

## Stability Test of Modified Chitosan Adsorbent to Improve the Quality of Used Cooking Oil

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**Abstract**—Chitosan membrane modified by pine charcoal has high stability and can be used as an adsorbent medium to improve the quality of used cooking oil. This study aims to determine the optimum concentration of chitosan membrane modified by activated pinecone charcoal, test the membrane stabilization in the refining process of used cooking oil, and characterize the refined use of used cooking oil. The method used in this study went through several stages, starting with the stages of producing activated charcoal from pine cones, then producing chitosan membranes modified by activated charcoal at various concentrations, which were then tested for membrane stabilization and refined oil characteristics. The results obtained from this study were the optimum concentration of the membrane in the ratio of chitosan and activated charcoal of pine cones, namely 3.5 : 1.5 with a total mass of 7 grams, resulting in a K2 membrane morphology with an elastic and dense state. In the FTIR test, a group absorption pattern amida function was obtained (N-CH<sub>2</sub>) stretching at a wave number of 2350 cm<sup>-1</sup> against K2 membranes and membranes that are not resistant to acidic and alkaline chemicals, as well as the purified membrane's fatty acid content decrease of 0.442% and water content test of 0.6%, the tensile strength of the membrane at break, which is 2.25 and high porosity pine charcoal chitosan membrane has a 27%.

**Keywords**— Chitosan membrane; pine charcoal; cooking oil; adsorption method.

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### I. INTRODUCTION

The problem is decreasing availability of cooking oil to meet daily needs. This encourages repeated use of cooking oil or used cooking oil. However the repeated use of cooking oil will have an impact on health [1]. After used, the cooking oil will undergo changes and when viewed from its chemical composition, used cooking oil contains compounds that are carcinogenic, which occur during the frying process [2]. This change in properties makes the cooking oil unfit for use as a food ingredient [3]. The various purification methods have been developed to reduce the levels of fatty acids and peroxide value in used cooking oil, so that it meets the requirements as a food ingredient and is suitable for consumption and safe for health [4]. The purification method that has been widely developed is the adsorption method using a membrane [5]. The adsorption membrane method has many advantages, namely large adsorption power, fast adsorption time and easy and inexpensive to use [6].

Chitosan is produced by chitin and has the same chemical structure as chitin, consisting of long molecular chains and high molecular weights [7]. The difference between chitin and chitosan is that in each ring of the chitin molecule there is an acetyl group (-CH<sub>3</sub>-CO) on the second carbon atom, whereas in chitosan there is an amine group (-NH) [8]. Chitosan can be produced from chitin through a deacetylation process, namely by reacting it with high concentrations of alkali for a relatively long time and at high temperatures. Chitosan is a unique biopolymer, namely in an acidic solution, chitosan has cationic characteristics and has a positive charge, whereas in an alkaline solution, chitosan will precipitate [9].

Chitosan is a white amorphous solid which is insoluble in alkalis and mineral acids except under certain circumstances [10]. The best solubility of chitosan is in a solution of 2% acetic acid, 10% formic acid and 10% citric acid [11]. Chitosan cannot dissolve in pyruvic acid, lactic acid and inorganic acids at a certain pH, even after being heated and stirred for a long time [12]. Chitosan has unique properties

that can be used in various ways and has several different uses. Various among others adhesives, additives for paper and textiles, cosmetics, purified water purifiers, as well as to accelerate wound healing, and improve the binding properties of colors [13].

Chitosan from crab shells is a polysaccharide that has been widely used as an adsorbent due to chitosan being a good ion-complexing agent, which is characterized by shifts in the wave numbers of several functional groups in the chitosan chain [14]. Chitosan adsorbents are much more effective in adsorbing metal ions  $\text{Fe}^{2+}$ ,  $\text{Ni}^{2+}$  and  $\text{Cu}^{2+}$  than adsorbents from sawdust. This shows that chitosan has greater potential than sawdust for heavy metal adsorption applications [15].

