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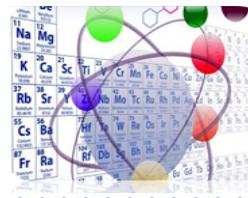
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Preparation and Characterization of Adsorbents from Tea Drains Coated with Nanochitosan From Green Mussels Shells

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ABSTRACT

Tea dregs are abundant organic waste containing high cellulose content, making them a potential raw material for activated carbon production. Chemical activation using NaOH was selected to enhance adsorption capacity. Chitosan was obtained from green mussel shells through demineralization, deproteinization, and deacetylation processes. This study aimed to synthesize an adsorbent based on tea dregs activated carbon coated with nanochitosan derived from green mussel shells. Characterization was conducted using Fourier Transform Infrared Spectroscopy (FTIR) to identify functional groups and Particle Size Analyzer (PSA) to determine chitosan particle size. FTIR analysis indicated decreased absorption intensity of –OH, C=O, C–O, and C–H groups after activation, suggesting removal of organic components and increased carbon purity. The isolated chitosan exhibited NH₂ groups at 1653 cm⁻¹, while PSA results showed an average particle size of 62.08 nm. The resulting composite adsorbent demonstrated promising characteristics for water treatment, particularly in heavy metal adsorption applications.

Keywords: tea waste, activated carbon, green mussel shell, nanocomposite chitosan

1. INTRODUCTION

Used tea leaves (*Camellia sinensis*) are organic waste derived from brewed tea leaves. These residues are often discarded directly into the environment without prior treatment. Tea waste contains approximately 90% cellulose, accounting for about 33.54% of its dry weight. As a result, tea waste is widely utilized by the public as fertilizer, animal feed, natural cosmetic ingredients, and adsorbents due to its low cost, easy availability, and eco-friendly nature.¹ The high cellulose content in tea waste gives it great potential as an adsorbent. The hydroxyl (–OH) groups bound to cellulose can be removed through heating at high temperatures, which eliminates the oxygen and hydrogen atoms and leaves behind carbon atoms at every section of the structure.³

Chitosan, which predominantly contains hydroxyl ($-\text{OH}$) and amine ($-\text{NH}_2$) functional groups, is also an effective adsorbent. These functional groups play a key role in binding heavy metal ions. The adsorption capacity of chitosan is influenced by several factors including salt concentration, pH, and others, all of which can affect its effectiveness in capturing pollutants.¹⁰ Through isolation and further processing into nanostructures, chitosan can be converted into nanokitosan, which has advantages such as a higher specific surface area and greater chemical reactivity compared to conventional chitosan.⁸ Using the ionic gelation technique with sodium tripolyphosphate (TPP), chitosan can be transformed into nanokitosan, characterized by smaller particle size, broader surface area, and higher reactivity. The glucosamine units in chitosan can form bonds with transition metals and 1,4-glycosides due to their resistance to chemical degradation. As a result, chitosan in nano form exhibits more effective responses when applied in adsorption processes.

One type of waste produced by green mussels (*Perna viridis*) is their shells, which are rarely utilized by the public and can potentially cause environmental pollution.¹⁴ Based on the information outlined above, this study aims to conduct the preparation and characterization of a biosorbent derived from used tea leaves coated with nanokitosan synthesized from green mussel shells.⁸

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

The materials used in this study are: tea waste, green mussel shell, 0,1 N sodium hydroxide (NaOH), 1 N hydrochloric acid (HCl), 3.5% sodium hydroxide (NaOH), 60% sodium hydroxide (NaOH), 1% sodium tripolyphosphate solution (NaTPP), 1% M acetic acid (CH₃COOH), synthetic dye remasol yellow, distilled water (Aquadest), 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, 200 mesh sieve, hot plate, magnetic stirrer, Fourier Transform InfraRed (FTIR) Spectrophotometer, Particle Size Analyzer (PSA).

2.2. Research Procedure Natural Adsorbents

2.2.1. Carbonization of Tea Waste

The tea waste was first dried under sunlight, then oven-dried at 110°C for one hour until completely dry. The dried tea waste was then carbonized using a furnace at 300°C for 30 minutes. The resulting charcoal was ground and sieved using a 200-mesh sieve.

2.2.2. Carbon Activation Process

After adding the tea waste carbon to a 0.1 N NaOH solution, the mixture was stirred using a magnetic stirrer for 1 hour. The carbon was then washed with distilled water, separated, and rinsed until a neutral pH was reached. The activated carbon was subsequently dried in an oven at 105°C for one hour.

