Development and Optimization of Activated Carbon from Rice Husk Using $ZnCl_2$ as an Activating Agent and the Double Crucible Method: A Study on the Effect of Activation Time

B.Tech Project Report Submitted

for the Course

CL 498

Submitted By

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Content

ABSTRACT

This study focuses on the preparation and characterization of activated carbon derived from rice husk using zinc chloride ($ZnCl_2$) as the activating agent. Rice husk, an abundant agricultural by-product, was utilized as a precursor due to its high carbon content and low cost. The synthesis was carried out through chemical activation followed by carbonization using the double crucible method under controlled conditions. The influence of activation time (60–180 minutes) on the material's properties was systematically studied.

Characterization techniques, including FESEM, EDX, XRD, FTIR, BET, and TGA, were employed to evaluate the surface morphology, elemental composition, crystallinity, functional groups, surface area, porosity, and thermal stability of the prepared activated carbon. The results demonstrated that the optimized activation conditions yielded a highly porous material with a large surface area and enhanced adsorption properties.

The study highlights the potential of rice husk-derived activated carbon as an efficient and cost-effective adsorbent for industrial applications such as wastewater treatment and gas adsorption. By addressing key gaps in activation time optimization, this work contributes to the advancement of sustainable materials derived from agricultural waste.

1. INTRODUCTION AND LITERATURE REVIEW

1.1 Prominence Of The Study

Rice husk is an agricultural by-product generated in substantial quantities around the world, especially in regions where rice is a staple crop. Its potential as a carbon source is significant due to its unique structural composition, high availability, low cost, and potential for environmental benefits. The outer portion of the rice grain is referred to as the rice husk. It makes up between 20–33% of the rice's overall weight. With annual rice production exceeding 700 million tons, rice husk is abundantly available. For every ton of rice produced, around 200 kg of rice husk is generated, which equates to millions of tons of rice husk waste worldwide. Rice husks and other agricultural solid waste are converted into goods with added value, which helps to reduce solid waste and preserve the environment. Additionally, this lessens the need for incineration and the expense of disposing of solid waste. Reusing and recycling rice husks helps to reduce soil and air pollution brought on by the open burning and disposal of agricultural trash.

It can be carbonized using the physical activation method in an inert environment. However, the resulting biochar has a limited adsorption capability due to closed pores. In order to expose the biochar to even higher temperatures in the presence of carbon dioxide, steam, or air, a second stage of carbonization is therefore required. The elimination of sticky materials, the formation of new pores, a rise in pore diameter, a bigger pore volume, and a huge surface area would all result from this. Chemical activation, on the other hand, activates and carbonizes rice husks in a single step. Smaller pores may form as a result of the chemical reagents' deeper penetration and increased surface contact inside the carbon structure. Compared to physical activation, chemical activation offers a number of benefits, such as lower working temperatures and higher product yield. The use of activated carbon in adsorption technologies has become more significant as an adsorbent for treating industrial wastewater from the production of chemicals, food, textiles, and medications. Activated carbon is a non-toxic, carbonaceous substance with a large internal surface area and a highly porous structure. Heavy metals and various colors are among the many things that the activated carbon can absorb, and has been proven to be effective for removal of wide variety of inorganic (Gupta et al.,2008) and organic (Gupta et al.,2005) pollutants dissolved in aqueous media, or from gaseous environment (Foo and Hamid,2010). The commonly used Precursor to prepare Activated Caron are Bituminous coal (Qada et al.,2006),Coconut Shell (Yang et al.,2010, Afrane et al.,2008, Guo et al., 2009) and Polymers (Seredych et al.,2010 , Zhu et al., 2007). It is referred to as an adsorbent because of its ability to draw molecules to its interior surface. The ability of activated carbon to adsorb substances is one of its primary features.