Charcoal or carbon is a porous solid biomass resource, including various trees, leaves, plant roots, fruit peels, and grasses, which is the result of combustion through the carbonization process. The components of charcoal consist of fixed carbon, ash, water, nitrogen and sulfur. Activated charcoal can adsorb certain gases and chemical compounds or its adsorption properties are selective, depending on the size or volume of the pores and surface area. The use of activated charcoal as an adsorbent molecule has also been developed where activated charcoal has advantages in absorbing cationic anions as well as organic and inorganic molecules in the form of solutions and gases. So the aim of the study was to determine the optimum conditions for the composition and adsorption time, the effectiveness of the chitosan-activated pine charcoal membrane which included surface area, iodine adsorption and the characteristics of refined used cooking oil which included the decrease in free fatty acids, peroxide number and Pb metal ion content.

## II. MATERIAL AND METHOD

### A. Materials

Used cooking oil, chitosan, pine activated charcoal, Phenolphthalein/PP indicator (pa Merck), sodium thiosulfate/ $\text{Na}_2\text{S}_2\text{O}_3$  0.1N (pa Shagufuta Laboratory), chloroform (pa Merck), potassium iodide/KI (pa Merck), aquadest (Rofa), 96% ethanol (food ROFA), sodium hydroxide/ $\text{NaOH}$  0.1 N (pa Merck), glacial acetic acid (acs Merck), and starch/starch indicator (pa Merck). Used cooking oil (used frying oil), Chitosan, coconut shell, 50% KOH, 0.1000 N  $\text{Na}_2\text{S}_2\text{O}_3$  Ethanol 70%

### B. Instrumentation

Digital balance (uniweigh), 100 mesh standard sieve (CBN), hotplate, magnetic stirrer, 250 ml Erlenmeyer (Pyrex), 100 ml, 250 ml and 1000 ml beakers (Pyrex), stative and clamps (Usbeck Germ), thermometer, 100 ml, 50 ml and 10 ml (Pyrex) measuring cups, glass funnel (Pyrex), 50 ml burette (Schoot-Ger), Rubber bulb or pipette filler, spray bottle, spatula (Usbeck Germ), 25 ml measuring pipette, 10 ml and 5 ml (Pyrex), desiccator (DURAN), pipette (Pyrex), watch glass (Pyrex), blender (Philip), knife, scissors, mortar and pestle (RRC), aluminum foil (Best Fresh), universal indicator (Merck) and filter paper (Whatman). The tools used in this study were: laboratory glassware, hotplate, magnetic stirrer, teflon, centrifuge,

porcelain cup, oven, furnace, adsorption box, mercury (Hg) lamp 150 watts, BET Quatachroma Nova 2000 version 11.0, FTIR-8400S SHIMADZU.

### C. The process of making membranes uses NIPS (Non-solvent Induced Phase Separation) with the immersion precipitation method.

Chitosan is dissolved in a solvent in the form of a 5% acetic acid solution, then activated charcoal is added with a ratio chitosan : pine charcoal of 4 : 1 ; 3.5 : 1.5 ; 3 : 2 ; 1.5 : 3.5 and 1 : 4 dissolved in  $\text{NaOH}$  then added 15% fiberglass. The polymer solution was dissolved and stirred with a magnetic stirrer until homogeneous for 3 hours. Then it was poured onto a glass plate, and waited for about 5 minutes. Then the membrane characteristics were tested using SEM and a digital microscope to test the membrane morphology, FTIR to determine the functional groups and the wavelength of the cellulose acetate membrane

### D. Process of refining used cooking oil using activated charcoal substituted chitosan membrane

The membrane is placed into a container containing 200 ml of used cooking oil. Furthermore, the chitosan membrane was soaked in the used cooking oil for 3 hours. Characterization and effectiveness test of membrane performance is carried out with the aim of knowing the physical properties and performance results of the membrane in carrying out its functions optimally as desired. The characterization carried out included the contact angle test (Contact Angle Goniometer), mechanical properties (tensile test), FT-IR, membrane porosity test, membrane morphology test (SEM-EDX), and rejection flux.

