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Synthesis and Characterization of Banana Frond-Nanocytosan Bioadsorbent

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ABSTRACT

FTIR characterization of the activated carbon showed the presence of functional groups including –OH (3207 cm^{-1}), aliphatic C–H (2886–2837 cm^{-1}), C≡C or CO_2 (2352–2120 cm^{-1}), aromatic C=C (1592 cm^{-1}), C–O (1086 cm^{-1}), and metal–O (533 and 465 cm^{-1}). The FTIR spectrum of chitosan indicated the presence of –OH and –NH groups (3641 cm^{-1}), $–COO^-$ (1411 cm^{-1}), and glucose ring structures (870 and 711 cm the optimum result was achieved with 1% nanochitosan-coated adsorbent at a mass of 2,5 g and a contact time of 30 minutes, resulting in an FFA reduction of 2.51%. The nanochitosan-coated activated carbon showed the highest adsorption efficiency at 73.74%

Keywords: *Banana Frond, Citosan, Carbon, Adsorbent.*

1. INTRODUCTION

Cellulose, which is known to be abundant in banana pseudostems, can be used as an adsorbent to absorb free fatty acids on CPO. This pseudostem has a lot of cellulose and is plentiful in quantity. Its chemical composition is as follows: water (10-15%), lignin (5-10%), cellulose (60-65%), and hemicellulose (6-8%)[14]. Even though various pre-treatment methods have been used, with or without burning, banana leaves have been widely studied as adsorbents and have shown good performance. Generally, agricultural waste materials like this, especially those that have undergone delignification (removal of lignin), possess the best adsorption properties. Due to their naturally high cellulose content, these materials have advantages in the adsorption process. The natural structure of the cellulose-containing material will provide a porous structure and have many OH groups. One of the quite abundant results is green mussels, whose production continues to increase every year. This increase in production contributes to the amount of waste, considering that only about 30% of the total weight of green mussels consists of meat that can be consumed[15]. The remaining biomass is in the form of shells, which contain chitin—a substance that can be utilized as a raw material for the production of chitosan [1]

Nano chitosan is a nano-sized particle derived from chitosan that has higher absorption capacity and antibacterial and antifungal effectiveness compared to regular chitosan. The use of chitosan in nano form continues to develop,

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because nano chitosan has better solubility and antifungal activity. In addition, chitosan also has advantages as a biodegradable material, biologically compatible, and non-toxic. The antibacterial activity of chitosan can be enhanced through physical engineering using nano technology, resulting in nano-chitosan [2]. According to [3], the ionic gelation method is the easiest and most commonly used way to produce chitosan nanoparticles. This method alters the physical structure of chitosan, which affects its absorption capacity. High temperature (400–600°C) is the standard in many studies to facilitate the comparison of results. Although materials such as banana pseudostems are relatively easy to carbonize, this temperature ensures that the carbonization process runs optimally and uniformly. Cellulose and hemicellulose begin to break down at temperatures of 200–300°C, but lignin, which is more complex and stable, requires higher temperatures for complete decomposition (up to 400–500°C)[4]

The ability of activated carbon to absorb can be improved by modifying its morphology, such as through variations in temperature, time, and treatment with H_2SO_4. This results in an increase in the levels of carboxylic acids and phenols, as well as a reduction in lactone and total base compounds. The activated carbon before modification has small pores, is dense, and has no layers, while after modification, its pores become larger and more visible [5]

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

The materials used in this study are: banana fronds, green shellfish shells, crude palm oil, hydrochloric acid, sodium hydroxide 3.5%, sodium hydroxide 60%, sodium tripolyphosphate 1%, acetic acid, distilled water (aquadest), universal pH indicator paper. The tools used are: measuring glass, grinder, furnace, electric oven, analytical balance, measuring flask, burette, stand and clamp, stopwatch, centrifuge, dropper pipette, volumetric pipette, centrifuge tubes, stirring rod, filter paper, 200 mesh sieve, hot plate, magnetic stirrer, Fourier Transform InfraRed (FTIR) Spectrophotometer, Particle Size Analyzer (PSA).

