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Refining Used Cooking Oil Using Tapioca Starch (*Manihot esculenta*) and Lime (*Citrus aurantifolia*)

Fitri Hadiah [1*], Rona Alisya[1], Della Fitriyani[1]
Department of Chemical Engineering Faculty of Engineering Universitas Sriwijaya
Jl. Masjid Al Gazali Bukit Lama Kota Palembang Sumatera Selatan 30128
*E-mail Korespondensi : fitrihadiah@ft.unsri.ac.id
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Abstrak

Minyak goreng bekas atau biasa disebut minyak jelantah merupakan minyak goreng yang telah dipakai berkali-kali sehingga mengalami penurunan kualitas. Penurunan kualitas ini terjadi karena adanya degradasi komponen-komponen yang ada di dalam minyak goreng. Masyarakat khususnya pedagang lebih memilih untuk menggunakan minyak jelantah atau minyak goreng yang dipakai berulang kali karena lebih menguntungkan dibandingkan dengan menggunakan minyak goreng yang baru. Penggunaan minyak jelantah ini dapat menyebabkan masalah kolesterol pada tubuh. Penelitian ini bertujuan untuk memurnikan minyak jelantah menggunakan tepung tapioka yang kaya akan selulosa dan jeruk nipis yang kaya antioksidan yang diharapkan dapat mengembalikan kualitas minyak jelantah. Penelitian ini menggunakan metode eksperimen dengan memvariasikan massa adsorben yaitu perbandingan massa tepung tapioka sebesar 5%, 10%, dan 15% serta perbandingan massa jeruk nipis sebesar 5%, 10%, 15%, dan 20%. Proses pemurnian minyak jelantah dilakukan dengan mencampurkan minyak jelantah dan adsorben selama satu jam dengan kecepatan 200 rpm kemudian didiamkan selama 2 hari. Penelitian ini bertujuan untuk mempelajari pengaruh jumlah tepung tapioka dan jeruk nipis yang digunakan sebagai adsorben terhadap viskositas, densitas, dan bilangan asam minyak jelantah. Penggunaan adsorben yang optimal adalah 20% tepung tapioka dan 10% jeruk nipis, yang menghasilkan massa jenis 0,9760 g/cm^3, viskositas 18,9452, dan bilangan asam 2,8719 mg KOH/gram minyak.

Kata Kunci: adsorben, jeruk nipis, minyak jelantah, tepung tapioka, biodiesel

Abstract

Used cooking oil is cooking oil that has been used multiple times, leading to decreased in quality. This decline in quality occurs due to the degradation of the cooking oil's components. The community, especially traders, prefers to use used cooking oil or reuse cooking oil multiple times because it is more profitable than using fresh cooking oil. Using this used cooking oil can cause cholesterol issues in the body. This research aims to refine used cooking oil using carbon-rich tapioca starch and antioxidant-rich lime which is expected to restore the quality of used cooking oil. This research uses the experimental method by varying the mass of the adsorbent, specifically the ratio of tapioca starch mass at 5%, 10%, and 15%, and the lime mass ratio at 5%, 10%, 15%, and 20%. The used cooking oil refining process was conducted by mixing used cooking oil and adsorbents for one hour at a speed of 200 rpm and then allowed to stand for 2 days. This research aims to study the effect of the amount of tapioca starch and lime as adsorbents on the viscosity, density, and acid number of refined used cooking oil. The optimal use of adsorbent is 20% tapioca starch and 10% lime, resulting in a density of 0.9760 g/cm^3, viscosity of 18.9452, and an acid number of 2.8719 mg KOH/gram oil.

***Key words**: adsorbent, lime, used cooking oil, tapioca starch, biodiesel*

INTRODUCTION

Indonesia's abundant cooking oil should be sold at an economical price. This is because Indonesia is one of the largest palm oil-producing countries in the world. Indonesian Minister of Agriculture Syahrul Yasin Limpo stated that Indonesia has an oil palm area of 16.38 million hectares and produces 46.8 million tons of crude palm oil (Kementrian Pertanian Direktorat Jenderal, 2022). However, the price of cooking oil on the market is increasing daily. The price of cooking oil is rising because producers are manipulating the market. The government revoked the regulation and enacted Minister of Home Affairs Regulation No. 11 Year 2022, setting the price of bulk cooking oil at Rp14,000/L since March 16, 2022. The price of packaged cooking oil also increased to Rp18,000-Rp25,000/L (Fadilah, 2022). This situation has led people, especially traders, to use used cooking oil as cooking oil.

