HYDROGEN
JURNAL KEPENDIDIKAN KIMIA
https://e-journal.undikma.ac.id/index.php/hydrogen/index

February 2025. 13(1)
e-ISSN: 2656-3061
p-ISSN: 2338-6487
pp. 197-208

Application of Silica Adsorbent Rice Husk Ash-Activated Carbon in Refining Waste Cooking Oil

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Article History
Received: 18-01-2025
Revised: 27-02-2025
Published: 06-03-2025

Keywords: activated carbon; adsorbent; adsorption; silica; waste cooking oil.

Abstract
A study has been conducted on "Application of Silica Adsorbent Rice Husk Ash-Activated Carbon in Refining Waste Cooking Oil". This study aims to describe the comparison of silica-activated carbon composite in refining waste cooking oil and describe the optimum time of silica-activated carbon composite in refining waste cooking oil. The sample in this study was waste cooking oil from fried geprek chicken which was fried 5 times. While the adsorbent used was silica composite from rice husk ash and activated carbon. The method used in this study was adsorption. The stages in this study consisted of four stages, namely silica extraction from rice husk ash, making silica-activated carbon adsorbent composite, silica-activated carbon composite ratio (30:70; 40:60; 50:50; 60:40; and 70:30), and time variations, namely (30, 45, 60, 90) minutes. Silica extraction from rice husk ash using NaOH 12% solution obtained a silica content of 88%. The results of the study of silica-activated carbon composites in the purification of used cooking oil showed that the optimum silica-activated carbon composite adsorbent at a ratio of 60:40 grams and an optimum contact time of 30 minutes obtained a water content of 0.13%, free fatty acids of 0.23% and a peroxide number of 10.92 meq O_2/Kg. The results of organoleptic tests with color, odor, and turbidity parameters obtained bright yellow results, less fried chicken odor, and not cloudy.

How to Cite: Gala, D., Rahmanpiu, R., & Mulyana, W. (2025). Application of Silica Adsorbent Rice Husk Ash-Activated Carbon in Refining Waste Cooking Oil. Hydrogen: Jurnal Kependidikan Kimia, 13(1), 197-208. doi:https://doi.org/10.33394/hjkk.v13i1.14802

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INTRODUCTION

Based on data from the Central Statistics Agency in 2023, it is known that the area of Indonesia's rice fields reached 10.20 million hectares with dry grain production of 53.63 million tons. Dry grain processing produces husks and has been used as biomass in feed production (Asfar et al., 2023), scouring ash (Amir & Basry, 2019), a mixture to increase the strength of bricks (Julliano et al., 2024) and raw materials for making bokashi fertilizer (Meilina et al., 2022). Rice husk contains 31,37- 49,92% fiber, 34,34-43,80% cellulose, and 21,40-46,97% lignin (Safrinda et al., 2022); silica (15-20%) (Ariyetti et al., 2024); water content 9,02%, crude protein 3,03%, fat 1,18%, crude fiber 35,68%, ash 17,17%; dan carbohydrates 33,71% (Meriatna et al., 2015). High silica content, rice husk ash is widely used as an adsorbent. In addition to being easy to obtain and cheap, silica adsorbents are also smoother and more reactive (Agung et al., 2013). Silica is also inert, has good adsorption and ion exchange capabilities, and is easily modified with certain chemical compounds to improve its performance (Hardyanti et al., 2017).

Silica as an adsorbent has low effectiveness and selectivity on the surface in interaction (Astuti et al., 2019). Oxygen on the active site of silica is a species that is relatively small in size and is a hard base so that the tendency to interact with relatively large atoms and soft acids becomes weak (Hastuti et al., 2021). This condition allows modification of the silica

surface. Modification of silica through covalent bonds with the sol-gel process (Putra et al., 2022). Therefore, one of the adsorbents that can be used for silica surface modification is activated carbon. The ability to absorb substances in liquid, solid or gaseous forms of activated carbon is widely used as an adsorbent or mixture of adsorbents such as silica. Activated carbon absorbs water molecules in the air (Octarya & Fernando, 2016).

The amorphous structure and surface area between 300-3500 m^2/gram with high porosity of activated carbon are effectively used as adsorbents (Lubis et al., 2020). Silica which has a large surface area with active siloxane (Si-O-Si) and silanol (-Si-OH) groups and activated carbon with high porosity can be modified in the form of composites (Noviasari, 2017). Composites are microscopic combination materials of two or more different materials, with the aim of obtaining certain properties and characteristics that are better than the properties of each component (Dyana & Triwikantoro, 2017).

