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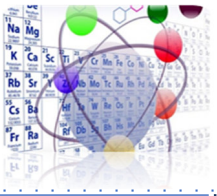

# Adsorbent Preparation From Rice Husks Coated With Nanochitosan From Crab Shells

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**ABSTRACT**

*Rice husks are a waste that is often found, containing 50% cellulose and 25-30% lignin so it has the potential to be used as an absorbent material. Therefore, rice husks can be used to make active carbon. The carbon activation process using HCl is preferred because compared to other acids, carbon activated with HCl shows better adsorption capacity. Chitosan comes from chitin found in crustaceans such as crabs. The crab shells are then processed to obtain chitosan. This research aims to produce rice husk adsorbent coated with nanochitosan from crab shells. Characterization in this study used FTIR which showed the presence of N-H groups as a characteristic of chitosan at a wavelength of 3354.42 cm-1. PSA analysis shows nano-sized chitosan with a size of 58.15 nm. Coating rice husk activated carbon with crab shell chitosan has produced a suitable adsorbent and can be used for further testing..*

Keywords: Rice Husk, Citosan, Carbon, Adsorbent

## 1. INTRODUCTION

Rice husk is a byproduct of rice milling used to produce rice. Indonesia, as one of the world's largest rice producers, generates a significant amount of rice husk. This rice husk is often considered waste because the primary product of rice milling is rice, consumed as a staple food by Indonesian society. However, rice husk has potential for further utilization as a raw material in various applications. For instance, it can be used as an alternative fuel, animal feed, compost material, and activated carbon. Activated carbon is carbon processed to have open pores and high adsorption capacity, achieved through physical or chemical activation methods[9]. Rice husk typically contains 50% cellulose, 25-30% lignin, and 15-20% silica[7], making it a potential absorbent material due to its cellulose and hemicellulose content.



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Chitosan is an aminopolysaccharide derived from the deacetylation of chitin. This compound is a crucial natural biopolymer with polycationic properties, utilized in various applications such as disinfectants, food preservatives, textile dye adsorbents, cosmetics, water purification, and the removal of heavy metals like Cu, Zn, Cd, Pb, Mg, and Fe. The active NH2 group in chitosan, in both NH2 and NH3+ protonated forms, facilitates heavy metal adsorption through complexation or ion exchange. Chitosan's biocompatibility, biodegradability, and non-toxicity make it highly suitable for environmentally friendly industrial applications[5].

Chitin is abundant in marine waste, particularly in crustaceans like shrimp, crab, crayfish, and lobster, with crab shells being a notable source. Ameilia & Haedyastuti[2] noted high mineral content in crab shells, especially calcium (19.97%), phosphorus (1.81%), and chitin (20-30%).Based on the information provided, research will focus on preparing adsorbents from rice husk coated with nanochitosan derived from crab shells.

## 2. EXPERIMENTAL

### *2.1. Chemicals, Equipment and Instrumentation*

The materials used in this study are: rice husks, crab shells, 1M hydrochloric acid (HCl), 3.5% sodium hydroxide (NaOH), 50% sodium hydroxide (NaOH), 1% sodium tripolyphosphate solution (NaTPP), 0.5 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*

#### *2.2.1. Carbonization of Rice Husk*

First wash the rice husks thoroughly to remove any dirt that is still attached. Then dry it in the sun until dry. After drying, the rice husks are ashed in a furnace for 2.5 hours at a temperature of 250° C. After that, the rice husks which have become activated charcoal undergo a blending process to reduce the size. Next, adjust it to a screening size of 200 mesh.

#### *2.2.2. Carbon Activation Process*

Rice husks that have undergone a screening process are then activated by adding 50 grams of rice husk charcoal to 250 ml of 1 M acid activator (HCl), stirring for 30 minutes and leaving for 24 hours. After that, the charcoal is filtered using filter paper and then rinsed with distilled water until the pH is neutral. The charcoal which has become activated carbon is then dried using an oven for 1 hour at a temperature of 110°C

#### *2.2.3. Isolation of Chitosan from Crab Shells*

*Preparation of crab shells*

The collected crab shells are washed first using running water to remove any remaining dirt. Next, the crab shells 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 crab shells are then sifted using a 100 mesh sieve.