The community, especially traders, prefers to use used cooking oil or reuse cooking oil multiple times because it is more profitable than using fresh cooking oil. However, used cooking oil can cause cholesterol issues because it affects High-Density Lipoprotein (HDL) and Low-Density Lipoprotein (LDL) cholesterol levels. Trans fats in the oil accumulate and form plaque on the artery walls, resulting in the narrowing of the arteries, which can lead to heart disease (Arlofa et al., 2021).

Cooking oil contains about 80% unsaturated fatty acids, including oleic acid and linoleic acid (Sani, 2017). This content decreases and produces other compounds with repeated use. Cooking oil that is used many times can produce free radical compounds that are toxic to organs and harmful to the body. Excessive free radicals in the body cause cell damage, including heart muscle cells. Used cooking oil undergoes oxidation due to high temperatures, which can spur the growth of cancer cells in the liver. Uncontrolled cell growth in the liver hampers its function and triggers cancer.

Tapioca starch and lime can be used in purifying used cooking oil. Tapioca starch can adsorb impurities and affect free fatty acids, improving oil clarity. Tapioca starch contains 17% amylose and 83% amylopectin, with a granule size of 3-3.5μ (Jayanti et al., 2017). The compound amylose $(C_6H_{10}O_5)n$ and amylopectin $(C_{15}H_{26}O_{13})n$ have structural formulas similar to cellulose $(C_6H_{10}O_5)n$, making them suitable as adsorbents. Amylose and amylopectin are polysaccharide carbohydrate derivatives, just like cellulose, and can replace it as a carbon source. The porous structure of amylose and amylopectin can adsorb harmful compounds and improve the quality of used cooking oil. These starch compounds contain hydroxyl groups or -OH groups that can bind to free fatty acids in used cooking oil. They also contain carbon with functional groups -C-H, C-O, C=O, and C-C, which function to adsorb harmful compounds from used cooking oil.

Tapioca starch can be combined with lime, as lime can also reduce the peroxide number in used cooking oil. Lime (Citrus aurantifolia) is recognized as a natural antioxidant. Natural antioxidants have been tested for their effectiveness in reducing the amount of saturated fat and trans fat in cooking oil (Hwang & Moser, 2016). Lime was chosen because it contains antioxidant compounds that are beneficial for inhibiting the activity of radical oxidation. These antioxidant compounds include alkaloids, phenols, saponins, tannins, steroids, and flavonoids (Pratiwi & Ferdiansyah, 2015). Both of these materials can be found in everyday life, making them easily accessible for implementation.

Refining used cooking oil by using tapioca starch as adsorbent, which is rich in carbon, and antioxidant-rich lime is expected to restore the quality of used cooking oil based on the Indonesian National Standard (SNI). Some studies had been conducted related to this study. Soaking cooking oil with cellulose from sugar cane bagasse can reduce the acid value by up to 52.31% and reduce the peroxide value by up to 68.36% and it could have the potential as an adsorbent for used frying oil (Wulandari & Dewi, 2018). The use of rice and aking rice as an adsorbent for used cooking oil uses a batch method that uses rice as an adsorbent for used cooking oil which allowed 0.2826% FFA for oil samples from household traders, while the adsorption of white rice contained 0.3159% FFA (Berghuis et.al, 2022). Study to improve the quality of used cooking oil include using activated charcoal from rice husks (Samangun et al., 2017), in this study it was said that used cooking oil decreased oxidant and peroxide nicai using adsorbents that have high enough cellulose. Research conducted by Qory et al., (2021) explained that activated carbon from salak seeds as an adsorbent can reduce free fatty acid levels, acid numbers, water content, and peroxide numbers. Research conducted by Permata et al, (2018) explained that lime has a fairly high antioxidant category. Research conducted by Susilo et al., (2023) stated that tapioca starch can reduce MDA levels, fatty acids, and reduce the turbidity of used cooking oil.

