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Preparation of Banana Peel Activated Carbon Coated with Nanochitosan from Shrimp shells

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ABSTRACT

Banana peel, an agricultural waste, contains valuable compounds suitable for use as an adsorbent to remove hazardous dyes and heavy metals from the environment. Activated carbon was prepared from banana peel using HCl as an activating agent. Chitosan was synthesized from shrimp shells through deproteinization, demineralization, and deacetylation, followed by characterization using FTIR. Nanochitosan was produced via ionic gelation and characterized using Particle Size Analyzer (PSA). The resulting nanochitosan was successfully coated onto activated carbon. FTIR analysis showed shifts in absorption intensity in both carbon and activated carbon, indicating successful activation and functional modification. The presence of chitosan was confirmed by the identification of N–H functional groups at 3248 cm^{-1}. PSA results showed that the synthesized nanochitosan had a particle size of approximately 86.14 nm. These findings demonstrate the successful synthesis of activated carbon coated with nanochitosan, with potential applications in environmental remediation through improved adsorption performance.

Keywords: banana peels, shrimp shells, activated carbon, chitosan, adsorbent

1. INTRODUCTION

Banana (*Musa paradisiaca*) ranks among the most commonly consumed fruits in Indonesia, either in its fresh form or as processed food products. In the food processing industry, banana peels constitute the primary waste generated. Generally, the utilization of banana peels in the community remains limited, commonly being used as animal feed. A significant portion of banana peels is discarded without being utilized effectively[2]. In fact, banana peels contain several valuable components that hold potential as raw materials for adsorbents in the removal of reactive dyes from the environment. These components include elements such as nitrogen and sulfur, as well as organic compounds like cellulose, carboxylic acids, chlorophyll pigments, hemicellulose, and pectic substances containing galacturonic acid, arabinose, galactose, and rhamnose[5]. Which contribute to their potential application as effective adsorbent materials.

Chitosan, which has the structure [β-(1→4)-2-amino-2-deoxy-D-glucose], is a derivative of chitin obtained through the deacetylation process. Chitosan is classified as a positively charged (polycationic) polymer. The presence of amino and hydroxyl groups distributed along its polymeric backbone renders chitosan highly effective in adsorbing heavy metal ions as well as cationic organic compounds such as proteins and lipids. The adsorption mechanism involves the formation of coordination complexes, in which nitrogen atoms from amino groups and oxygen atoms from hydroxyl groups participate in binding[10].

Chitosan is predominantly derived from crustacean waste, particularly shrimp shells. Shrimp shells represent one of the most abundant aquatic wastes that are often improperly disposed of, despite their rich composition of approximately 25–40% protein, 45–50% calcium carbonate, and 15–20% chitin[3]. To utilize shrimp shell waste that is commonly discarded, the chitin content can be extracted and converted into chitosan, which can then be applied as an adsorbent for dye removal. One of the key factors influencing the adsorption efficiency of chitosan is its particle size. Smaller chitosan particles possess a larger surface area, thereby increasing the contact area with target adsorbates and enhancing adsorption capacity. Therefore, modification of chitosan into nanoparticle form is necessary to improve its adsorption performance.

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

The materials used in this study are: banana peel, shrimp shells, 1M hydrochloric acid (HCl), 3.5% sodium hydroxide (NaOH), 60% sodium hydroxide (NaOH), 1% acetic acid (CH3COOH), 1% sodium tripolyphosphate solution (NaTPP), distilled water (Aquadest), whatman 1 filter paper, and universal pH indicator. The tools used in this study are: analytical balance, watch glass, chemical beaker, Porselin cup, measuring cup, glass funnel, Erlenmeyer flask, measuring flask, furnace, stir bar, vacuum pump, oven, 100 mesh sieve, hot plate, magnetic stirrer, Fourier Transform InfraRed (FTIR; variant Invenio-S and Particle Size Analyzer (PSA; variant Fritsch Analysette 22 NanoTec).

2.2. Research Procedure

2.2.1 Carbonization of Banana Peel

First, banana peels are thoroughly washed with distilled water to remove any impurities. Then, the banana peels were cut into small pieces, roughly 1-2 cm in size, and sun-dried for two days. Once dry, the banana peels are carbonized at 400˚C for about 1.5 hours. After carbonization, the banana peels carbon are ground into a fine powder to ensure uniform particle size. Finally, this powder is sieved through a 100-mesh.