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*Isolation of Chitosan from Crab Shells*

*Deproteination*

Crab shell powder is put into a beaker. Then 3.5% NaOH solution was added with a ratio of crab shell powder to solution volume of 1:10. Then heated at 65°C for 2 hours and stirred. After that, both mixtures were filtered and rinsed using distilled water until they had a neutral pH. The filtered solid was previously dried in an oven at 100°C for 4 hours.

*Demineralization*

The powder resulting from the deproteination stage was put into a chemical glass and 1N HCl was added in a ratio of 1:15. Then heated at a temperature of 30°C for 1 hour and accompanied by stirring at a speed of 150 rpm. Next, the mixture is filtered and washed using distilled water until it has a neutral pH. The obtained solid was dried in an oven at 100°C for 4 hours. Then chitin products are obtained from crab shells.

*Deacetylation*

A total of 20 grams of chitin powder that has been produced is put into a beaker. Then 50% NaOH was added with a ratio of 1:20. Then heated at a temperature of 120°C for 3 hours and accompanied by stirring at a speed of 150 rpm. After that, the mixture was filtered and washed using distilled water until the pH was neutral. The filtered solid was dried in an oven at 100°C for 4 hours. After that, chitosan products are obtained from crab shells

2.2.4. Making Nano Chitosan Ionic Gelation Method

A 0.2% chitosan solution was prepared in a 0.5 M acetic acid solution. The solution was added with 1% NaTPP solution with a ratio of chitosan solution: NaTPP solution of 5:1 and a stirring speed of 300 rpm. The stirring process was carried out for 4 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 Rice Husk*

The carbonization process was carried out for 2.5 hours using a temperature of 250°C, then black charcoal was obtained from rice husks. In this process, the contents of rice husks such as cellulose and lignin change to carbon and the organic content in rice husks evaporates, which is indicated by the smoke produced during the carbonization process. The smoke indicates that volatile compounds have been released from the rice husks, thereby opening the carbon pores.

### *3.2. Activation of Rice Husk Activated Carbon*

Carbon activation using HCl aims to remove impurities. In the activation process, these impurities will be removed and the more carbon pores that are open, the stronger the absorption capacity will be. The use of HCl



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as an activator is because charcoal activated using HCl produces better absorption compared to charcoal activated with other acid activators[1].

*3.3. Isolation of chitosan from chopped shells*

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

*3.3.1. Deproteination*

The deproteination process uses 3.5% NaOH solution. The purpose of using NaOH is to remove the protein contained in the shredded shell so that the existing protein will react with NaOH to form sodium potreinate by covalently bonding and in this process the chitin functional group will be isolated which will produce chitin and amino acids. In this deproteination process, a cream colored powder is produced with a yield weight of 88.66%.

*3.3.2. Demineralization*

Demineralization is carried out with the aim of releasing the minerals in the crab shells by adding an HCl solution with a concentration of 1N. In this mineral release process the yield obtained was 62.17%, the yield was chitin powder obtained from crab shells.

*3.3.3. Deacetylation*

The use of 50% NaOH in this process is mixed with chitin powder obtained from previous results, to remove the acetamide group ($NHCOCH_3$) in chitin to become the amine group ($NH_2$) in chitosan. In this process a hydrolysis reaction occurs, the function of adding NaOH is also to break the carbon bonds in the acetyl group with the nitrogen particles found in chitin to obtain the amine group ($-NH_2$). The yield obtained was 54.53%

*3.4. Chitosan Characterization with FTIR*

The deacetylated chitosan was analyzed using FTIR. FTIR data is used to see whether or not there are amine groups in chitosan. Characterization of chitosan using an FTIR spectrophotometer produced results as shown in the image below.