Other related research is the use of banana peel which contains natural antioxidants as which can improve oil quality in terms of free fatty acid parameters. Banana peels reduce free fatty acid levels and improve the physical properties of used cooking oil (Almagribi et al., 2022). Clove oil containing 78-98% eugenol is used as an adsorbent to reduce the amount of peroxide in cooking oil (Widodo et al., 2020). Mandarin and lemon peel antioxidants can be used as adsorbents on oxidative stability during repeated oil frying by evaluation using EPR and conventional methods. Each chemical method gives rise to different product associations with different oxidation stages (Aydın et al., 2021).

Therefore, this study was conducted to combine tapioca starch and lime where the collaboration of these two adsorbents has not been carried out by previous studies so that these two adsorbents can improve the quality of cooking oil. This is the basis for this research, which aims to determine the relationship between the mass percentage ratio of tapioca starch and lime, as well as the operating conditions used. Additionally, this research aims to study the effects of these variations on the viscosity, density, and acid number of purified used cooking oil.

METHODS

Preparation of Materials and Tools

The materials used are used cooking oil taken from the processing of household food processing, tapioca starch, lime, chloroform, 97% ethanol, pp indicator, and 0.1M KOH. The tools used in this research are beaker glass, measuring cup, erlenmeyer, spatula, funnel, drip pipette, magnetic stirrer, hot plate, stative, clamp, burette, screen printing monyl fabric t200, monyl fabric screen printing t90, 5ml pycnometer, Ostwald viscometer, analytical balance, suction ball digital balance, thermometer, aluminum pan, aluminum foil, knife, cutting board, oven, stopwatch, and sample bottles.

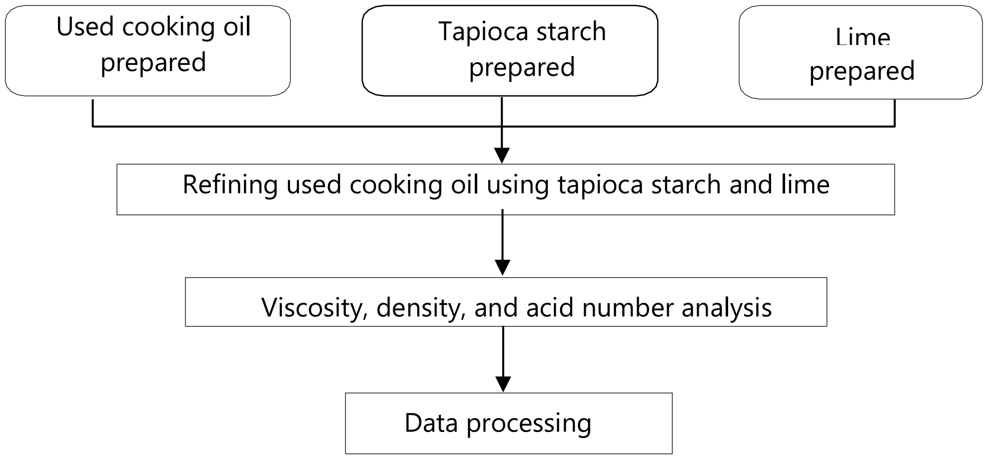

Figure 1.Research Methodology

Pretreatment of Used Cooking Oil and Lime

Used cooking oil is filtered first using a monyl screen cloth T90 screen printing cloth so that the impurities in the used cooking oil which is quite large can be separated. Lime is washed thoroughly with water. The lime is cut into pieces including the peel to a small size and then separated with the seeds. Heat the lime that has been cut into small pieces in an oven at 35ºC for 5 hours.

Refining Used Cooking Oil with Starch Tapioca Starch and Lime

The filtered used cooking oil was measured to a volume of 100 mL using a measuring cup, and then poured into a beaker. Tapioca starch, equal to 5% by weight of the used cooking oil, was added to the beaker containing the used cooking oil. Lime, also equal to 5% by weight of the used cooking oil, which had been cut into small pieces and dried, was then added to the sample of used cooking oil. The mixture was stirred using a magnetic stirrer at a speed of 200 rpm for 60 minutes at 35°C. The stirred mixture was filtered using a T200 monyl screen placed over another beaker. These steps were repeated for tapioca starch mass variations of 10% and 15%, and for lime mass variations of 5%, 7.5%, 10%, 12.5%, and 15%. The resulting purified cooking oil samples using tapioca starch and lime adsorbents were then tested for density, viscosity, and acid number.