### E. Chitosan Membrane Stability Test - Pine Charcoal

Resistance to solvents

Heat resistance

Stability for repeated use

### F. Characterization Chitosan Membrane \_Pine Charcoal

Porosity test is by immersing the membrane in water for 24 hours at room temperature, then the membrane is weighed. After that the membrane was dried in a vacuum oven at 60°C for 48 hours until completely dry and then weighed again. The magnitude of the membrane porosity can be calculated using the following equation:

$$\% \text{ Porosity} = (-W_{\text{dry}}) \times 100\%$$

### G. Morphology Test

SEM was used to determine the membrane morphology. The membrane sample is immersed or placed in liquid nitrogen for a few seconds until the sample hardens, then it is broken using tweezers on both sides. Then the sample is determined the surface morphology as well as the morphology of the cross section, followed by EDS to find out what elements are in the membrane.

### H. Determination of Free Fatty Acid Content

Weigh 20 grams of 250 ml Erlenmeyer input sample, then dissolve it in 50 ml of warm neutral 70% alcohol and add 5 drops of phenolphthalein indicator. Titrate the solution with  $\pm 0.01$  N  $\text{NaOH}$  so that a pink color is formed (the color does not disappear within 30 seconds). Furthermore, it is stirred

by shaking the Erlenmeyer during the titration. Record the required volume of  $\pm 0.01$  N NaOH solution.

### I. Tensile Test

The tensile test is used to determine the mechanical properties of the membrane. Preparation was carried out by measuring the thickness of the membrane, then the membrane was cut 2 mm x 4 cm. Then both ends of the membrane sample were clamped with a tensile tester and pulled until they broke using a speed of 10 mm/minute. Data obtained from the results of this tensile test include stress (stress) and strain (strain) at minimum and maximum conditions. The tool used for this tensile test is the Favigraf brand. Characterization of refined used cooking oil was carried out by physical testing of the oil using organoleptic quality tests of color, clarity, viscosity and aroma. While chemical testing uses laboratory tests with two parameters, namely free fatty .

### J. Peroxide number determination

Peroxide number is determined as follows: 5.00  $\pm$  0.05 g cooking oil is weighed and then put into a 250 ml Erlenmeyer flask with a lid. Next, 12 ml of chloroform and 18 ml of glacial acetic acid were added to the flask. The solution was shaken until all the ingredients dissolved. After all the ingredients were mixed, 0.5 ml of KI saturated solution was added. For 1 minute the solution mixture was allowed to stand while still being shaken. Then added 30 ml of distilled water. Next, 0.5 ml of 1% starch was added to the mixed solution and immediately titrated with 0.1000 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> until the solution changes color from blue until the blue color starts to disappear. The determination was carried out by repeating 3 times. Peroxide numbers are expressed in mg-equivalent peroxide in every 100g of sample.

## III. RESULTS AND DISCUSSION

The chemical resistance of the membrane is tested by immersing it in various chemicals. The chemicals used are H<sub>2</sub>SO<sub>4</sub> 3%, KOH 3%, and NaCl 10%. Table 1. shows the percentage of weight loss from the membrane. According to table 4.3, the membrane in salt state (10% NaCl) experiences an average weight loss of 21%, the lowest percentage loss between acidic and basic chemicals, the membrane in acidic state is average - an average weight loss of  $\pm$  45%, and modified membranes immersed in a 3% alkaline chemical solution (KOH) experienced the highest average weight loss percentage of  $\pm$  55%.

The lack of interactions in the chitosan membrane structure modified by activated pinecone charcoal caused the high percentage of weight loss. This demonstrates that the modified membrane is resistant to salt while remaining resistant to acids and bases. The presence of alkaline (base) resistance to hydrolyzed ester bonds present in the structure of nyamplung oil on the membrane, or the saponification process, is responsible for the high percentage of membrane weight loss in alkaline chemicals. This is because the membrane's surface area is smaller than the volume of the chemical being tested, so the chemicals react faster on membranes with small surfaces. The smaller the membrane's surface, the shorter the membrane's resistance to the membrane.

