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CHARACTERISATION OF ACTIVATED CARBON FROM CORN HUSK: ANALYSIS OF FUNCTIONAL GROUPS, MORPHOLOGY, STRUCTURE, AND TEXTURE

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ABSTRACT
This study aims to evaluate the characteristics of activated carbon from corn husk pyrolysis at 400°C. Corn husk charcoal was activated using HCl solution with concentrations of 1, 1.5, and 2 N for 24 hours, then washed until neutral, and dried at 110°C. Characterisation was carried out using Fourier Transform Infrared Spectroscopy (FTIR) to identify functional groups. Scanning Electron Microscopy (SEM) was used to study surface morphology, while X-Ray Diffraction (XRD) provided information on the crystal structure. The specific surface area, pore volume, and pore size distribution were determined using Brunauer-Emmett-Teller (BET) analysis and the N_2 adsorption-desorption method. The results showed that activation with HCl at a concentration of 1.5 N led to significant changes in the physicochemical properties. In addition, the specific surface area increased from 7.70 m^2/g to 22.48 m^2/g, the total pore volume increased from 0.0555 cm^3/g to 0.0683 cm^3/g, and the average pore diameter decreased from 28.8 nm to 12.1 nm. FTIR spectrum showed a decrease in the intensity of hydroxyl and aromatic groups, indicating a reduction in lignocellulose content, while the SEM image revealed a more porous and fragmented surface after activation. XRD analysis showed an amorphous nature with a broad peak at around $2\theta \approx 22°$ and an increase in local intensity after activation. These changes in texture and chemical aspects indicated that HCl activation was effective in improving the characteristics of activated carbon from corn husk, making it a low-cost adsorbent candidate for environmental applications.
Keywords: Activated Carbon, Corn Husk, HCl Activation, BET, FTIR, SEM, XRD
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INTRODUCTION

In recent years, water pollution from industrial waste, specifically waste containing dyes and heavy metals, has increased, becoming a global problem and concern. Consequently, various methods have been used to address the issue, such as coagulation, membrane filtration, oxidation, and photocatalysis. One method that has been widely developed is photocatalysis, using materials such as ZnO or TiO combined with ultraviolet light exposure.[1] Despite the potential, photocatalysis generally has drawbacks, including high cost, limited efficiency, and the ability to produce hazardous residues. Adsorption using activated carbon has become a popular alternative due to its effectiveness, economy, and environmental friendliness. Activated carbon is widely recognised as an effective adsorbent due to its high surface area, developed pore structure, and active functional groups that can support physicochemical interactions with the adsorbate. However, commercial activated carbon is relatively expensive and difficult to obtain locally. One promising strategy is the use of agricultural waste as a substitute raw material.[2] Corn husk is an abundant agricultural waste, with characteristics that allow it to be used as a high-quality activated carbon.[3] A study showed that activated carbon derived from corn husk could remove up to 99% of certain contaminants, including dyes and heavy metals, making it an effective alternative to traditional activated carbon in more sustainable wastewater treatment.[4] Studies have shown that the material has a surface area ranging from 90 to 450 m^2/g, depending on the activation method used, such as chemical activation with $ZnCl_2$, NaOH, or functional amines. In addition to corn husks, several agricultural waste has been used as raw materials for activated carbon, such as durian husks,[5] young coconut shells,[6] olive seeds,[7] sugarcane bagasse,[8] coffee grounds,[9] walnut shells,[10] rice husks,[11] wheat straw,[12] and other agricultural waste