Density Testing

A total of 20 grams of the sample was placed into a 250 mL beaker glass, then heated to a temperature of 40°C. The weight of the empty pycnometer was measured using an analytical balance, and the weight was noted. The sample was then added to the 5 mL pycnometer until it exceeded the pycnometer's capacity. The outside of the pycnometer was wiped and dried using a tissue. The pycnometer was then covered with its lid and weighed again using the analytical balance. The weight of the filled pycnometer was noted. The density was calculated using the equation (1) below:

$$\rho = \frac{m_2 - m_1}{V} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where:

ρ = Density (g/cm^3)

m_1 = Mass of empty pycnometer (g)

m_2 = Mass of pycnometer filled with the sample (g)

V = Pycnometer volume (mL) = 5 mL

Viscosity Testing

A total of 20 grams of the sample was placed into a 250 mL beaker glass and heated to a temperature of 40°C. The sample was then inserted into the viscometer until it exceeded the upper limit of the capillary tube and reached the middle of the reservoir boundary. The suction ball was removed, and the sample flow velocity was measured using a stopwatch as it passed from the upper limit to the bottom of the capillary tube. The viscosity was calculated using equation (2) as follows:

$$\eta_1 = \eta_2 \text{ x } \frac{\rho_1 \, x \, t_1}{\rho_2 \, x \, t_2} \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

where:

η_1 = Sample viscosity (cSt)

η_2 = Water viscosity (cSt)

ρ_1 = Sample density (g/cm^3)

ρ_2 = Water density (g/cm^3)

t_1 = Average sample time (s)

t_2 = Average water time (s)

Acid Number Testing

A total of approximately 10 grams of the sample was placed into a 250 mL Erlenmeyer flask. Then, 25 mL of 97% ethanol was mixed with 25 mL of chloroform in a 250 mL Erlenmeyer flask, and 5 drops of PP indicator were added. This mixture of ethanol, chloroform, and PP indicator was titrated with a 0.1M KOH solution until it turned pink and the color remained for 15 seconds, then the titration was stopped. The titration volume of this mixture was noted. The mixture was then combined with the sample in the Erlenmeyer flask containing the sample. The sample was titrated with a 0.1M KOH solution until it appeared pink, matching the color of the initial titration mixture, and remained for 15 seconds, then the titration was stopped.

$$\text{AA} = \frac{\text{NKOH} \, x \, BMas \, x \, V}{\text{m}} \ldots\ldots\ldots\ldots\ldots (3)$$

where:

AA = Acid number

NKOH = Normality of KOH = 0.1N

BMas = Molecular weight of fatty acid = 56.1

V = Volume of KOH titration (mL) = V_2 - V_1

m = Mass of the sample

RESULT AND DISCUSSION

Used Cooking Oil Refining

Adsorbents of tapioca starch and lime that have been prepared and are ready to be used as adsorbents can be seen in Figure 2. The adsorption of used cooking oil with these adsorbents were filtered and weighed to observe the mass difference before and after adsorption. The results of the used cooking oil are presented in Figure 1, with an initial mass of 100 gr. Figure 3 shows that generally, the more adsorbent used, the less used cooking oil is produced. This indicates that impurities are increasingly adsorbed with the use of more adsorbents.

Figure 1. Tapioca starch (a) and lime (b) adsorbents

The line graph as in Figure 3 shows used cooking oil adsorption results using tapioca starch and lime with their variation. The mass of used cooking oil after refining is highest on using 5.0% of lime mass whilst the lowest is on using 12.5% of lime mass. The same pattern occurs on using of percentage of tapioca starch mass. It can be implied that the less adsorbent is used, the more used cooking oil mass is produced. This is because the more adsorbent is used, the more impurities are adsorbed. In addition, with a high amount of adsorbent used, used cooking oil that is bound on adsorbents is also high which leads used cooking oil in high concentration passes the filtration. This causes the production of used cooking oil using a small amount of adsorbent is less than using a lot of adsorbent. However, the result of 15.0% of lime mass used is higher than 12.5% because samples with a high amount of adsorbents is very difficult to filtrate so there are still adsorbents and impurities in the final result of used cooking oil production.