TABLE I  
PERCENTAGE OF WEIGHT LOSS FROM THE MEMBRANE

Membrane	Solution	Initial weight (W)(g)	Final weight (W <sub>o</sub> )(g)
K1	H <sub>2</sub> SO <sub>4</sub> 3%	0,13	0,079
K2		0,17	0,086
K3		0,19	0,097
K1	KOH 3%	0,15	0,064
K2		0,20	0,091
K3		0,18	0,084
K1	NaCl 10%	0,16	0,13
K2		0,20	0,16

### A. Characterization

#### 1) Tensile Test

Mechanical strength analysis based on tensile tests and mechanical property measurements are performed to determine the strength of the membrane when subjected to forces that can damage the membrane. Tensile test measurements can be used to describe mechanical properties. The tensile test is designed to determine the amount of force required to break the pine charcoal chitosan membrane. Tensile tests were performed at room temperature with a Favigraf tool, which produced values for the tensile strength of the membrane at break and the tensile strength of the membrane at break, which is 2.25 MPa. Stress is defined as the ratio of force magnitude to cross-sectional area. Meanwhile, strain is the ratio of the increase in membrane length caused by equal and opposite tensile forces. Chitosan membranes with a concentration of 3% have the highest tensile strength and elongation (strain) compared to chitosan membranes with a concentration of 1%, 2%, 4% or 5% concentration. The data can be seen in Table 3. This happens because chitosan can dissolve well. in the 3% chitosan membrane to produce a membrane with a pore structure that is evenly distributed over the entire surface, is elastic and has stronger tensile strength and tensile strength.

TABLE II  
TENSILE STRENGTH OF PINE CHARCOAL CHITOSAN MEMBRANE

No	Membrane	thickness	$\Sigma$ N / mm <sup>2</sup>
1	4 : 1	0.03	0.0426
2	3.5 : 1.5	0.03	0.0537
3	3 : 2	0.03	0.0600
4	1.5 : 3.5	0.03	0.0275
5	2 : 3	0.03	0.0148

The membrane adsorbent of chitosan-activated pinecone charcoal was characterized to determine its functional group using FT-IR Figure 1).

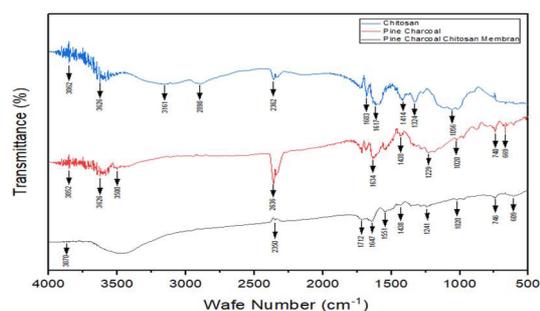


Fig. 1 Spektra FTIR pine Charcoal Chitosan membran

TABLE III  
THE FUNCTIONAL GROUP OF CHITOSAN

Functional groups	Wave Number (cm <sup>-1</sup> )		
	chitosan	Activated charcoal	Chitosan membrane modified
N-CH <sub>2</sub> ( <i>stretching</i> )	2362	2636	2350
C=O <i>primary amide</i> ( <i>stretching</i> )	1617	1634	1647
N-H <i>secondary amide</i> ( <i>bending</i> )	1324	1438	-
C-N <i>aliphatic</i> ( <i>stretching</i> )	-	1229	1241
C-O-C ( <i>bending</i> )	1056	-	1020

