (2015). A good adsorption oil must comply with SNI 3741:2013 with a maximum FFA content of 0.6% and a maximum peroxide value of 10 mek O_2/kg. The results of optimal process variables with good oil yields can be calculated using the main effect and interaction methods on the resulting FFA levels and peroxide numbers; this can be seen in Table 4, Figure 2, and Figure 3.

The variables that have the most influence on the adsorption process of used cooking oil with eggshell and siwalan coir adsorbents in the analysis of FFA and peroxide value can be seen in Table 4, the adsorption spin time with the total main effect and interaction, respectively 0.14025 and 2 with the total variable affect 92.856%.

The relationship between the Normal Probability Plot and the main effect of FFA levels and peroxide numbers shown in Figures 2 and Figure 3 is obtained by regression se-

TABLE 3. Results of adsorption of used cooking oil with eggshell and siwalan fiber adsorbents with variation of operating conditions.

Run	FFA(%)	Peroxide Number (mekO_2/kg)	Colour	Water Content (%)	Viscosity	Density
No. Adsorp	0.91646	15	brownish	0.191	1.116	0.91646
1	0.91192	4	normal	0.077	1.188	0.91192
2	0.9059	5	normal	0.097	1.28	0.9059
3	0.91086	3	normal	0.068	1.224	0.91086
4	0.91102	6	normal	0.116	1.406	0.91102
5	0.91057	3	normal	0.078	1.224	0.91057
6	0.91197	8	normal	0.155	1.26	0.91197
7	0.91022	9	normal	0.116	1.296	0.91022
8	0.90571	6	normal	0.174	1.296	0.90571

TABLE 4. Calculation of main effect and interaction on %FFA and peroxide numbers.

Effect	Total	%influence	Total	%influence
I_1, m_1	0.1122	78.571	1.5	78.571
I_2, m_2	0.02805	64.286	1	50
I_3, w	0.14025	92.857	2	92.857
I_{13}, m_1w	-5.55112E-17	35.714	-0.5	35.714
I_{23}, m_2w	0.02805	50	1	64.286
$I1_{12}, m_1m_2$	-0.0561	21.429	-1.5	21.429
$I1_{123}, m_1m_2w$	-0.1122	7.143	-2.5	7.143

quentially, namely 96.15% and 93.43%, which means that the total model variation can be represented by the regression equation which shows the adsorption spin time. The equation that shows the correlation between the FFA levels and the research process variables is y=0.0028x - 0.1202. Meanwhile, the equation showing the correlation between the peroxide value and the research process variable is y=17.797x + 47.458.

3.2 Optimizing the adsorption of used cooking oil with eggshell and siwalan coir adsorbents

From the analysis of variance that has been carried out, it can be seen that the process variable that influences the research process of used cooking oil adsorption with eggshell and siwalan coir adsorbents is the adsorption spin time, so for this optimization process, the variables m_1 (mass of siwalan coir) and m_2 (eggshell mass) becomes a fixed variable and variable w (adsorption spin time) becomes a changing variable. The optimization results that have been carried out can be seen in Table 5.

Table 5 shows that the most optimal adsorption cycle time is 63 minutes to 69 minutes; this is because the longer the adsorption cycle time, the peroxide number and FFA content also decrease, which shows the results obtained according to SNI 3741: 2013, which can be seen in the graph Figure 4. However, at 75 minutes, there was a decrease in absorption because the absorption capacity of the adsorbent was maximized (Susanti and Mawardi 2020). Thus, high concentrations lower absorption efficiency. As a result, the amount of adsorbate in the solution is not proportional to the number of adsorbent particles available, causing saturation and reducing the absorption efficiency (Rengga et al. 2021).