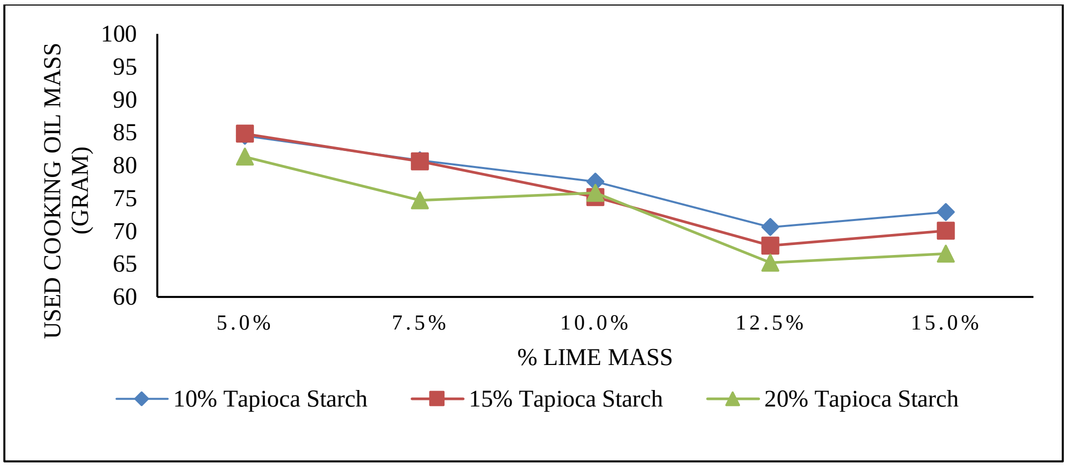

Figure 3 Graph of used cooking oil adsorption results

The used cooking oil after adsorption, as shown in Figure 4, shows that there is not significant color difference between the oil before and after adsorption. It can be concluded that tapioca starch and lime adsorbents can not adsorb color. The adsorption results also showed no difference in odor, which is the same as the used cooking oil that has not been adsorbed. The odor of used cooking oil before adsorption is caused by the rupture of triglycerides into glycerol and FFA (Milah, 2019). This is why used cooking oil adsorbed by tapioca starch and lime does not have a change in odor.

(a) (b) (c)

Figure 4 The yield of used cooking oil after adsorption from left to right is shown for lime compositions of 5%, 7.5%, 10%, 12.5%, and 15% for 10% tapioca starch (a), 15% tapioca starch (b), and 20% tapioca starch (c).

Analysis of Initial Sample Characteristics

The untreated used cooking oil and Fortune oil (one of the cooking oil brands in Indonesia) underwent analysis aimed at determining the potential of tapioca starch and lime as adsorbents by measuring changes in characteristics before and after adsorption. This analysis included acidity, density, and viscosity. The characteristics of these oils are presented in Table 1. The untreated used cooking oil has a lower density (0.9718 g/cm^3) compared to Fortune oil (0.9763 g/cm^3). This indicates that the used cooking oil has been heated, which reduces the molecular bonds between the oil molecules (Warsito & Miftahul, 2013). The reduction in these molecular bonds results in a decrease in the density of the oil. The unused frying oil has a higher density because it hasn't been heated, so its molecular bonds remain intact and haven't stretched.

Table 1. Characteristics of used cooking oil before adsorption

No	Characteristic	Untreated used cooking oil	Fortune Oil
1	Density	0,9718 g/cm^3	0,9763 g/cm^3
2	Viscosity	22,3829 cSt	19,7964 cSt
3	Acid Number	5,5049 mg KOH/g sample	0,7742 mg KOH/g sample

The viscosity of the untreated used cooking oil is higher (22.3829 cSt) compared to Fortune oil (19.7964 cSt). This suggests that the repeatedly used cooking oil has undergone complex reactions during the frying process (Nasir, 2020). These complex reactions include the dissolution of fats from food. The dissolved fats remain in the used cooking oil, contributing to a significant increasing in its viscosity.