2.2.2 Carbon Activation Process

A total of 50 grams of sieved banana peel carbon was activated using 1 M hydrochloric acid (HCl) with a ratio of 1:5 (w/v). The mixture was stirred for 30 minutes and then allowed to stand for 24 hours. After that, the carbon is filtered using filter paper and then rinsed with distilled water until the pH is neutral. The produces activated carbon was dried in an oven at 105°C for 2 hours and was characterized with FTIR.

2.2.3 Isolation Chitosan from Shrimp Shells

Preparation of Shrimps Shells

For the preparation of shrimp shells, they were first cleaned and rinsed with distilled water, then sun-dried for 48 hours. The dried shells were subsequently ground into powder and sieved using a 100-mesh screen.

Isolation of Chitosan from Shrimp Shells

Deproteinization

70 grams sample of shrimp shell powder was added to a 3.5% (w/v) NaOH solution at a 1:10 (w/w) ratio. The mixture was then stirred and heated for 2 hours at 70°C. Following this, the residue was filtered using filter paper and rinsed with deionized water until a neutral pH was achieved. The purified residue was subsequently dried at 60°C until it dried.

Demineralization

The deproteinized shrimp shell powder was combined with 1 M HCl at a 1:10 (w/v) ratio. The mixture was then heated at 75°C for 1 hour with continuous stirring. Subsequently, the resulting mixture was filtered and rinsed with deionized water until a neutral pH was achieved. The obtained solid was then dried in an oven at 60°*C*.

Deacetylation

For the deacetylation step, 10 grams of chitin powder were placed into a beaker, followed by the addition of 60% (w/v) NaOH solution at a 1:10 (w/v) ratio. The mixture was then heated at 100°C for 2 hours with continuous stirring. The resulting solid was subsequently filtered and rinsed with distilled water until a neutral pH was achieved. Finally, the solid was dried in an oven at 65°C. The resulting chitosan was characterized by Fourier Transform Infrared (FTIR).

2.2.4 Synthesis of Nanochitosan with Ionic Gelation Method

A chitosan solution was prepared by dissolving 0.2 grams of chitosan in 100 mL of 1% (v/v) acetic acid (CH_3COOH). Subsequently, a 1% (w/v) sodium tripolyphosphate (NaTPP) solution was added to the chitosan solution at a ratio of 5:1 (chitosan solution : NaTPP solution). The mixture was continuously stirred at 400 rpm for 1 hour. The resulting nanochitosan was characterized by Particle Size Analyzer (PSA).

2.2.5 Preparation of Activated Carbon Coated with Nanochitosan from Shrimp Shells

10 grams of activated carbon was added to 100 mL of a 0.2% (w/v) nanochitosan solution. The mixture was then thoroughly stirred until homogeneous and subsequently air-dried at room temperature for 3 days.

3. RESULTS AND DISCUSSION

3.1 Carbonization of Banana Peels

Banana peels were carbonized at 400°C for 1.5 hours to produce banana peel carbon. During the carbonization process, hydrocarbon compounds such as cellulose, hemicellulose, and lignin, along with volatile components, decompose within the banana peels. This decomposition is evidenced by the emission of smoke throughout the carbonization, which in turn leads to the formation of carbon pores.

3.2 Activation of Banana Peels Carbon

The purpose of carbon activation is to enlarge the pore size, increase the pore volume, and create new pores, thereby enhancing the performance of activated carbon in the adsorption process. Chemical activation was carried out using hydrochloric acid (HCl) as the activating agent. HCl was chosen due to its superior ability to dissolve impurities, resulting in the formation of more pores and improving the adsorption efficiency compared to other acid-based activators[4].

3.3 Characterization of Carbon and Activated Carbon Using FTIR

Carbon and activated carbon derived from banana peel was characterized for its functional groups using Fourier Transform Infrared (FTIR) spectroscopy. As shown in Figure. 1, a shift in the FTIR spectral bands was observed for banana peel-derived carbon before and after activation with 1 M HCl. In the spectrum of carbon, a sharp and broad absorption band at 3248.343 cm^{-1} corresponds to O–H stretching vibrations, indicating the presence of hydroxyl functional groups. An absorption band at 2843.725 cm^{-1} is associated with aliphatic C–H stretching vibrations, while the band at 1587.129 cm^{-1} indicates aromatic C=C stretching. The C–H bending vibration is observed at 1377.696 cm^{-1}, and the band at 1005.846 cm^{-1} corresponds to C–O stretching, likely from ester groups.