Based on the FFA content and peroxide number analyzed, as presented in Table 5 and the graph in Figure 4, it can be seen that the adsorption spin time (w) of 69 minutes is the best result with an FFA content of 0.3049% and a peroxide value of 2 mekO_2/kg with siwalan coir adsorbent mass 2 grams and the eggshell adsorbent mass is 10 grams. This optimal variable indicates that the amount of mass in the adsorbent can reduce the free fatty acids present in the oil. The oil's presence of free fatty acids and high peroxide value is undesirable as it produces an unpleasant taste and odour. The quality of the oil can be determined by the amount of free fatty acids it contains; the greater the free fatty acid value, the lower the quality and peroxide value (Febrianto et al. 2020; Sahani and Inayah 2016).

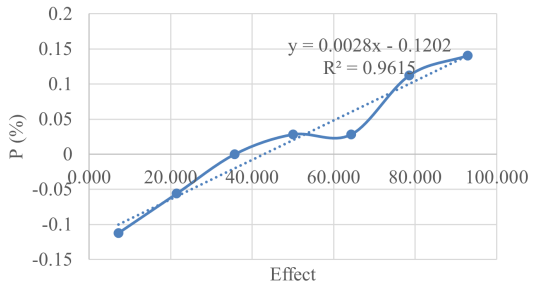


FIGURE 2. Normal Probability Plot (P Vs Effect) FFA Levels of Adsorption Used Cooking Oil for Factorial Design 2^3.

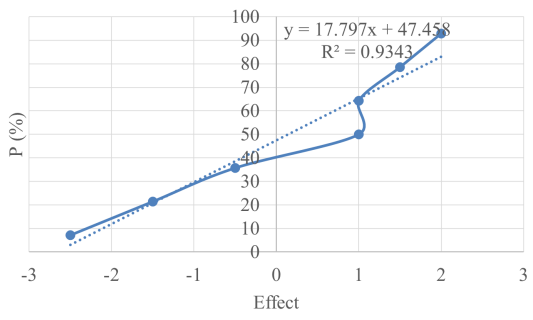


FIGURE 3. Normal Probability Plot (P Vs Effect) Number of Peroxide of Adsorption Used Cooking Oil for Factorial Design 2^3.

3.3 Analysis of the adsorption results of used cooking Oil with eggshell and siwalan fiber adsorbents

3.3.1 Test Color, viscosity, and density

The colour test of used cooking oil after adsorption was carried out by three panellists who could see colours and distinguish colours clearly (Tarigan and Simatupang 2013), which resulted in the colour before being adsorbed still looked brownish, and after being adsorbed, it was more translucent yellow so that the colour of the adsorbed results has a category with a normal description according to SNI 3741: 2013.

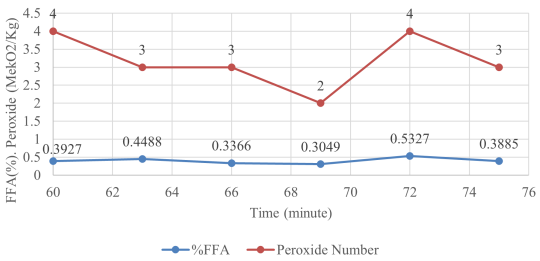
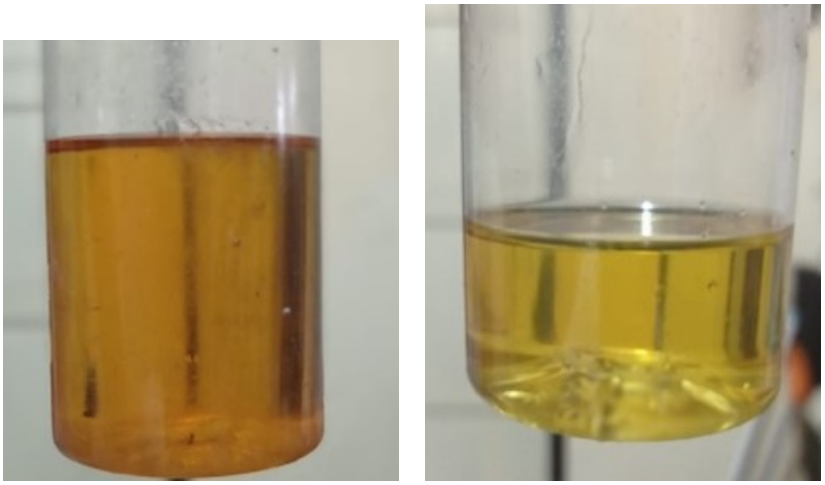