Meanwhile, the wave number is 1324 cm<sup>-1</sup> and 1436 cm<sup>-1</sup> is the N-H secondary bond functional group *bending* which is a bond in the secondary chain or *secondary amide*. The presence of N-H and C=O absorption indicates that polyurethane has been formed (Marlina et al., 2017). C-N functional group *aliphatic* (*stretching*) clusters formed at wave number 1229 cm<sup>-1</sup> and 1241 cm<sup>-1</sup> and the C-O-C functional group is found at the wave number of 1056 cm<sup>-1</sup> and 1020 cm<sup>-1</sup>. The addition of PEG gives a slight difference in absorption, the absorption is quite sharp in the 1095.57 cm<sup>-1</sup>, which indicates the presence of a C-O-C group from PEG which is bound 26 to chitosan (Nurratri et al., 2020). The shift of wave numbers that occurs is still in the same range of functional groups so that differences in these wave numbers do not result in different interpretations of functional groups. The functional group that causes the adsorbent properties of the oil.

### 2) Membrane Morphology

Figure 2 depicts the surface morphology structure, which consists of a distribution of homogeneous dispersed grains and agglomeration of chitosan constituent molecules. Because of the interaction between porous activated pine charcoal and porous chitosan, the pine charcoal chitosan membrane has a more porous membrane structure with a greater number of membrane pores. The chitosan membrane had a grain size of 17.2 nm, whereas the pine charcoal chitosan had a grain size of 9.27 nm. The entry of -NH<sub>3</sub> ions into the pores causes changes in the surface structure of the membrane, which can facilitate particle contact in the membrane. The contact between the particles causes the membrane to become more flexible, stable in size and dimension, and to have free moving cations or anions that function as charge carriers and are not hygroscopic. The resulting membrane is thinner, less stiff, and does not easily absorb water.

### 3) Porosity test

Figure 2 depicts the porosity value trend of the chitosan-pine charcoal composite membrane. The pine charcoal chitosan membrane has a high porosity of 27%. This is due to the increased porousness of the resulting membrane. Porosity decreased to 57.2% chitosan mass and increased significantly when 20% pine charcoal was added. This decrease can be attributed to the membrane becoming denser and less porous. While this sharp increase is influenced by the intrinsic properties of pine charcoal, which is a material with large pores, a low concentration of chitosan means

more solvent, resulting in the formation of more and more pores. The trend of the porosity value of the chitosan-pine charcoal membrane can be seen in Figure 1. The porosity of the pine charcoal chitosan membrane of 23% has a high value. This is because the resulting membrane is more porous. Porosity decreased to 35.1% chitosan mass and increased sharply with the addition of 15% pine charcoal. This decrease can occur because the membrane is getting denser and less porous. While this sharp increase is influenced by the intrinsic properties of pine charcoal which is a material that has large pores and a small concentration of chitosan means more solvent so that more and more pores are formed.

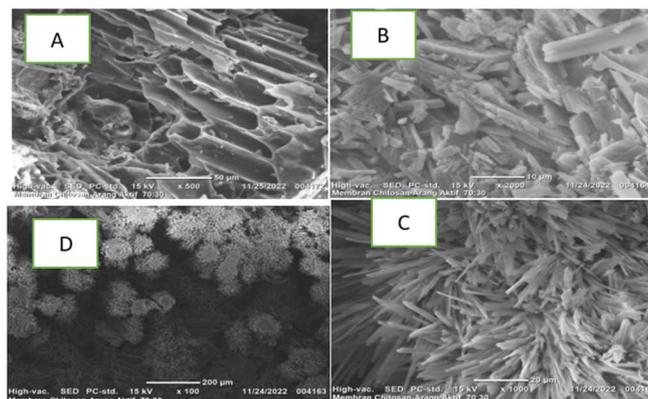


Fig. 2 Membrane morphology

### 4) Testing of Chitosan Membrane Adsorption Capability on Used Cooking Oil

The cooking oil used in this study is one that has been continuously used for frying various types of ingredients. The home industry provides used cooking oil.

### 5) Levels of Free Fatty Acids

Table 4 shows the results of the test for free fatty acid levels in purified used cooking oil.