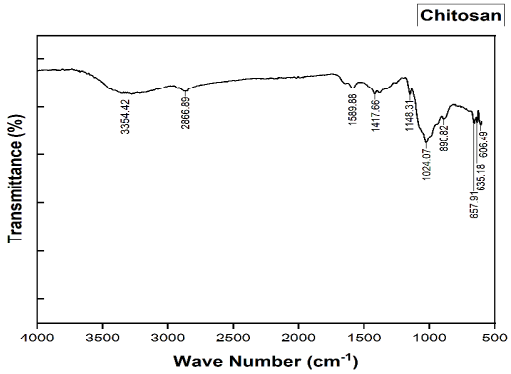


**Figure 1.** FTIR Spectrum of Chitosan



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Chitosan characterization using FTIR above was carried out at a wavelength of 4000-500. The following table displays data from the graph above to analyze the presence of the functional groups found.

**Table 1.** Gugus Fungsi Kitosan

| Functional Group | Wavelength Range $cm^{-1}$ | Wavelength Number $cm^{-1}$ |
|---|---|---|
| N-H stretching | 3300-3500 | 3354.42 |
| OH | 3200-3600 | |
| C-H stretching | 2850-3000 | 2866.89 |
| N-H bending | 1600 | 1589.88 |
| C-H bending | 1350-1480 | 1417.66 |
| C-O-C | 1000-1300 | 1024.07 |

In the FTIR spectrum of chitosan, the absorption peak is seen at a wavelength of 3354.42 $cm^{-1}$. This wavelength occurs due to the extensive absorption of crab chitosan, causing an overlap between the OH and N-H amine groups. Furthermore, there are other absorption peaks 2866.89 $cm^{-1}$ (C-H stretching vibrations), 1589.88 $cm^{-1}$ (N-H bending vibrations), 1417.66 $cm^{-1}$ (C-H bending vibrations), 1024.07 $cm^{-1}$ (C-O-C bridge tensile vibrations)[3]. The formation of chitosan from deacetylated chitin is characterized by a typical absorption peak of chitosan, namely at a wavelength of 3354.42 $cm^{-1}$, the N-H functional group included in chitosan is found.

*3.5. 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 0.5M acetic acid solution, then stirring using a magnetic stirrer for 45 minutes. 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 3 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[6].

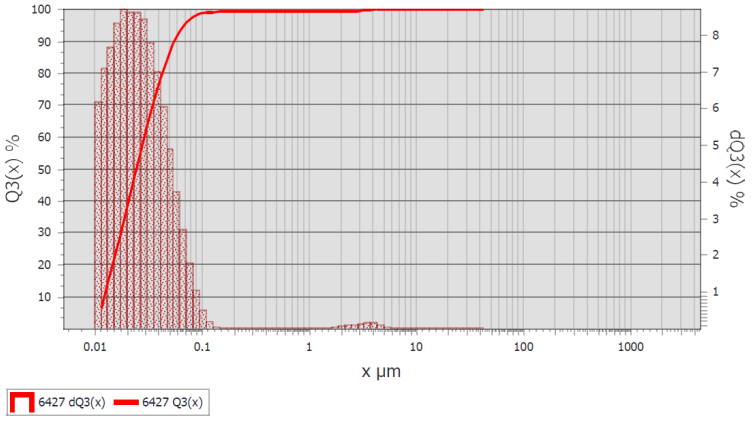


**Figure 2.** PSA Spectrum of Nanochitosan



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In this process, the size of the nanochitosan obtained is 0.05815µm or 58.15nm. 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[4].

*3.6. 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 rice husk adsorbent was obtained which was coated with crab 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**.**

## 4. CONCLUSION

Based on the results of the research that has been carried out, it can be concluded that the preparation and isolation of chitosan from crab shells has been successfully characterized by the discovery of the N-H group from the results of the FTIR test at a wavelength of 3354.42 $cm^{-1}$ which is a characteristic of chitosan. The results of PSA characterization show that the chitosan produced is nano-sized with a size of 58.15 nm. Furthermore, the coating of rice husk activated carbon with crab shell nanochitosan was successfully carried out with the final result being an adsorbent which can be used for the next process.

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