2.2.3. Isolation of Chitosan from Green Mussel Shell

Preparation of green mussel shell

The collected green mussel shell are washed first using running water to remove any remaining dirt. Next, the green mussel shell are dried in the sun. After that, mash it until it becomes smaller in size. Then grind it using a grinder to produce a fine powder. The green mussel shell are then sifted using a 200 mesh sieve.

Isolation of Chitosan from Crab Shells

Deproteinization

In a 1:10 ratio, 70 grams of green mussel shell powder were added to a 3.5% NaOH solution. The mixture was then stirred while being heated at 70°C for two hours. Afterward, the solid was filtered and rinsed with distilled water until the pH returned to neutral. The solid was then dried in an oven at 60°C.

Demineralization

The deproteinized powder was then placed into a beaker and mixed with 1N HCl at a ratio of 1:10 (w/v). The mixture was left to stand at room temperature for soaking, then heated at 75°C for 1 hour while being continuously stirred. After heating, the mixture was sieved and rinsed with distilled water until a neutral pH was achieved. Finally, the sample was dried in an oven.

Deacetylation

A total of 10 grams of chitin powder was placed into a container, followed by the addition of 60% NaOH solution. The mixture was then heated at 90°C for 3 hours while being stirred. The resulting solid was filtered, rinsed with distilled water until a neutral pH was reached, and subsequently dried in an oven.

2.2.4. Making Nano Chitosan Ionic Gelation Method

A 0,2% chitosan solution was prepared in 1% acetic acid. The solution was then mixed with a 1% NaTPP solution at a chitosan to NaTPP ratio of 5:1, with a stirring speed of 300 rpm. The stirring process was carried out for 1 hour.

2.2.5. Provision of Nano Chitosan Coated Activated Carbon

10g of activated carbon was mixed with 100 mL of 0.2% nanochitosan solution then stirred until smooth, then dried at room temperature for 3 days. Next, mix with nanochitosan concentrations of 0.4%, 0.6%, 0.8%, 1.0% to get the most effective formula.

3. RESULTS AND DISCUSSION

3.1. Carbonization of Tea Waste

Carbonization of the used tea leaves was carried out at 300°C for 30 minutes until black charcoal was obtained. The release of smoke during the carbonization process indicated the evaporation of organic compounds from the tea waste. This smoke signifies the release of volatile compounds, which contributes to the opening of the carbon pores.

3.2. Activation of Tea Waste Activated Carbon

In this study, carbon activation was carried out using a 0.1 N NaOH solution with the aim of improving the pore structure and introducing active functional groups on the surface of the carbon derived from tea waste. Chemical activation with 0.1 N NaOH operates through the penetration of hydroxide ions (OH^-) into the carbon matrix, which can lead to the release of volatile compounds and the opening of pores. This process breaks down weak bonds within the lignocellulosic structure of the tea waste, resulting in activated carbon with a more developed microporous structure. Moreover, activation using an alkaline agent such as NaOH can also introduce functional groups such as OH or Na, which play a role in adsorption interactions.¹³

3.3. Isolation of chitosan from chopped shell

Preparation is done by washing the green mussel shell clean, then drying them in the sun, then grinding them. The crushed shells are then sifted using a 100 mesh sieve. The green mussel shell powder obtained is cream colored.

3.3.1. Deproteinization

Deproteinization was carried out to remove the protein content from green mussel shells. This process was performed using NaOH. The purpose of adding NaOH was to release the proteins in the mussel shells, resulting in the formation of soluble sodium proteinate. The proteins contained in the shells are broken down in the NaOH solution, where they form covalent bonds and allow for the isolation of chitin functional groups. This process produces chitin and amino acids.^{11,12} The deproteinization resulted in a cream-colored solid residue from the green mussel shells with a yield of 92.74%. This indicates that the proteins in the mussel shells were successfully removed and reacted with the NaOH.

3.3.2. Demineralization

Demineralization was carried out by adding 1N HCl at a ratio of 1:10 to the deproteinized solid. The demineralization process yielded a residue with a yield of 60%. This indicates that HCl effectively facilitated the removal of minerals from the green mussel shells, thereby reducing the weight of the reacted deproteinized powder.