FIGURE 4. Graph of Optimization Time of Used Cooking Oil Adsorption on %FFA and Peroxide Numbers.

TABLE 5. FFA content and peroxide number adsorption of used cooking oil using siwalan coir adsorbents and eggshells in the optimization process.

Adsorption Cycle Time (minute)	Siwalan Coir Adsorbent Mass (gram)	Eggshell Adsorbent Mass (gram)	FFA (%)	Peroxide Number ($mekO_2/kg$)
60	2	10	0.3927	4
63			0.4488	3
66			0.3366	3
69			0.3049	2
72			0.5327	4
75			0.3885	3



(a) (b)

FIGURE 5. (a) Used cooking oil before adsorption (b) used cooking oil after adsorption.

Table 3 shows that used cooking oil without adsorption has a lower viscosity of 1.116 cSt and after adsorption is carried out on run 1 to run eight, respectively, namely 1.188 cSt; 1.28 cSt; 1.224 cSt; 1.406 cSt; 1.224 cSt; 1.26 cSt; 1.296 cSt; and 1.296 cSt; Cooking oil that is still new or compared to used cooking oil, high-quality cooking oil has a greater level of viscosity. As a result of heating during frying, the density of the oil decreases. The oil temperature will also rise as the batter is cooked (Islami 2020). Heating raises the temperature, which weakens the bonds holding the oil molecules together, causing the viscosity to drop (Misrawati et al. 2015). The adsorption process using siwalan coir and eggshell adsorbents can improve the quality of cooking

The density test results in Table 3 show that the results are similar to the density of used cooking oil before adsorption. However, the results already comply with SNI 3741: 2013, which states that the density of cooking oil is around 0.91 grams/cm^3.

3.3.2 Moisture Content

From Table 6, data analysis of water content in this study obtained a result of 0.083%. This result meets the SNI standard, namely a maximum of 0.15%. From these results, the analysis results on the optimal variable are per SNI 3741:2013. The gravimetric technique determines the water content in cooking oil samples (Hutapea 2014). The idea behind the gravimetric approach is to compare the actual weight of the sample before and after heating to determine how much water is present in the sample. The water physically bound to the cooking oil sample under study is removed from the oil by heating it at 130 °C. To get accurate results, the water content check in this study was also carried out three times (triple) (Ulfindrayani and A'yuni 2018)).

TABLE 6. Analysis of moisture content result of used cooking oil adsorption process using siwalan coir and eggshell adsorbents.

Test	SNI 3741:2013	Adsorption Moisture Content
Moisture Content (% b/b)	Maximum 0.15	0.083

4. CONCLUSION

This research on used cooking oil adsorption aims to improve the cooking oil quality by adsorbing eggshell and siwalan coir adsorbents. The procedure and analysis technique used factorial design level 2^3 experimental design. The most influential process variable was obtained by analyzing the quicker method by looking at the number of main effects and interactions with FFA levels and peroxide number; it was found that the adsorption spin time was the variable that had the most influence. Optimization of the adsorption cycle time (w) of 69 minutes was the best result, with an FFA content of 0.3049% and a peroxide value of 2 $mekO_2/kg$ with a mass of siwalan coir and shell adsorbents of 2: 10 grams. Analysis of the optimization sample results according to SNI 3741: 2013 with a density of 0.91 gram/cm^3, clear yellow colour, and 0.083% water content.

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