TABLE IV  
FREE FATTY ACID LEVELS OF REFINED USED COOKING OIL

Membrane	Free fatty acid levels
No membrane	4,608×10 <sup>-4</sup>
K1	1,0752×10 <sup>-3</sup>
K2	1,792×10 <sup>-4</sup>
K3	2,8432×10 <sup>-3</sup>

In this study, the test for fatty acid levels was carried out by titrating the NaOH solution on the used cooking oil samples using various membranes variation. Table 3 shows the test results, which show that the ALB level of used cooking oil before purification using a membrane was 0.46% and the free fatty acid content of used cooking oil after refining was 0.018% using a K2 membrane. ALB levels of used cooking oil decreased by 0.352% after purification using K1 membrane, and the lowest ALB levels were after purification using K3 membrane, with free fatty acid levels of 0.248% and a large decrease in ALB levels for used cooking oil by 0.212%. When compared to other membrane purification methods, purification of used cooking oil using a K2 membrane results in the greatest reduction in ALB concentration levels.

The concentration of the modified membrane with the concentration of chitosan and the mass of activated carbon as an adsorbent affects the ALB content in used cooking oil, namely, the higher the concentration of the membrane modified with chitosan and activated pinecone charcoal, the lower the ALB level in used cooking oil after adsorption. This is because the more optimal the membrane composition, the more stable and active centers of the adsorbent react with the ALB content in used cooking oil, resulting in quite effective interactions and the occurrence of very effective interactions between the active sites on the adsorbent and the adsorbate.

The smaller the adsorbent, the lower the ALB content in used cooking oil after adsorption. This is because the smaller the adsorbent used, the greater the surface area, resulting in a highly effective interaction between the active side of the adsorbent and the adsorbate. This is also consistent with research (Robiah et al., 2018), which states that adsorption efficiency is a function of adsorbent surface area. The greater the surface area of an adsorbent, the greater its capacity for adsorbing an adsorbate.

#### 6) Moisture Content of Refined Used Cooking Oil

Data on the results of the water content test for refined used cooking oil can be seen in Table 5 below.

TABLE IV  
WATER CONTENT OF USED COOKING OIL AS A RESULT OF REFINING

Concentration	Initial weight (M1) (g)	Final weight (M2) (g)	% Water level
K1	4,75	4,26	0,10
K2	4,45	4,17	0,06
K3	5,3	4,99	0,05

Water content analysis reveals the amount of water in the oil and provides an indication of its quality. The higher the water content of the oil, the easier it is to hydrolyze it to produce free fatty acids and glycerol. The water content is calculated by subtracting the weight of the oil before the water in the oil evaporates from the weight of the oil after the water in the oil evaporates. Table 4 shows that the water content of used cooking oil produced by membrane purification K1, 0.10% of that produced by membrane purification K2, and 0.15% of that produced by membrane purification K3. This value satisfies the SNI requirements. The concentration of the modified membrane with the mass composition of activated carbon as an adsorbent affects the absorption capacity.

The ability of chitosan in adsorbing is because it has a large molecular mass so it has a large absorption power and is non-toxic because chitosan has amino acid groups and hydroxyl groups. Chitosan is a natural biopolymer which is polycationic so that it can be applied in various fields such as metal adsorbents, textile dye absorbents, cosmetic and antibacterial ingredients. As a liquid waste processing material, chitosan is able to bind the heavy metal Pb. Chitosan is alkaline because it contains 2 amine groups (Fig. 3 and fig. 4) each repeating unit can bind free fatty acids through acid-base bonds, [13] capable of binding the heavy metal Pb. The adsorption capacity of the membrane cross-

links to ions Pb metal In the adsorption of 10% chitosan, it was found that the Pb content in the oil decreased to 1.8421 mg/kg. However, it is still above the SNI for palm oil. Meanwhile, in the active charcoal adsorbent, the Pb content decreased to 0.5687 mg/kg, this was still above the SNI for oil. However, the absorption capacity of activated charcoal is better than chitosan.