3.3.3. Deacetylation

Deacetylation of demineralized chitin was performed by adding concentrated NaOH or a highly concentrated alkaline solution to convert the acetamide groups (NHCOCH_3) in chitin into amine groups (NH_2) in chitosan. The deacetylation cycle was completed by adding NaOH to the chitin powder. The purpose of adding NaOH was to break the carbon bond in the acetyl group attached to the nitrogen atom in chitin, thereby forming amine groups ($-\text{NH}_2$). A hydrolysis reaction occurs during this process.

3.4. Characterization of Carbon and Activated Carbon Using FTIR

Functional group analysis of carbon and activated carbon was carried out using Fourier Transform Infrared (FTIR) spectroscopy. FTIR spectra were used to identify the functional groups present in the carbon

samples before and after activation (activated carbon). In Figure 1, two spectral curves are shown: carbon (black line) and activated carbon (red line). The comparison of these spectra provides information on the chemical structure changes that occurred after the activation process.

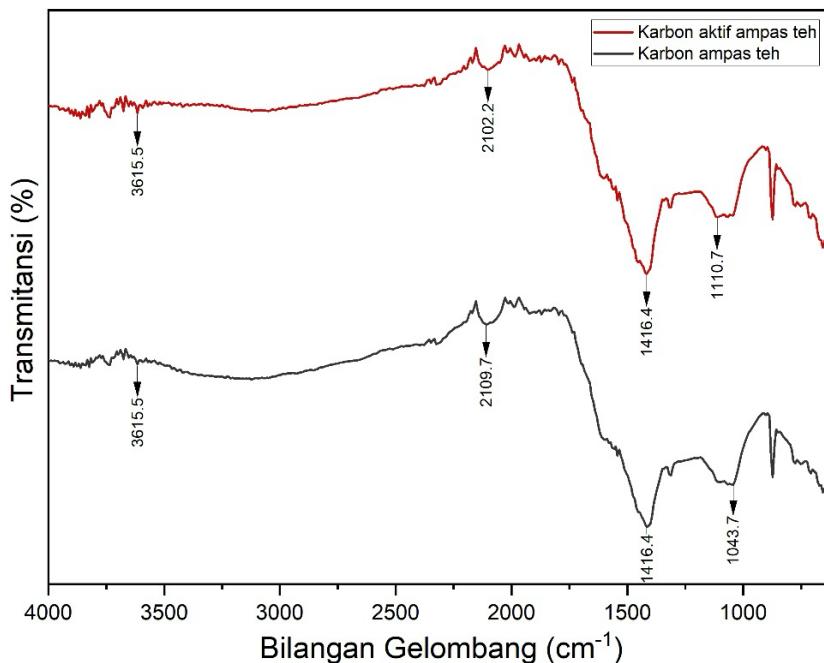


Figure 1. FTIR Spectrum of Carbon and Activated Carbon

Based on the FTIR analysis shown in Figure 1, there are noticeable differences in the absorption bands between tea waste carbon and activated tea waste carbon. In tea waste carbon (black line), a broad absorption band appears at 3615.5 cm^{-1} , which corresponds to the stretching vibration of hydroxyl groups ($-\text{OH}$), commonly derived from phenols, alcohols, or bound water. The absorption peak at 2109.7 cm^{-1} is associated with the stretching vibration of cumulative double bonds such as $-\text{C}\equiv\text{C}-$ or $-\text{C}\equiv\text{N}$. Meanwhile, the band at 1416.4 cm^{-1} indicates the presence of aromatic $-\text{C}=\text{C}$ groups or carboxylate stretching vibrations. In addition, the peak observed at 1043.7 cm^{-1} suggests $\text{C}-\text{O}$ stretching vibrations originating from alcohol, ether, or residual cellulose groups in the tea waste.

After the activation process (red line), shifts and reductions in intensity are observed in several absorption bands. The $-\text{OH}$ peak at 3615.5 cm^{-1} is still present but appears broader, indicating enhanced hydroxyl group activity as a result of activation. The band at 2102.2 cm^{-1} remains relatively unchanged, although its intensity decreases, suggesting partial degradation of organic compounds. The band at 1416.4 cm^{-1} is still visible, confirming the presence of aromatic groups. A significant change is observed at 1110.7 cm^{-1} , where the $\text{C}-\text{O}$ stretching vibration becomes more pronounced compared to the non-activated carbon, indicating the formation of new functional groups due to activation.