2.2. Research Procedure.

2.2.1. Carbonization of Banana Fronds

The preparation of banana pronds begins with washing them using aquades to remove the attached dirt, and then they are dried under sunlight until their moisture content decreases. After drying, the pronds are cut into smaller sizes and subsequently carbonized in a furnace at a temperature of 400°C. The carbonization results in a powder, which is then placed into a 500 ml BOD bottle[9]

2.2.2. Carbon Activation Process

Carbon is then activated with H_2SO_4 in a 1:10 ratio. Activation is carried out for 24 hours at room temperature. After that, the activated adsorbent is washed with distilled water until neutral pH. The adsorbent is then dried in an oven at 100°C for 3 hours. The final process involves sieving to obtain a uniform particle size of the adsorbent[13]

2.2.3. Isolation of Chitosan from green shellfish shell

Preparation of green shellfish shell

The green shells harvested from the Belawan Harbor area were first washed using flowing water to remove adhering dirt. After that, the shells were sun-dried until completely dry. The dried shells were then crushed into small pieces, then ground using a grinder to obtain a fine powder. Next, the resulting powder was sifted using a sieve with a mesh size of 200 to obtain uniformly sized particles.

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Deproteination

Prepare 70 g of green ceramic shell powder, then put it into three lidded containers. Add a 3.5% NaOH solution at a ratio of 1:10. Heat at 70°C for 2 hours while stirring. Then the mixture is filtered and rinsed with aquades until a neutral pH is achieved. The solid obtained is then dried in an oven at 90°C for 2 hours[6]

Demineralization

Deproteinized powder were placed into a beaker three and then added with 1N HCl using a ratio of 1:10. Then, let it sit at room temperature for soaking, and then heat at 75°C for 1 hour while stirring. Then the mixture is filtered and rinsed with aquades until neutral pH. The solids obtained are then heated at 60°C for 2 hours. Chitin will be obtained from the green crab shell.

Deacetylation

10 g of chitin powder is dissolved with a 60% NaOH solution, then placed in a Becker at a temperature of 90°C for 3 hours while stirring. Filter and rinse with distilled water until neutral pH. The obtained solid is then dried at 60°C for 2 hours.

2.2.4. Making Nano Chitosan Ionic Gelation Method

Dissolve chitosan 0.2% in acetic acid solution (CH3COOH). Add 1% NaTPP solution with a ratio of chitosan solution: NaTPP solution of 5:1 with a stirring speed of 400 rpm, and the stirring is done for 1 hour[7]

2.2.5. Provision of Nano Chitosan Coated Activated Carbon

Activated carbon as much as 10 g was added to 100 mL of 0.2% chitosan solution, then stirred and dried at room temperature for 3 days. Then repeat the same steps with variations in chitosan concentrations of 0.4%, 0.6%, 0.8%, and 1.0%.

2.2.6 Provision of Crude Palm Oil

As much as 165 g of crude palm oil is heated at a temperature of 70 °C until it melts with a stirring speed of 250 rpm for 10 minutes. Add 10 mL of neutral 96% ethanol. The mixture is heated on a hot plate while stirring with an attractive stirrer until the solution becomes homogeneous, then add 2-3 drops of 1% PP indicator and titrate with 0.1 N KOH until the color changes to light red as the endpoint of the titration[8]

2.2 7 Characterization using FTIR

Characterization using FTIR was conducted to identify the functional groups present in the carbon, activated carbon, and activated carbon coated with nanokitosan.