The acidity of the used cooking oil exceeds the standard quality for cooking oil (with Fortune oil as a comparison). The untreated used cooking oil has a higher acid number (5.5049 mg KOH/g sample) compared to Fortune oil (0.2132 mg KOH/g sample). The high acidity in the untreated used cooking oil is due to the presence of high levels of free fatty acids. These free fatty acids are produced from heating processes that involve hydrolysis and oxidation reactions (Masyithah et al, 2018) The very high acid content can pose health risks if reused, such as bad cholesterol and heart disease (Arlofa et al, 2021) The used cooking oil will be adsorbed using tapioca starch and lime to achieve characteristics that are close to or meet the quality standards for cooking oil, particularly in terms of acidity, density, and viscosity.

The Effect of Adsorbent Mass on Density

The density data of the used cooking oil after adsorption is presented in Figure 5. The used cooking oil before adsorption has a density of 0.97522 g/cm^3. The density of the used cooking oil after adsorption shows values that are not significantly different from the untreated used cooking oil. The density of Fortune oil is higher than that of the untreated used cooking oil, measuring 0.9757 g/cm^3.

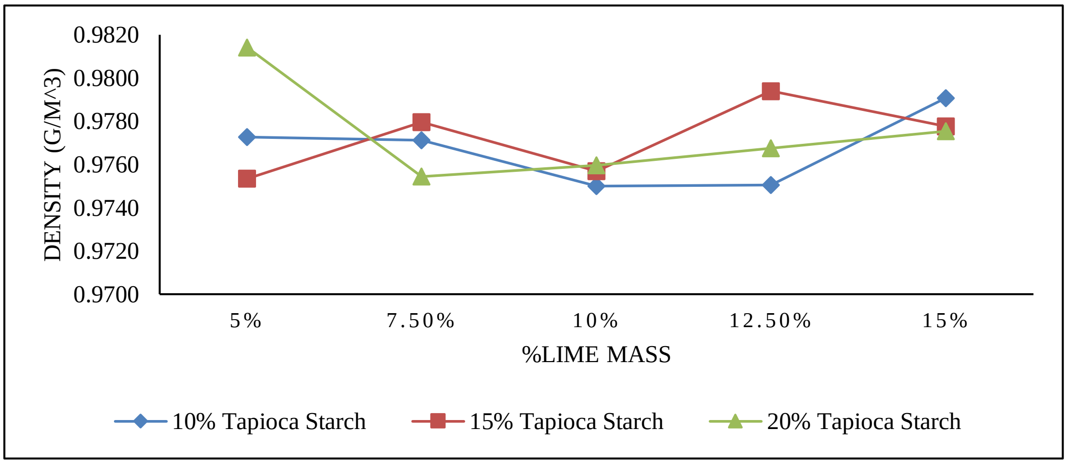

Figure 5 Data analysis graph of used cooking oil purification density results

The 20% tapioca starch adsorbent has a pretty high density. Using 20% tapioca starch mass along with 15% lime mass, the density goes up compared to used cooking oil that hasn't been treated. Using 15% tapioca starch adsorbent results in higher density than 10% tapioca starch because the filtration isn't complete. This means some of the adsorbent is still in the oil that's been treated. Basically, the more tapioca starch and lime adsorbent are added, the higher the density gets. This happens because adding the adsorbent makes the density of the used cooking oil increase, bringing it closer to the density of fresh cooking oil. The not-so-great separation of the used cooking oil and adsorbent also plays a role in this issue. Some adsorbents with adsorbed compounds get left behind after separation, which adds to the density of the used cooking oil.

Effect of Adsorbent Mass on Viscosity

Viscosity is one of the important factors in selecting refined used cooking oil for reuse. This variable aims to determine the resistance value of the liquid to flow (Sugiharta et.al, 2021). The viscosity data of the refined used cooking oil in this study are presented in Figure 5. Figure 5 shows that the more tapioca starch and lime are used, the lower the viscosity value. This is because the adsorbent absorbs complex reaction products such as decomposed fat. The use of 15% tapioca starch adsorbent shows a higher viscosity than 10% tapioca starch due to less than optimal filtration. This causes the adsorbent to still be in the adsorbed oil. Overall, the use of more adsorbent produces used cooking oil with lower viscosity because more impurities are absorbed by the adsorbent. This statement is also supported by previous research, namely related research conducted by Hadiah et.al. (2020) who purified used cooking oil with moringa seeds and bentonite. This study explains that the lower the viscosity of used cooking oil, the better the adsorption process of free fatty acids in used cooking oil.