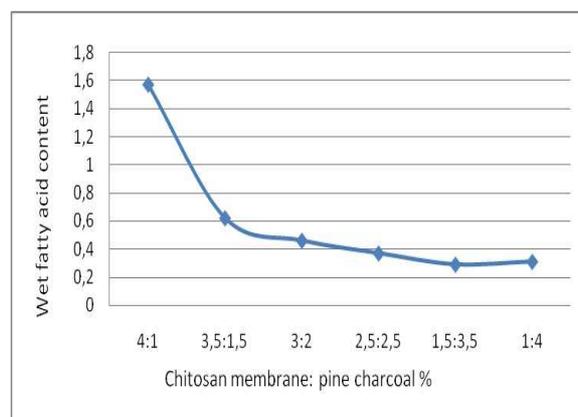


Fig. 3 Decreasing Fatty acid

From the figure 3 can be concluded that the higher the adsorbent concentration, the lower the free fatty acid content and peroxide value in used cooking oil. This happens because many adsorbents come into direct contact with used cooking oil. This is caused by the pores in the adsorbent being able

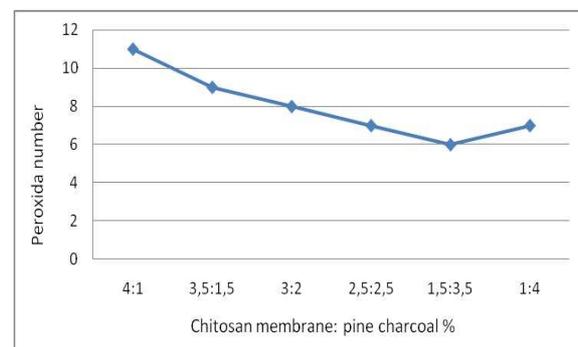


Fig. 4 Decreasing Peroxide number

From these results it can be seen that the concentration of 10% activated charcoal is more effective in adsorbing the Pb content in used cooking oil than 10% chitosan. Concerning the Manufacture and Use of Activated Charcoal has proven the ability of activated charcoal as an adsorbent for Hg, Pb, Cd, Ni, Cu metals in radiator industrial wastewater, nickel plating and copper plating. The ability of activated charcoal as a metal remover is influenced by pH and carbon concentration. Increasing the carbon content increases the percentage of activated charcoal adsorption on metal ions. The use of activated charcoal is very important in the water and air purification process. Meanwhile, chitosan is a natural biopolymer that is polycationic so that it can be applied in various fields such as metal adsorbents, textile dye absorbents, cosmetic and antibacterial ingredients. As a liquid waste processing material, chitosan is able to bind the heavy metal Pb [16]-[22].

#### IV. CONCLUSION

Based on the research that has been done, it can be concluded that the results of the stabilization test of chitosan membrane modified with activated pinecone charcoal are as follows: The best morphology is found at a membrane concentration of 7 : 3 with an elastic, smooth and flat surface, Fourier Transform Infrared (FTIR) analysis revealed that NCH<sub>2</sub> is taken up by chitosan at a wave number of 2362 cm<sup>-1</sup>, activated pinecone charcoal at a wave number of 2636 cm<sup>-1</sup>, and the membrane at a wave number of 2350 cm<sup>-1</sup>. The double group (C=O) can be found at wave numbers of 1617 cm<sup>-1</sup>, 1634 cm<sup>-1</sup>, and 1647 cm<sup>-1</sup>, and is the main group of amide or primary amide, The chemical resistance test on the membrane reveals that it is resistant to salt but not to acids and bases, resulting in an average weight loss of 45-55%, The free fatty acid content of used cooking oil purified using a membrane resulted in the greatest decrease in ALB levels, which was 0.442% using a K2 membrane. The tensile strength of the membrane at break, which is 2.25 and high porosity pine charcoal chitosan membrane has a 27%. The concentration of active charcoal in the membrane affects the water content of used cooking oil as a result of purification, the more active charcoal concentration in the membrane, the more water content absorbed by the membrane due to the adsorbent properties of the activated charcoal.

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