Following chemical activation with HCl, the O–H stretching band exhibited a reduction in absorption intensity and a slight shift to 3245.493 cm^{-1}. This decrease is attributed to the ability of HCl to remove surface impurities, including hydroxyl groups, thereby reducing their abundance on the carbon surface[8]. The aliphatic C–H stretching vibration appeared at 2846.574 cm^{-1}. The aromatic C=C band also shifted from 1587.129 cm^{-1} to 1605.650 cm^{-1}, indicating enhanced aromatic character in the activated carbon structure[5]. Furthermore, the C–H bending vibration was detected at 1371.997 cm^{-1}, and a shift of the C–O stretching band to 1082.781 cm^{-1} was observed, confirming structural modifications post-activation.

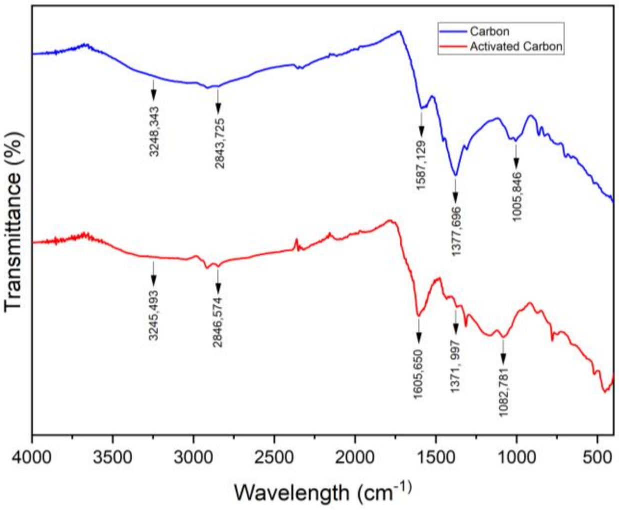

Figure 1. FTIR Spectrum of Carbon and Activated Carbon

3.4 Isolation of Chitosan from Shrimp Shells

3.4.1 Deproteinization

Deproteinization of shrimp shells was carried out to reduce or eliminate the protein content present in the shells. This process involved treatment with a strong alkaline solution, commonly sodium hydroxide (NaOH). The use of NaOH in the deproteinization stage breaks the covalent bonds between proteins and the functional groups of chitin, resulting in the formation of soluble sodium proteinate. Following deproteinization, the resulting solid appeared light brown in color, with a yield of 75.15%.

3.4.2 Demineralization

Following deproteinization, the next step is demineralization, which involves the removal of inorganic mineral salts such as calcium carbonate ($CaCO_3$). In this stage, the deproteinized powder is mixed with 1 M hydrochloric acid (HCl). During the reaction, vigorous bubbling and foaming are observed for approximately 5 minutes. These air bubbles result from the formation of carbon dioxide (CO_2) and water (H_2O) at the surface of the solution. The evolution of CO_2 gas indicates that hydrochloric acid is reacting with the mineral salts present in the shrimp shells, serving as an indicator of successful demineralization. In this process, calcium compounds react with hydrochloric acid to form calcium chloride, which is soluble in water. The resulting product is cream-colored chitin with a yield of 60.66%.

3.4.3 Deacetylation

In the deacetylation stage, the acetyl groups ($-COCH_3$) present in chitin are removed to produce chitosan. This process involves the treatment of chitin with a concentrated alkaline solution, typically sodium hydroxide (NaOH), under elevated temperatures. The conversion of chitin to chitosan is a hydrolysis reaction between a base and an amide, where chitin acts as the amide and NaOH functions as the base. During this reaction, the hydroxide ions (–OH) from NaOH interact with the $NHCOCH_3$ groups, leading to the elimination of CH_3COO^- as a by-product, thereby yielding an amine compound in the form of chitosan[3]. At this stage, shrimp shell-derived chitosan with an off-white to light brown color was obtained, with a yield of 58.90%.

3.5 Characterization of Chitosan Using FTIR

FTIR characterization of chitosan was conducted to identify the presence of amine functional groups. The characterization was shown in the figure below.