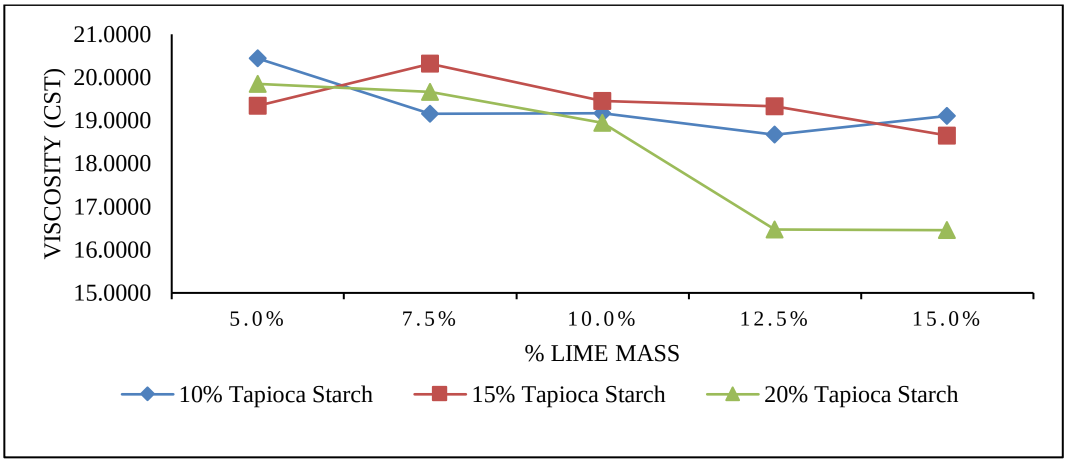

Figure 6 Data analysis graph of used cooking oil purification viscosity results

The results of refining used cooking oil using tapioca starch adsorbent with lime on viscosity testing are not much different from the viscosity of fortune oil (19.7964 cSt) which has also been tested in this study. The use of both adsorbents above 10% gives a lower viscosity value than fortune oil. This also means that this used cooking oil has a lower flow resistance than fortune oil. This oil can be a good choice of used cooking oil for reuse if determined based on low viscosity or flow resistance.

Used cooking oil can be utilized as raw material for biodiesel. The viscosity of diesel oil according to SNI 04-1782-2006 is 2.3-6.0 cSt (at 40°C). Low viscosity biodiesel can lead to leaks in the fuel injection pump, while if the viscosity is too high, it can affect the performance of the fuel injection system and make it harder to atomize the fuel (Hartono et al, 2023) By using this adsorbed used cooking oil, it is expected to produce biodiesel with viscosity that meets SNI standards. Using used cooking oil as a raw material for biodiesel can be done through transesterification reaction aimed at reducing the oil's viscosity (Cahyati & Pujaningtyas, 2018). This is because the viscosity of this used cooking oil is not much different from that of Fortune cooking oil that has never been used for frying, so the viscosity will also be quite similar.

The Effect of Adsorbent Mass on Acid Number

The acid number of used cooking oil is shown in Figure 7. The untreated used cooking oil had an acid number of 5.5049 mg KOH/g of oil, which decreased to average acid number of 2.9350 after adsorption. This reduction in acid number is quite significant, showing a decrease of 53.31% acid number in the used cooking oil. However, this reduction is still not good enough as it hasn't approached the acid number of Fortune oil, which is 0.2132.

Figure 7 shows that using tapioca starch adsorbent with lime adsorbent at 5-10% will result in a lower acid number. This is because the more adsorbent used, the more free fatty acids (FFA) contained in the used cooking oil are adsorbed. Using lime at more than 10% or using 12.5-15% causes an increase in the acid number. This happens because the separation process of the used cooking oil after being adsorbed with the lime adsorbent is not optimal, leaving a small amount of lime adsorbent still in the used cooking oil. The acidic nature of lime affects the acid number analysis results.