Waste cooking oil is oil that has been used repeatedly so that its quality decreases. This oil is often used to increase livestock appetite. Giving waste cooking oil up to 2% can increase the weight gain of quail by 29.75 grams (Oktaviana et al., 2023). However, consuming eggs or meat containing free fatty acids from used cooking oil will contribute to increasing cholesterol levels. In addition, consuming oxidized fat from trans fats is also harmful to human health. Excessive reactive species of oxygen and nitrogen can cause oxidative damage to tissues and organs. Oxidative stress has been considered a concurrent pathological mechanism, and contributes to the initiation and development of liver damage (Cichoż-lach & Michalak, 2014) and human bondy cell tissue (Ardhany & Lamsiyah, 2018); causing damage to the nervous system, fat deposit disorders, cancer, and other health problems (Azizanie et al., 2023). Giving waste cooking oil as an additive in animal feed will have a carry over effect that has the potential to enter the human body (Sumantri et al., 2012). In addition, if waste cooking oil is disposed of into the environment, it will damage the soil structure because it inhibits the movement of water in the soil pores (Al Qory et al., 2021).

Because waste cooking oil if consumed will cause health problems, if disposed of will damage the environment, and is dangerous if given to livestock, it is necessary to purify waste cooking oil to meet the quality of the Indonesian National Standard, namely a maximum peroxide number of 10 meq O_2/kg, a maximum water content of 0.1% and a maximum free fatty acid content of 0.3%. Research on the purification of waste cooking oil has been conducted by Istiningrum et al. (2017) using rice husk ash to purify waste cooking oil by reducing free fatty acids. Reubun et al. (2024) using silica can reduce the level of free fatty acids in waste cooking oil by 0.59%. Irawan et al. (2013) using a mixture of coconut fiber and rice husks can reduce the level of free fatty acids by the lowest by 0,2944%.

Research on silica-activated carbon composites is generally only on metal adsorption. Research on silica-activated carbon composites has been conducted by Alshammari et al. (2021) for the removal of heavy metals from groundwater. Lilli et al. (2024) used rice husk-based carbon-silica composites for the adsorption of sodium diclofenac and carbamazepine from aqueous solutions. However, research on silica-activated carbon composites in the purification of waste cooking oil is less explored and is still limited. Therefore, the purpose of this study is to describe the comparison of silica-activated carbon composites and the optimum time of silica-activated carbon composites in the purification of waste cooking oil.

METHOD

Tools and Materials

The tools used are 200 mesh sieve, measuring flask, scale pipette, filler, thermometer, beaker, hot plate, Erlenmeyer flask, glass funnel, filter paper, stirring rod, magnetic stirrer, oven,

mortar, analytical balance, spatula, dropper pipette, furnace, beaker, stand, clamp, burette, porcelain cup, and desiccator. While the materials used are waste cooking oil, rice husk ash, activated carbon, 12% sodium hydroxide, 1 M sulfuric acid, aquadest, 95% ethanol, 1% pp indicator, 0.01 N sodium hydroxide, glacial acetic acid, 1% starch, 0.01 N sodium thiosulfate.

Silica Extraction

A total of 40 grams of sifted rice husk ash was put into a 500 mL beaker and 240 mL of 12% NaOH solution was added. he mixture was heated at 85^{o}C for 90 minutes while stirring using a magnetic stirrer. The mixture was filtered and the filtrate was neutralized with 1 M H_2SO_4 until a gel was formed. The gel formed was left for 18 hours. The gel formed was separated from the liquid by filtering, while being washed with 1000 mL of hot aquadest. The residue on the filter paper was dried in an oven at 105^{o}C for 5 hours. The results obtained were in the form of white powdered silica, cooled and weighed (Harimu et al., 2019). The silica content obtained was calculated using the following formula.

$$\text{Kadar Silika} = \frac{\text{berat silika yang diperoleh}}{\text{berat abu sekam padi}} \times 100\%$$

Silica-Activated Carbon Composite

Silica-activated carbon composites were made by mixing silica and activated carbon each with a mass of 2.5 grams (50 : 50). Ethanol was added as much as 40 mL, stirred, then dried in an oven at a temperature of 90^{o}C for 24 hours. The mixture was calcined for 4 hours at a temperature of 200^{o}C. Other composites were made with a ratio of (30:70), (40:60), (60:40), and (70:30) (Kesumaningrum et al., 2011).