3. RESULTS AND DISCUSSION

3.1. Carbonization of Banana Fronds

After the banana fronds are dried, grinding is carried out until fine using a grinder. Once the banana fronds are finely ground, a carbonization process is performed on the banana Fronds. This carbonization process aims to convert organic materials (such as cellulose, hemicellulose, and lignin) into carbon (char) with a more stable structure, enhancing activation efficiency. The carbonization process is conducted at a temperature of 400°C for 30 minutes, aiming to remove volatile substances and transform the banana frond material into carbon. The progress of the

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carbonization process can be indicated by the amount of smoke produced, which signifies the evaporation of organic components in the material.

3.2. Activation of Banana Frond Activated Carbon

The carbon that has been produced is then activated by soaking it in a 3 M H_2SO_4 solution while being stirred and left for 24 hours. The activation process aims to remove contaminants, separate cellulose from lignin, open pores that are still closed in the carbon structure, attract interfering compounds, and enlarge the pore structure by facilitating the formation of new pores in the appearance. Sulfuric acid is chosen as an activator because the use of H_2SO_4 as a chemical activation agent can develop a porous structure by deconstructing the amorphous domains of cellulose and aromatizing the carbon framework[12]

3.3. Isolation of chitosan from Green Shellfish Shell

The waste of green crab shells is washed using flowing water to remove impurities that stick to the crab shells, such as soil, dust, and other pollutants. After being washed, the green crab shells are crushed. Then, the crushed green crab shells are ground using a grinder until they become finer particles. After grinding, the crab shell particles are sieved using a 200mesh sieve.

3.4. Characterization with FTIR

3.4.1 Characterization Active Carbon of Banana Fronds

Results of FTIR spectrum analysis. The FTIR spectrum of banana peel is shown in image below.

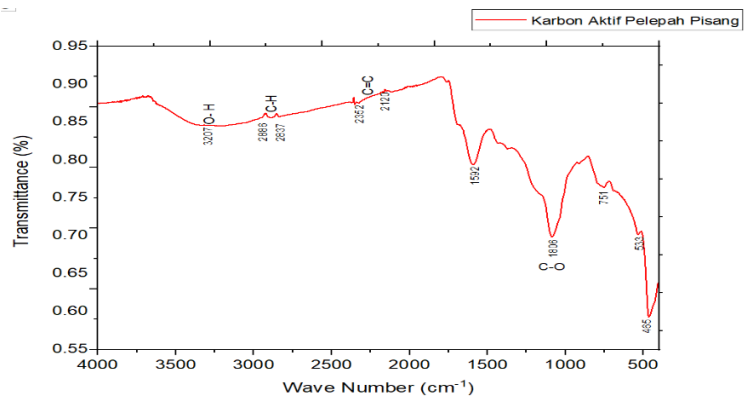

Figure 1. FTIR Spectrum of Active Carbon

The analysis of the FTIR spectrum of banana pseudostem carbon shows that after the activation process, the FTIR spectrum of the carbon exhibits significant differences. The intensity of the peaks in the range of 3600–3900 cm^{-1} decreases drastically, leaving only one dominant peak at 3207 cm^{-1} that still indicates the –OH group but in smaller amounts. This decrease reflects the dehydration and decomposition of polar groups during carbonization. The peaks at 2886 cm^{-1} and 2837 cm^{-1} still show the presence of aliphatic C–H, although with lower intensity, indicating partial degradation of the aliphatic structure. The peaks at 2352 cm^{-1} and 2120 cm^{-1} can be associated with C≡C groups or the possible adsorption of CO_2 on the surface of activated carbon.

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3.4.2 Characterization of Chitosan

The FTIR spectrum of chitosan extracted from green shellfish shells is shown in image below.