The purification results of this used cooking oil can not be reused as cooking oil. This oil needs to be further purified, such as by re-adsorbing it with new adsorbent. However, the used cooking oil obtained from this adsorption can be used as raw material for biodiesel production. The oil with an average acid number of 2.9350 meets the requirements for the acid number of biodiesel raw materials, which is a maximum of 5 without the esterification process (Busyairi et al, 2020).

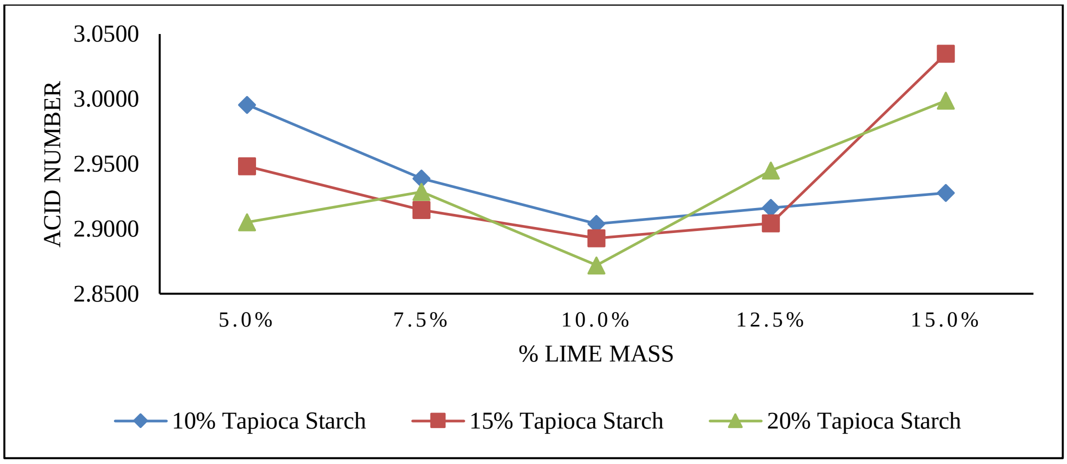

Figure 7 Data analysis graph of used cooking oil purification acid number results

Lime contains citric acid which works by binding to polar molecules such as free fatty acids and precipitating them, thereby reducing free fatty acid levels in the oil. At certain concentrations (up to 10%), citric acid is effective in binding polar compounds. When the concentration of lime increases above the optimal point (e.g. 10%), the excess citric acid may cause the oil to no longer be able to hold the additional citric acid and thus the effectiveness decreases (Waluyo et al., 2020). Excess citric acid can attract water dissolved in the oil, accelerating the hydrolysis reaction of triglycerides to free fatty acids, which increases free fatty acid levels (Sasangko & Legahati, 2020).

Tapioca starch has a good adsorption capacity to bind dirt, colored compounds, and some polar compounds. However, an unbalanced increase in lime concentration can reduce the synergy between these two ingredients. Excess citric acid molecules can compete with other polar compounds to interact with tapioca starch, making the adsorbent less effective in adsorbing free fatty acids. Research by Yustina & Rosdiana (2014) showed that the addition of lime to a certain concentration was effective in reducing the acid number in used cooking oil. However, an excessive increase in citric acid concentration can cause a decrease in the effectiveness of reducing acid numbers. At a fixed concentration of tapioca starch (10%, 15%, or 20%), the adsorption capacity can reach saturation or near saturation of the adsorbate. After this point, increasing lime will not help the reduction of free fatty acids.

CONCLUSIONS

Purifying used cooking oil with tapioca starch and lime adsorbents affects the mass yield of the adsorbed used cooking oil, density, viscosity, and acid number. The more tapioca starch is used, the less used cooking oil is produced, the density increases, the viscosity decreases, and the acid number decreases. The more lime is used, the less used cooking oil is produced, the density decreases, the viscosity decreases, and the acid number increases. The optimal use of adsorbents is 20% tapioca starch and 10% lime, with characteristics of density 0.9760 g/cm^3, viscosity 18.9452, and acid number 2.8719 mg KOH/gram of sample oil.

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