Refining of Waste Cooking Oil

Determination of Adsorbent Composite

The mass ratio of silica-activated carbon (30:70), (40:60), (50:50) (60:40), and (70:30), each was put into a beaker containing 50 mL of waste cooking oil. The mixture was stirred using a magnetic stirrer for 30 minutes. The mixture was filtered, then the filtrate in the form of oil was tested for organoleptic. The test results were compared with the organoleptic of the Indonesian National Standard (SNI) (Irawan et al., 2013)

Determination of Adsorption Time

The silica-activated carbon composite adsorbent with organoleptic test results that meet SNI standards was applied to determine the contact time. The determination of the optimum contact time of the silica-activated carbon composite follows the procedure developed by (Hadiah & Meliasari, 2017). Each 1 gram of silica-activated carbon composite adsorbent was put into a beaker, then 50 mL of waste cooking oil was added, stirred with a magnetic stirrer for 30 minutes, 45 minutes, 60 minutes, and 90 minutes. The mixture was filtered and tested for organoleptic. The results were compared with SNI organoleptic (Irawan et al., 2013).

Waste Cooking Oil Quality Test

Water Content

A clean porcelain cup is placed in an oven at 105^{o}C for 30 minutes, cooled in a desiccator for 15 minutes, then weighed. This process is carried out 3 times until the weight of the cup is constant. Next, 2 grams of waste cooking oil is weighed in a cup, placed in an oven at 105^{o}C for 3 hours. The cup containing waste cooking oil is cooled in a desiccator for 15 minutes, then weighed. The cup containing waste cooking oil is reheated, cooled in a desiccator 3

times until a constant weight is obtained (the difference in consecutive weighing is less than 0.2 mg). Determination of water content is carried out by evaporating 3 samples of waste cooking oil (SNI, 2019).

$$\text{Water Content} = \frac{m_2 - m_3}{m_2 - m_1} \times 100\%$$

Description:
m_1 = empty cup weight (g)
m_2 = empty cup weight + sample (g)
m_3 = weight of cup + sample after heating (g)

Free Fatty Acid

Into three 250 mL Erlenmeyer flasks of known weight, each was put 1 gram of waste cooking oil. Add 10 mL of 95% ethanol, stir and add 2-3 drops of phenolphthalein (pp) indicator. Then titrate with 0.01 N NaOH until the solution is pink and does not disappear for 30 seconds. The volume of NaOH used is recorded and calculate the free fatty acid content (Untari et al., 2020).

$$\%\text{FFA} = \frac{\text{BM fatty acid} \times \text{mL NaOH} \times \text{N NaOH}}{1000 \times \text{W}} \times 100\%$$

Description:
%FFA = Free Fatty Acid Content (%)
BM Fatty Acids = BM Palmitic Acid (256)
N NaOH = Normality of NaOH solution
mL NaOH = Volume of NaOH titrant
W = sample weight (g)

Peroxide Value

Into three 250 mL Erlenmeyer flasks with lids, each was put 5 grams of waste cooking oil, added 12 mL of chloroform and 18 mL of glacial acetic acid. The mixture was homogenized by shaking the Erlenmeyer flask slowly, then added 0.5 mL of saturated potassium iodide solution. The resulting solution was left in a dark room for 30 minutes, then added 30 mL of aquadest and added 0.5 mL of 1% starch (dark blue color formed) and immediately titrated with 0.01 N sodium thiosulfate until the blue solution disappeared (SNI, 2019).

$$\text{Peroxide Value} = \frac{(V_0 - V_1) \times \text{N} \times 1000}{\text{W}}$$

Description:
V_0 = Sample titration volume
V_1 = Blank titration volume
N = Normality of $Na_2S_2O_3$ solution
W = Weight of oil sample

Organoleptic Properties Test

Organoleptic testing was carried out using a scoring test method (color, odor, and turbidity) developed by (Nusa & Sipahutar, 2018). A total of 42 mL of waste cooking oil before and after the application of silica-activated carbon composite adsorbent was placed into a beaker container. The panelists were then given a questionnaire containing statements about the measurement scale of color, odor and turbidity of the oil. The questionnaire contained a hedonic scale and a numeric scale.