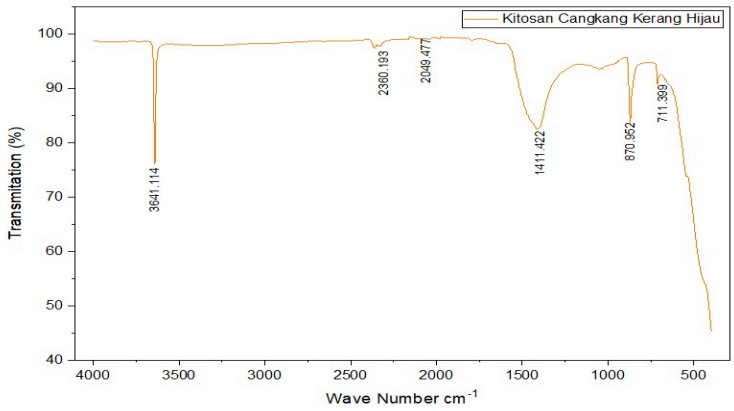

Figure 2. FTIR Spectrum of Chitosan

Table 1. chitosan functional group

Functional Group	Wavelength Range cm^{-1}	Wavelength Number cm^{-1}
N-H stretching	3000-3750	3641,11
OH	3200-3750	
C-H bending	1350-1480	1411,42
C-O-C	1000-1500	1050.42

In the FTIR spectrum of chitosan shows several peaks at different wave numbers, indicating the presence of different functional groups. At a wave number of 3641.11 cm^{-1}, this peak indicates the stretching of –OH (hydroxyl) groups overlapping with the stretching of –NH (secondary amine), which is characteristic of chitosan as a polymer that has many polar groups. The wavenumber 1411.42 cm^{-1} indicates the presence of bending vibrations of -CH as well as contributions from the $-COO^-$ groups (carboxylate), which may indicate the presence of a partial deacetylation process in chitosan derived from chitin.

The FTIR spectrum obtained is in accordance with the characteristic spectrum of chitosan. This confirms that the isolate obtained from the deacetylation process of chitin successfully been converted into chitosan. The presence of $–NH_2$ and –OH groups also indicates the hydrophilic nature and potential biological activity of chitosan.

3.5. Nanochitosan preparation and characterization with PSA

Nanokitosan is a nano-structured material derived from chitosan. The preparation process begins by dissolving 0.2% chitosan into an acetic acid (CH_3COOH) solution, as chitosan, being a polysaccharide, is not soluble in water. This dissolution stage results in a yellowish chitosan solution with slight viscosity, indicating that the chitosan has been completely dissolved in acetic acid. Subsequently, 100 mL of the dissolved chitosan solution is mixed with 20 mL of

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1% NaTPP solution, and then stirred using a magnetic stirrer at a speed of 400 rpm for one hour. Chitosan that has been made into nano size is then tested with Particle Size Analyzer (PSA) aimed at observing the size of particles in chitosan nanoparticles, which is shown in image below.

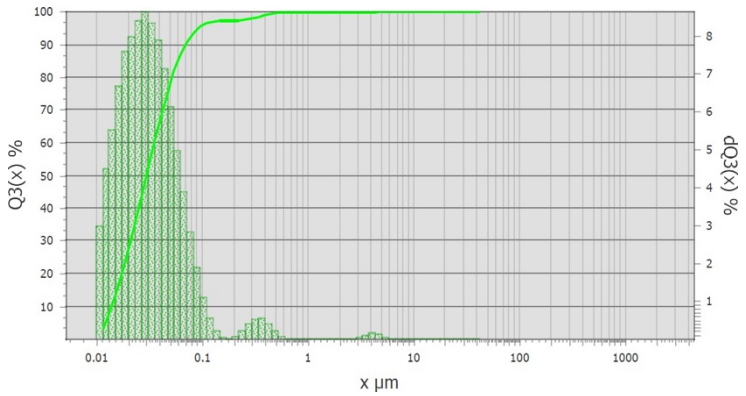

Figure 3. PSA Spectrum of Nanochitosan

The results of the particle size measurement using the PSA (Particle Size Analyzer) show that the particle size is 0.06720 µm or equivalent to 67.20 nm, indicating that the particles produced are within the range of nanoparticle sizes, which is between 1-100 nm.