The comparison of both spectra demonstrates that activation leads to the removal of certain organic groups and the emergence of more dominant oxygen-containing functional groups ($\text{C}-\text{O}$ and $-\text{OH}$). This indicates that

the activation process enhances the surface area and the number of active sites on the carbon, thereby improving its adsorption potential for heavy metals.⁹

3.5. Chitosan Characterization with FTIR

The degree of deacetylation indicates the purity of the chitosan obtained. A higher degree of deacetylation indicates the purity of the chitosan obtained and will be more optimally utilized in lowering the pH of water. The value of the degree of deacetylation itself can be seen using the FTIR (Fourier Transform Infrared Spectroscopy) characterization test. In FTIR analysis, functional groups are produced in chitosan which can then be determined the degree of deacetylation of chitosan.^{2, 4, 5} The results of the FTIR analysis of green mussel shell chitosan are presented in the figure below:

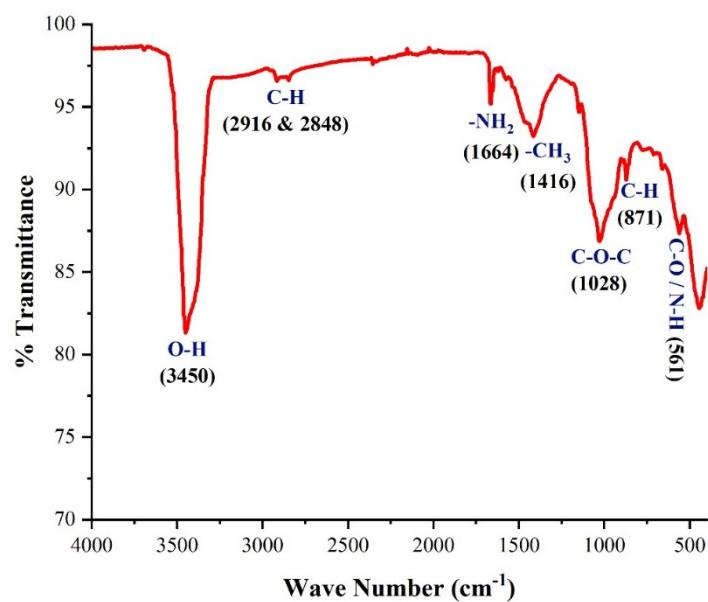


Figure 2. FTIR Spectrum of Chitosan

From the FTIR spectra of chitosan shown above, several absorption bands can be observed indicating the vibrational modes of the functional groups present in the chitosan structure. More detailed information regarding these functional groups can be seen in Table 4.1 below:

Table 1. Chitosan Functional Groups

Functional Group	Wavelength Range (cm ⁻¹)	Wavelength Number (cm ⁻¹)	Information
–OH	3450	3450	Stretching vibrations of hydroxyl and amine groups indicate the presence of polar groups.
C–H stretch (alifatik)	2870–2930	2916,4	Stretching vibrations of methyl and methylene groups in polysaccharide chains
C–H stretch (addition)	2870–2930	2848	Additional C–H stretching vibrations support the presence of aliphatic chains of chitosan.
–NH ₂ buckling	1595–1655	1653	The bending vibration of the primary amine group indicates the success of the deacetylation process.
–CH ₃ bending	1375–1450	1415,9	Symmetrical bending vibration of the methyl group of the glucosamine ring
C–O–C stretching	1000–1150	1029,3	The stretching vibration of glycosidic bonds indicates that the polysaccharide structure is still intact.
β-glikosidik linkage	870–890	871,9	Deformation vibration of the pyranose ring, typical of β(1→4)-glycosidic bonds
C–O / N–H swish	650–750	561,6	The vibration of the polar group is located in the fingerprint region.
Saccharide ring vibrations	400–600	445,4	Complex vibrations of the glucosamine ring, typical of polysaccharides

Based on the FTIR test results of chitosan isolated from green mussel shells, an absorption spectrum was obtained indicating the presence of functional groups typical of chitosan, consistent with literature data. The peak at wavenumber 3450 cm⁻¹ indicates the stretching vibrations of hydroxyl (–OH) and amine (–NH) groups, which are important polar groups in the chitosan structure. These groups play a crucial role in the adsorption process because they can form hydrogen bonds with metal ions.

The peaks at 2916.4 cm⁻¹ and 2848 cm⁻¹ indicate the C–H stretching vibrations of methyl and methylene groups in the aliphatic chain, respectively, strengthening the evidence for the presence of a polysaccharide framework. Meanwhile, the peak at 1653 cm⁻¹ indicates the bending vibrations of primary amines (–NH₂), which is an indicator of the successful deacetylation of chitin to chitosan. These amine groups play an active role in binding metal ions through a complexation mechanism.