Table 1. Color Parameter Scale

No.	Hedonic Scale	Numeric Scale
1.	Yellow	4
2.	Reddish yellow	3
3.	Brownish yellow	2
4.	Blackhish yellow	1

Table 2. Odor Parameter Scale

No.	Hedonic Scale	Numeric Scale
1.	No smell of fried chicken	4
2.	Less smell of fried chicken	3
3.	Smells like fried chicken	2
4.	Smells very much of fried chicken	1

Table 3. Turbidity Parameter Scale

No.	Hedonic Scale	Numeric Scale
1.	Very not cloudy	4
2.	Not cloudy	3
3.	Cloudy	2
4.	Very cloudy	1

RESULTS AND DISCUSSION

Silica and Activated Carbon Composite

Silica in this study was obtained from the extraction of rice husk ash using the alkali method, where the rice husk ash was reacted with 12% NaOH solution. After going through the heating, neutralization, and filtration processes, silica was obtained as much as 88%. The silica content obtained was not different from that obtained by Harimu et al., (2019) which was 86,90%-97,30%. Meanwhile, the activated carbon used is activated carbon that is already available in the laboratory. The application of silica as an adsorbent in this study refers to the research procedure of Kesumaningrum et al. (2011) namely the modification of silica-activated carbon adsorbents (composite). Silanol and siloxane groups in silica and the surface area and pores of activated carbon in absorbing water, solids are the basis for modifying each adsorbent into a composite. The silica-activated carbon composite is carried out through a calcination process at a temperature of 200^{o}C, so that the composite structure becomes more regular (Hindryawati et al., 2014).

This comparison of silica-activated carbon composites is intended to determine the good adsorption capacity between silica and activated carbon. The comparison of silica-activated carbon composites used is (30:70), (40:60), (50:50), (60:40), and (70:30). The waste cooking oil used is oil from frying geprek chicken which has been used for five frying times. The volume of waste cooking oil used is 50 mL with a contact time of 30 minutes. The comparison of silica-activated carbon composite adsorbents in the purification of waste cooking oil is shown in Table 4.

Based on Table 4, it shows that the waste cooking oil from the adsorption of the silica-activated carbon composite is optimum at a composite ratio (60:40) of 84%. Based on the results of the organoleptic test, at this ratio the oil produced is bright yellow, has less fried chicken odor, and is not cloudy. The oil produced at this ratio is similar to the new oil, namely it has a bright yellow, odorless, and is very not cloudy. Silica-activated carbon composite (60:40) shows that the mass of silica in this composite is greater than the mass of

activated carbon. With a larger silica mass, it allows impurities in used cooking oil to be selectively bound to the adsorbent. This is because silica has active sites in the form of silanol (Si-OH) and siloxane (Si-O-Si) which can absorb water, free fatty acids, and peroxides.

Table 4. Silica and Activated Carbon Composite

No	Adsorbent (Silika: Activated Carbon)	Waste Cooking Oil Volume (mL/%)	Organoleptic								
			Color			Smell			Turbidity		
			BA	AA	C	BA	AA	C	BA	AA	C
1	(30:70)	40 (80%)		Yellow			Less smell of fried chicken			Not cloudy	
2	(40:60)	41 (82%)		Yellow			Smells like fried chicken			Cloudy	
3	(50:50)	39 (78%)	Brownlish Yellow	Yellow	Bright Yellow	Smells very much of fried chicken	Smells very much of fried chicken	Not Smell	Very cloudy	Cloudy	Very not cloudy
4	(60:40)	42 (84%)		Bright Yellow			Less smell of fried chicken			Not cloudy	
5	(70:30)	39 (78%)		Yellow			Smells like fried chicken			Cloudy	

Description:
BA = Before adsorption
AA = After adsorption
C = Control

This is supported by research Laurentius et al. (2020) stated that the greater the amount of silica (SiO_2), the better it will be in attracting free fatty acids, organic substances and other polar substances, namely peroxide. Peroxide compounds will react with silica so that peroxide can be adsorbed to form hydrogen bonds on the silica surface. In contrast, activated carbon in the silica-activated carbon composite (60:40) has large pores and is non-polar, so that non-polar impurities will be bound to activated carbon. This is in line the statement of Novitriani & Intarsih (2015) that the large pores and non-polar surfaces of activated carbon function to absorb non-polar contaminants that pass through it. So that with the ratio of silica-activated carbon composite (60:40) it can absorb water content, free fatty acids and peroxides so that it can improve the color quality of the oil by absorbing colloidal suspensions.