This feature is controlled by the active carbons porous structure and surface chemistry, both of which are influenced by their crystalline makeup. A disorganized collection of carbon atoms makes up activated carbon. Both chemical and physical methods are used to activate it. It increases carbon materials surface area and porosity. Zinc Chloride acts as Lewis acid and dehydrating agent, which removes hydrogen and oxygen from biomass . This prevent tar from accumulating on the carbon surface and helps from a porous structure.It can increase the yield of activated carbon by lowering the activation temperature. It can also encourage the development of oxygen-containing groups, which improves the activated carbon's surface hydrophilicity and ion adsorption capability. Activated carbon has grown in importance in adsorption technology as an adsorbent for treating industrial effluent from the manufacturing of chemicals, food, textiles, and medications. Because of its high mechanical strength, enhanced chemical stability, widespread porosity, and large surface area (more than 400 m^2 g^{-1}), activated carbon, a type of carbonized material, has a wide range of uses (Hong et al., 2017).

1.2 Literature Review

The interest in using agricultural waste materials like rice husk for producing activated carbon has grown significantly due to environmental and economic benefits. Rice husk, as a by-product of rice milling, is abundantly available and offers a sustainable raw material for activated carbon due to its carbon-rich composition.

Utilizing rice husk for activated carbon production addresses the disposal issue of agricultural waste and reduces reliance on synthetic carbon sources. According to this study, ash from rice husk char can be leached at a moderate temperature rather than being applied at a high temperature to create porous biochar. The pore structure of porous biochar was shown to grow as the heat treatment temperature rose. Intermediate levels of porousness are generated in the alkali-treated rice husk char, and chemical agents can be used to further develop the pore structure of the alkali-leached rice husk char. The double crucible method used by Ahiduzzaman and Islam, 2016 offers an alternative to expensive inert gas environments by creating an oxygen-reduced atmosphere during carbonization, lowering production costs and making the process more sustainable. The adsorption capacity of activated carbon derived from rice husk has been widely studied, especially for pollutants in wastewater.

Arifin et al.,2020 exhibited the synthesis of GRHA in the absence of inert gas using rice husk as starting material via chemical activation using H_3PO_4 as activating agent . A small number of graphene-like material layers with defect structures were produced using this process. The material exhibits a low degree of graphitization and an amorphous structure. However, TGA analysis revealed minimal weight loss, indicating that all of the samples had acceptable thermal stability. Furthermore, the activation with H_3PO_4 exhibits a significant increase in surface area. The pseudo second order model, which suggested that the gas molecule was adsorbed in the material by chemisorption, was followed by the hydrogen adsorption. In the meantime, a multi-step adsorption process governed the intraparticle particle diffusion model of hydrogen on GRHA 1:1 and GRHA 1:3, but intraparticle diffusion alone governed GRHA 1:5.

Futalan et al.,2023 used rice husk as a carbon resource and lemon juice as an activating agent . SEM micrographs demonstrated that the use of lemon juice as an acidic activation agent resulted in the development of more holes with larger sizes. The ability of activated carbon made from rice husks to absorb substances was examined. The activated carbon contained amides, alkenes, carboxyl, and hydroxyl groups, according to FTIR analysis. The most important independent variable in improving the removal efficiency of Pb(II) was found to be pH. RHAC-LJ (rice husk activated carbon – lemon juice) is demonstrated in this work as a potential inexpensive adsorbent for Pb(II) removal from artificial wastewater.

Yousfi et al.,2020 focused on Comparing the effectiveness of activated carbons made in self-generated atmospheres versus inert environments. Use of nitrogen sorption isotherms to determine BET surface area and pore volumes, Similarities observed between carbons prepared in inert and self-generated atmospheres in terms of porosity and micropore distribution. FTIR and Boehm titration results highlighting a reduction in oxygenated functional groups in self-generated atmosphere samples, impacting hydrophilicity and adsorption behavior. Cyclohexane adsorption tests demonstrating similar efficiency for both types of carbons. Slightly slower adsorption kinetics in carbons prepared in inert atmospheres due to higher functional group density. Activated carbons prepared in a self-generated atmosphere show slightly reduced oxygen functional groups, indicating a more hydrophobic nature, while maintaining comparable adsorption capacities. This approach offers cost-effective alternatives to inert-atmosphere carbonization, showcasing potential for scalable, efficient adsorbent production for environmental applications.