The peak at 1415.9 cm⁻¹ indicates the symmetrical bending vibration of the methyl group in the glucosamine ring, indicating that the glucosamine unit in chitosan remains intact. The peak at 1029.3 cm⁻¹

indicates the presence of the C–O–C glycosidic bond, indicating that the primary structure of the polysaccharide was not damaged during extraction. The peak at 871.9 cm^{-1} shows the deformation vibration of the pyranose ring, typical of the $\beta(1\rightarrow4)$ -glycosidic bond, demonstrating that chitosan retains its natural structural form.

Furthermore, the peak at 561.6 cm^{-1} associated with the vibration of the polar group (C–O or N–H) and the peak at 445.4 cm^{-1} of the glucosamine ring further confirm that the chitosan molecular structure remains intact. Overall, the FTIR results demonstrate that the isolation process successfully produced chitosan with active functional groups ready for use as an adsorbent for removing heavy metals from water.

3.6. Nanochitosan preparation and characterization with PSA

Making a 1% nanochitosan solution was carried out by dissolving 0.2 grams of chitosan in 20 mL of 1% acetic acid solution, then stirring using a magnetic stirrer for 30 minutes.¹⁵ The nanochitosan solution was then added with 1% NaTPP solution with a concentration ratio of chitosan solution: NaTPP solution of 5:1 and a stirring speed of 300 rpm. The stirring process was carried out for 1 hours. The process of making nanochitosan is called glass ionic, where the process of forming nanochitosan is based on electrostatic interactions between types of opposite charges containing at least one polymer under mechanical stirring conditions.

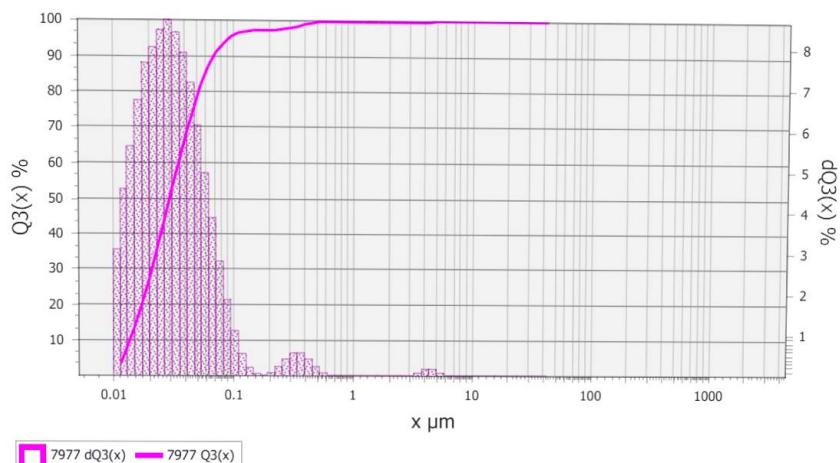


Figure 3. PSA Spectrum of Nanochitosan

In this process, the size of the nanochitosan obtained is $0.06208\mu\text{m}$ or 62.08nm . The longer the stirring time produces smaller particle sizes because more particles are invited and broken down into nano-sized particles. The higher the concentration of acetic acid used, the larger the size of the nanoparticles produced.

3.7. Activated Carbon Coating with nanochitosan from crab shells

10 grams of activated carbon was mixed with 0.2% nanochitosan solution then stirred well and left to dry at room temperature for 3 days. After drying, a tea waste adsorbent was obtained which was coated with green mussel shell nanochitosan. Then it can be repeated with different nanochitosan solution concentrations with variations of 0.4%, 0.6%, 0.8%, 1% to find a better comparison of absorption capacity.^{6,7}

4. CONCLUSION

Based on the results of FTIR analysis, tea dregs carbon and tea dregs activated carbon showed that the activation of tea dregs resulted in a shift and strengthening of the –OH and C–O groups, as well as a reduction in organic compounds. This indicates the formation of more active sites on activated carbon, thus potentially increasing the adsorption capacity. Preparation and isolation of chitosan from green mussel shells were successfully carried out, characterized by the presence of the NH₂ group at a wavelength of 1653 cm⁻¹ based on FTIR analysis as a characteristic of chitosan. The results of PSA analysis showed that the chitosan obtained was nano-sized with a particle diameter of 62.08 nm. Coating tea dregs activated carbon with nanochitosan from green mussel shells produced an adsorbent with suitable characteristics, so it has high potential for use in heavy metal adsorption applications in water treatment.

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