Adsorption Time

The contact time of the silica and activated carbon composite adsorbent (60:40) is intended to determine the adsorption capacity of the silica-activated carbon composite in the purification of waste cooking oil. The contact time carried out in this variation is 30, 45, 60, and 90 minutes. The mass of the silica-activated carbon composite used is 1 gram with a volume of waste cooking oil of 50 mL. The variation of the contact time of silica and activated carbon in the purification of waste cooking oil is shown in Table 5.

Table 5. Contact Time of Silica-Activated Carbon Adsorbent (60:40) in Oil Refining

No	Contact Time (minutes)	Waste Cooking Oil Volume (mL/%)	Organoleptic								
			Color			Smell			Turbidity		
			BA	AA	C	BA	AA	C	BA	AA	C
1	30	42 (84%)	Brownish Yellow	Bright yellow	Bright yellow	Smells very much of fried chicken	Less smell of fried chicken	Not Smell	Very cloudy	Not cloudy	Very not cloudy
2	45	41 (82%)		Yellow			Less smell of fried chicken			Cloudy	
3	60	43 (86%)		Yellow			Smells like fried chicken			Cloudy	
4	90	41 (82%)		Yellow			Smells very much of fried chicken			Cloudy	

Description:
BA = Before adsorption
AA = After adsorption
C = Control

Based on Table 5, it can be seen that the optimum time for silica-activated carbon adsorbent (60:40) is 30 minutes. At this contact time, oil with bright yellow color quality, less fried chicken smell and not cloudy is obtained. The oil produced in this comparison has organoleptic similarities with new oil, namely bright yellow, odorless, and very not cloudy. Therefore, time has an effect on the efficiency of increasing color clarity, process time and efficiency of all oil parameters are directly proportional to the opportunity or potential for adsorbate to be adsorbed by the adsorbent will be greater (Alimano & Syafila, 2014).

Table 5 also shows that with increasing contact time of silica-activated carbon composite adsorbent with waste cooking oil, the organoleptic properties of the oil, especially the aroma or smell, tend not to meet SNI quality. This happens because the longer the adsorption time, the longer the contact between the surface of the adsorbent and the adsorbate, so that the adsorbent will reach saturation point and cannot absorb optimally. This saturation can be influenced by the active sites on the silica-activated carbon composite which are getting smaller so that the desorption process is getting bigger. This is supported by the statement of Hadiah & Meliasari (2017) who reported that the adsorbent's ability to absorb will decrease along with the increase in adsorption time because the pores of the adsorbent have been completely filled so that the adsorbent can no longer adsorb the components in the used cooking oil. On the other hand, a short adsorption time is also not good because there is not enough interaction time between the active silica-carbon composite and the waste cooking oil. This is supported by research by Novitriani & Intarsih (2015) that the short adsorption process causes not enough time to move the adsorbate to the adsorbent so that the impurities in the waste cooking oil have not been absorbed into the surface of the active silica-carbon composite.

Oil Quality Test Result from Adsorption

In this study, the chemical quality of the adsorption oil tested was the parameters of water content, free fatty acids, and peroxide numbers. These parameters are the main indicators of oil quality. Oil quality data after adsorption at optimum composition and time are shown in Table 6

Table 6. Waste Cooking Oil Sample Quality Test Data

No	Sample	Water Content (%)	Free Fatty Acid (%)	Peroxide Value (meq O_2/Kg)
1.	Waste Cooking Oil	0,46	0,43	22,32
2.	Oil adsorption results with a ratio of silica-activated carbon composite adsorbent (60:40) and an optimum time of 30 minutes	0,13	0,23	10,92
3.	SNI 2019	0,1	0,3	10
	Percentage (%)	71,74	48,88	51,07

Based on Table 6 shows the quality test in the form of water content, free fatty acids, and peroxide numbers in waste cooking oil and oil after adsorption using silica-activated carbon composite adsorbent (60:40) with a contact time of 30 minutes. The water content of waste cooking oil before adsorption was 0.46%. The high water content in waste cooking oil causes a hydrolysis reaction due to the presence of heat and catalysts. So that the oil (triglycerides) will break down into its components. The components of the oil are free fatty acids and glycerol. After the oil was adsorbed, the water content was 0.13% with a percentage decrease of 71.74%. The water content obtained has approached SNI 2019, which is 0.1%. The decrease in water content is due to the high mass ratio of silica in the silica-activated carbon composite (60:40), where silica has silanol groups so that it can bind water. This is supported by the statement of Sulo et al. (2019) who reported that silica has silanol groups (Si-OH) which have hydrophilic properties (like water) so that it can bind waterr.