Table 1 : Physiochemical properties of the rice husk and activated carbon prepared from rice husk

Precursor	Temperature(°C)	Chemical Treatment	Reference
RH	600,700,800	HCL	[10]
RHA	700	H_3PO_4	[14]
RH	500	Lemon Juice	[12]
Algerian Olive Waste	445	$ZnCl_2$	[13]

1.3 Gaps

In their study, Ahiduzzaman and Islam ,2016 successfully synthesized activated carbon using $ZnCl_2$ as the activation agent, employing the double crucible method. They systematically varied the activation temperature within the range of 500°C to 900°C and analyzed its influence on the physicochemical properties of the resulting activated carbon. Their findings provided significant insights into the optimization of activation temperature as a critical parameter for enhancing the quality of activated carbon. However, the activation time was maintained constant, leaving its potential impact on the material's properties unexplored. To address this gap, our study will focus on evaluating the influence of varying activation time (60-180 mins) on the characteristics of activated carbon. By systematically altering the activation time while keeping other parameters constant, such as the activation agent, activation agent concentration and temperature, we aim to comprehensively understand its role in determining the surface area, porosity, adsorption capacity, and structural morphology of the activated carbon. This investigation will provide deeper insights into the activation process and could contribute to optimizing the production of activated carbon for specific industrial applications, such as water treatment, gas adsorption, and energy storage.

1.4 Objective

To optimize the characteristics of activated carbon by systematically varying the activation time(60–180 minutes) using $ZnCl_2$ as an activation agent using double crucible method.

2. MATERIAL AND METHODS

2.1 Chemicals and Materials

Rice husk used was bought from one of the rice mills from Guwahati. Deionized water used for pre - processing was bought from Chemical Lab IIT Guwahati. Zinc Chloride used for activation of processed rice husk was from HiMedia Laboratories Pvt. Ltd. (Purity 97 – 102.0 %).

2.2 Preparation Of Rice Husk Derived Activated Carbon

Soaked Rice husk in water in a beaker for 1 hour to remove external dirt and impurities. After removing water It was washed with Deionized water for 3 times and kept for an interval of half an hour to settle down impurities. After cleaning it with Deionized water, wet rice husk was kept in tray which was then placed in Oven for 24 hours at 110°C for drying. Before chemical activation with Zinc chloride the sample is grinded.

Afterward, a chemical activation with $ZnCl_2$ (purity >97%) is conducted using a ratio of precursor: $Zncl_2$ of 1:2 . About 25 gram of RH was mixed with 50 mL of 0.5 M $ZnCl_2$ solution at 90°C for 3 h in magnetic stirrer at Stirring speed of 500 revolution per minute. Prior to second activation, the sample was dried for 24 h at 80°C in an oven.

The sample was put in a double crucible with ground silicon after it had dried. A larger porcelain crucible is placed within the smaller one that contains the rice husk char. After the temperature reached 700°C, the larger crucible was covered with a lid and placed in a muffle furnace that was set to 700°C for 1.5 hours. The space inside the larger crucible was filled with raw husk to reduce the oxygen environment inside the crucible. The sample was allowed to lower the muffle temperature for a full day after this muffle was turned off. Later the sample was collected in glass bottle and kept in desiccator which contain silica so that sample does not observe moisture.

2.3 Characterization

TGA - TGA analysis is performed to evaluate the thermal characteristic of the GRHA. Analyzes the thermal stability of the material and quantifies weight loss due to moisture or volatile compounds. This helps assess the thermal robustness of the activated carbon for industrial applications.

FESEM - A Field Emission Gun (FEG) serves as the electron source for FE-SEMs. To emit the electron beam in FEGs, a potential gradient is employed. Used to observe the surface morphology of the activated carbon derived from rice husk, ensuring the development of a porous structure critical for adsorption efficiency.

EDX- Complementary to FESEM, EDX identifies the elemental composition of the material. It helps confirm the presence of key elements such as carbon and residues from the activation process, like Zn.