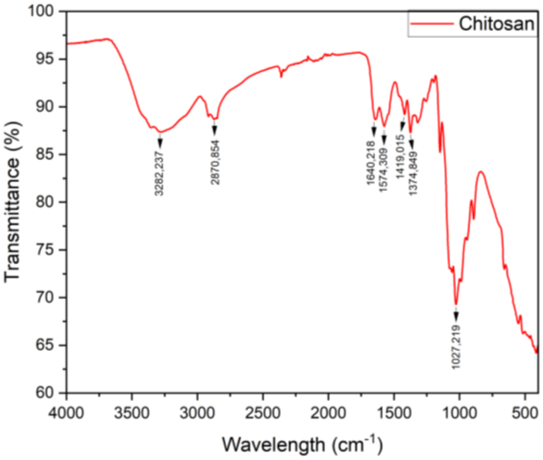

Figure 2. FTIR Spectrum of Chitosan

As shown in Figure 2, absorption bands corresponding to the vibrational modes of various functional groups in chitosan are observed. In the FTIR spectrum of chitosan, a broad peak at 3282.237 cm^{-1} is assigned to the overlapping stretching vibrations of O–H and N–H groups. The absorption band at 2870.804 cm^{-1} indicates the C–H stretching of aliphatic $-CH_2-$ groups, which is reinforced by the C–H bending vibration detected at 1419.015 cm^{-1}. Additionally, the band appearing at 1640.218 cm^{-1} is attributed to C=O stretching. The N–H bending vibration of primary amines ($-NH_2$) is evident at 1574.309 cm^{-1}. Additionally, the band at 1027.219 cm^{-1} is attributed to the C–O stretching vibration within the glycosidic linkage. These FTIR results confirm the presence of characteristic N–H functional groups, indicating that chitosan was successfully synthesized.

3.6 Preparation and Characterization of Nanochitosan from Shrimp Shells

Nanoparticle chitosan (nanokitosan) was prepared using the ionic gelation method. A 1% sodium tripolyphosphate (NaTPP) solution was added to 100 mL of chitosan solution at a ratio of 5:1 (chitosan:NaTPP). NaTPP acts as a cross-linking agent, where its negatively charged ions interact with the amine groups in chitosan, which are protonated by H^+

from the acid to form NH_3^+. This interaction leads to the formation of an intramolecular network structure[9]. Particle size analysis of shrimp shell-derived chitosan nanoparticles was performed using a Particle Size Analyzer (PSA). The particle size distribution profile is presented in Figure 3.

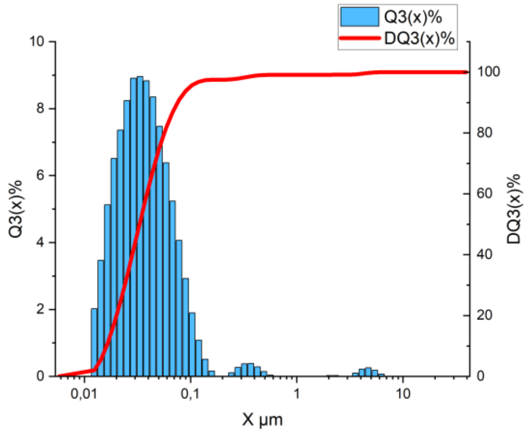

Figure 5. Particle Size Distribution Graph of Shrimp Shell-Nanochitosan

As shown in the figure, the average particle size obtained was 0.08614 µm, equivalent to 86.14 nm. The results of this study indicate that the synthesized chitosan nanoparticles fall within the nanoscale range. A material is classified as a nanoparticle when its particle size ranges from 1 to 100 nm[1].

3.7 Activated Carbon Coated with Nanochitosan

The coating of activated carbon with shrimp shell-nanochitosan was carried out by dispersing 10 grams of banana peel-based activated carbon into a 0.2% nanokitosan solution, followed by continuous stirring until a homogeneous mixture was obtained. This ensured uniform dispersion and optimal coating of nanochitosan particles over the entire surface of the activated carbon. The nanokitosan-coated activated carbon was then dried at room temperature for 3 days. Room temperature drying was employed to prevent structural alterations and quality degradation of the nanokitosan-coated carbon, as nanokitosan is prone to decomposition and aggregation at elevated temperatures, which can reduce the effective surface area and coating efficiency.

4. CONCLUSION

The findings of this study indicate that the synthesis of activated carbon coated with nanochitosan was successfully achieved. This was supported by FTIR analysis, where both carbon and activated carbon exhibited noticeable shifts in absorption intensity after activation with HCl, suggesting structural and functional modifications. The successful identification of the N–H functional group at a wavenumber of 3282.237 cm^{-1}, further confirmed the presence of chitosan. Moreover, particle size analysis (PSA) revealed that the synthesized nanochitosan had a nanoscale particle size of approximately 86.14 nm. Additionally, the banana peel–based activated carbon coated with shrimp shell–derived nanochitosan demonstrated significant potential as an effective adsorbent for the removal of various environmental pollutants.

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