The high water content in waste cooking oil is followed by high free fatty acids due to the hydrolysis reaction. Free fatty acids produced from this hydrolysis process are divided into two, namely saturated fatty acids and unsaturated fatty acids. Free fatty acids in used cooking oil are 0.43% while free fatty acids in oil from adsorption with silica-activated carbon composite adsorbent (60:40) are 0.23%. Free fatty acids obtained from adsorption have met SNI 2019, which is 0.3%. The high percentage of free fatty acid reduction in adsorption oil is due to the presence of silica in the adsorbent which is able to reduce free fatty acids because silica has silanol groups (Si-OH). This is supported by research by Yustinah & Hartini (2011) which reported that silica is able to reduce FFA levels due to the presence of silanol groups in the adsorbent. So that the oxygen-carbonyl group in FFA will react with hydrogen-silanol causing FFA molecules to be adsorbed on the silica surface. Research conducted by Reubun et al. (2024) using 1 gram of silica and a contact time of 90 minutes obtained a reduction in free fatty acids of 42.38%. Meanwhile, using a silica-activated carbon composite (60:40) with a contact time of 30 minutes obtained a reduction in free fatty acids of 48.88%. So the use of silica-activated carbon composites is very good in terms of mass and time efficiency compared to using only silica.

The last parameter in the quality test of waste cooking oil is the peroxide number. Waste cooking oil before adsorption has a peroxide number of 22.32 meq O_2/Kg. The high peroxide number is caused by unsaturated fatty acids resulting from hydrolysis which react with oxygen through the formation of free radicals. While the peroxide number in the oil resulting from adsorption with silica-activated carbon composite adsorbent (60:40) is 10.92 meq O_2/Kg. The resulting peroxide number has approached SNI 2019, which is 10 meq O_2/Kg.

The low peroxide number produced is due to the binding of free fatty acids to the adsorbent, so that unsaturated fatty acids cannot react with oxygen. Research conducted by Novitriani & Intarsih (2015) using activated carbon obtained a percentage reduction in peroxide number of 47.28%. Meanwhile, using a silica-activated carbon composite (60:40) with a contact time of 30 minutes obtained a percentage reduction in peroxide number of 51.07%. So the reduction in peroxide number using a silica-activated carbon composite (60:40) is more effective than using activated carbon adsorbent alone.

By testing the quality of the adsorption oil using a silica-activated carbon composite (60:40) with a contact time of 30 minutes, the water content and peroxide number were close to SNI 2019 and free fatty acids that met the requirements of SNI 2019 for cooking oil quality standards. Chemical parameter tests in the form of water content, free fatty acids, and peroxide numbers are directly proportional to their organoleptic properties. Legasari et al. (2023) stated that when oil contains a high water content, it causes hydrolysis and oxidation reactions.

CONCLUSION

The adsorbent ability of silica from rice husk ash and activated carbon applied to the purification of used cooking oil is optimum at a ratio of 60:40 with a contact time of 30 minutes. The results of the used cooking oil quality test obtained a water content of 0.13%, free fatty acids of 0.26% and a peroxide number of 10.92 meq O_2/Kg with an organoleptic test of bright yellow, less fried chicken odor and not cloudy. This silica-activated carbon adsorbent is superior in the purification of used cooking oil compared to using only silica or technical activated carbon alone so that it can be used to reduce waste cooking oil.

RECOMMENDATIONS

To complete the information related to the application of silica composite adsorbents from rice husk ash and activated carbon, further research is needed on the variables that influence the adsorption capacity of silica-activated carbon composites, such as temperature in the refining of waste cooking oil.

ACKNOWLEDGEMENTS

The author would like to express her infinite gratitude to Dr. Rahmanpiu, S.Pd., M.Sc. and Wa Ode Mulyana, S.Si., M.Sc. who have taken the time to provide motivation, guidance, direction and guidance with full sincerity so that this research can be completed well.

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