FTIR - A powerful analytical technique used to identify the chemical structure, and functional groups present in a material. It works by measuring how a sample absorbs infrared light at different wavelengths. Identifies functional groups on the surface of the activated carbon. Groups like hydroxyl, carboxyl, and others impact hydrophilicity and adsorption efficiency for various pollutants.

BET - Measures the specific surface area and porosity, key indicators of the adsorption capacity of the activated carbon. Higher surface area correlates with better adsorption. This is essential for assessing the adsorption capacity of the rice husk-derived activated carbon. It provides insights into the material's surface area, pore size, and pore volume, which are critical factors for its performance as an adsorbent.

XRD - Used to determine the crystalline structure of the rice husk-derived activated carbon. It helps in confirming the amorphous or partially crystalline nature of the material, essential for its adsorption capabilities.

Proximate Analysis - The physical and chemical characteristics of the material, including its moisture content, volatile matter, ash content, and fixed carbon, are the main focus of proximate analysis. While volatile matter refers to chemicals released after heating that impact combustion efficiency, moisture content indicates the amount of water present.

Ultimate Analysis - It provides a detailed breakdown of the material's elemental composition, including carbon, hydrogen, oxygen, nitrogen, and sulfur. This analysis helps assess the material's potential energy output, environmental impact, and suitability for specific applications like adsorption or fuel production.

2.4 Methodology

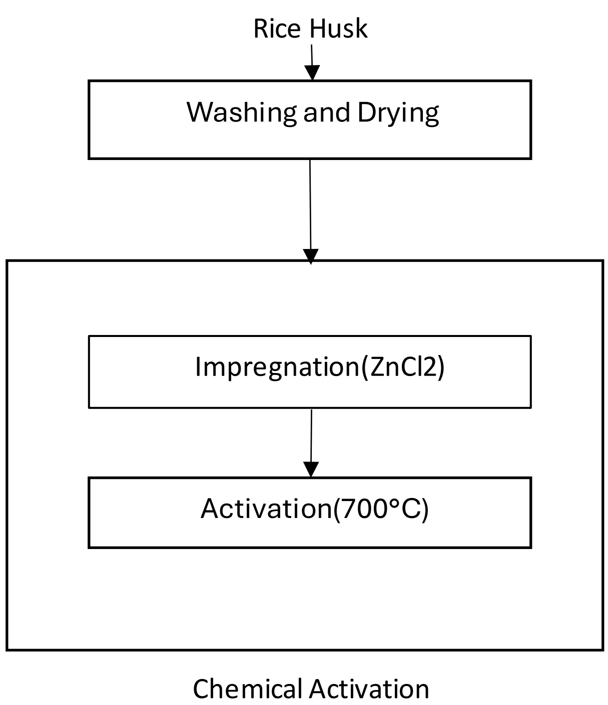

Fig 1. Schema of the activated carbons preparation from rice husk.

3. FUTURE WORK

This study provides a foundational understanding of the preparation and characterization of rice husk-derived activated carbon using $ZnCl_2$ activation. Future research can explore several avenues to enhance and expand the application of this material. Firstly, optimizing other parameters, such as activation agent concentration, temperature, and precursor-to-activating agent ratios, could further improve the material's porosity and adsorption capacity. Additionally, testing the activated carbon's performance in removing specific pollutants, including heavy metals, dyes, and pharmaceuticals, from real industrial wastewater would provide practical validation.

Activated carbon with better qualities could be produced by investigating other chemical activating agents or by combining chemical and physical activation techniques. Additionally, the material would be ideal for industrial uses if the process could be scaled up while maintaining economic and environmental viability. Finally, research into the activated carbon's regeneration and reusability would support its sustainability in long-term uses. Future studies can optimize the material's potential and expand its uses in a variety of industries by tackling these issues.

4. TIMELINE

S. No	Activity	Timeline			
		Aug-Oct 2024	Nov-Jan 2024	Feb-April 2025	April-June-2025
1	Literature Review				
2	Preliminary Experiments				
3	Objectives of BTP				
4	BTP Report Writing				

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