3.6. Activated Carbon Coating with nanochitosan from green shellfish shells

To determine the optimal concentration of chitosan in coating activated carbon on the adsorbent, variations of chitosan concentrations of 0.2%, 0.4%, 0.6%, 0.8%, and 1% were used. As much as 2.5 grams of adsorbent with each concentration of chitosan was then mixed with 50 mL of previously melted CPO oil and stirred using a magnetic stirrer for 30 minutes. The goal was to accelerate the adsorption process. Then the oil that had been mixed with the chitosan-coated adsorbent with various variations was placed into a centrifuge tube to separate the oil from the remaining solid adsorbent. The oil that has been separated is then subjected to titration to determine the amount of free fatty acids (FFA) contained in crude palm oil (CPO). The free fatty acid content decreased from 9.56% to 2.51% after 30 minutes of treatment.

Adsorption involves a unique isotherm pattern, as it is influenced by several variables including the properties of the adsorbate, its concentration, the concentration of the adsorbent, the type of adsorbent, its surface area, as well as the temperature and duration of contact between the adsorbent and the substance. The adsorption isotherm describes how molecules are distributed between the liquid phase (adsorbate) and the solid phase (adsorbent) during the adsorption process[10]

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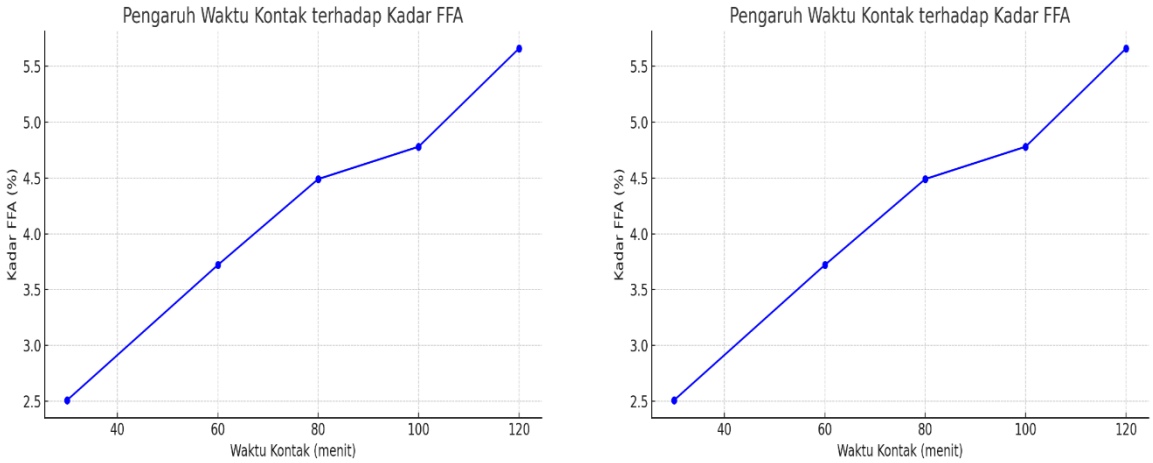

Figure 4. FFA Adsorption with Variations in Mass and Contact Time

At a mass of 2.5 grams, the FFA content further decreased to around 2.5%, indicating a higher adsorption efficiency at the maximum adsorbent mass. At a contact time of 30 minutes, the lowest FFA content recorded was 2.5%, and it gradually increased to 5.6% at a contact time of 120 minutes, indicating that the FFA adsorption process is most effective at shorter contact times.

4. CONCLUSION

Activated carbon from banana peels shows hydroxyl groups (–OH) at 3207 cm^{-1}, crucial for adsorbing Free Fatty Acids (FFA) in crude palm oil (CPO). Chitosan shows N-H groups at 3641.11 cm^{-1} and strong C-H groups at 1411 cm^{-1}. Nanokitosan has an average particle size of 67.20 nm. Using banana peel-based activated carbon in a 1% chitosan solution (2.5 g, 30 minutes), FFA levels in CPO decreased from 9.56% to 2.51%, achieving a 73.74% adsorption rate, showing effective FFA removal under these optimal conditions.

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