containing organic and inorganic carbon elements. According to previous studies, corn husk is highly suitable as a raw material for activated carbon. This is due to the chemical composition, which includes cellulose (36.81%), hemicellulose (27.01%), lignin (15.7%), and ash (6.04%). These components are processed into a material with a large surface area through a carbonisation process.[13] The biosorbent manufacturing process can also be combined with renewable energy technology through pyrolysis, which produces biochar and syngas as alternative energy sources.[14] Pyrolysis is a form of incomplete combustion because it avoids the conversion of complex carbon molecules into carbon dioxide, specifically in components such as cellulose, hemicellulose, and lignin.[15] In addition to pyrolysis, chemical activation is also used because it is generally superior to physical activation and can produce a larger surface area as well as a more even pore distribution.[16] This activation process uses certain chemicals, one of which is HCl, due to its advantages.[17] Several studies have shown that the use of HCl in the biomass activation process can improve the purity of activated carbon, increase surface area, and add active sites to interact with dye molecules.[18] Other studies have reported that the selection of activator type and concentration can change the pore distribution and surface properties of activated carbon, thereby affecting the adsorption capacity.[19] This current study focuses on the characterisation of activated carbon produced from biomass through physical, chemical, and morphological analysis. Instruments, such as FTIR, SEM, XRD, and BET, were used for the characterisation. The analysis results provide important information regarding the structural and chemical properties of activated carbon, while demonstrating its potential as a sustainable and environmentally friendly material for various applications. Within the framework of the United Nations Sustainable Development Goals (SDGs), the development of locally available, low-cost biomass-derived adsorbents for water and wastewater remediation aligns directly with SDG-6 (Clean Water and Sanitation), particularly Target 6.3, which emphasises improving water quality by reducing pollution, eliminating dumping, and minimising the release of hazardous chemicals and materials.[20]

EXPERIMENTAL

Making Charcoal from Corn Husks

Corn husk waste was first cleaned, dried in the sun for 3 days, and then cut into small pieces (± 2-3 cm). The charcoal production process was carried out through a pyrolysis process. Approximately 1.8 kg of corn husk was placed in the reactor, then pyrolysed at 400ºC for 3 hours. This process produced liquid smoke, tar, and charcoal as shown in Fig-1. Subsequently, the charcoal was sieved with a size of 80 mesh and then ground to nano-size using a Ball Mill (Retsch Emax).

Chemical Activation of Corn Husk Charcoal

The charcoal was activated with HCl for 24 hours with 3 variations of activator, namely 1 N, 1.5 N, and 2 N. Subsequently, it was washed with distilled water until the pH was neutral. The sample was dried in an oven at a temperature of 110ºC for 4 hours.

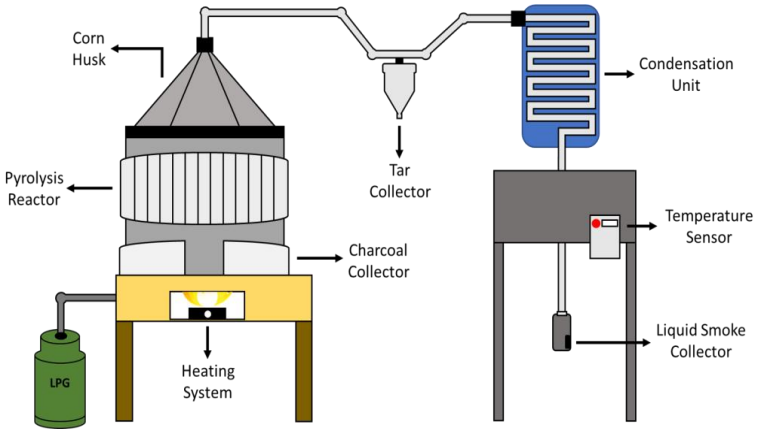

Fig-1: Pyrolysis Process Scheme

RESULTS AND DISCUSSION

FTIR (Fourier Transform Infrared Spectroscopy) Analysis

Analysis of chemical functional groups in the material was carried out using the FTIR method. This analysis aimed to identify functional groups presented on the surface of the adsorbent, which played an important role in the absorption process. FTIR spectrum was obtained in the wavelength range of 500 to 4000 cm^{-1}, and was used to compare the chemical characteristics of the charcoal before and after the chemical activation process using HCl with various concentrations of 1, 1.5, and 2 N as seen in Fig.-2.

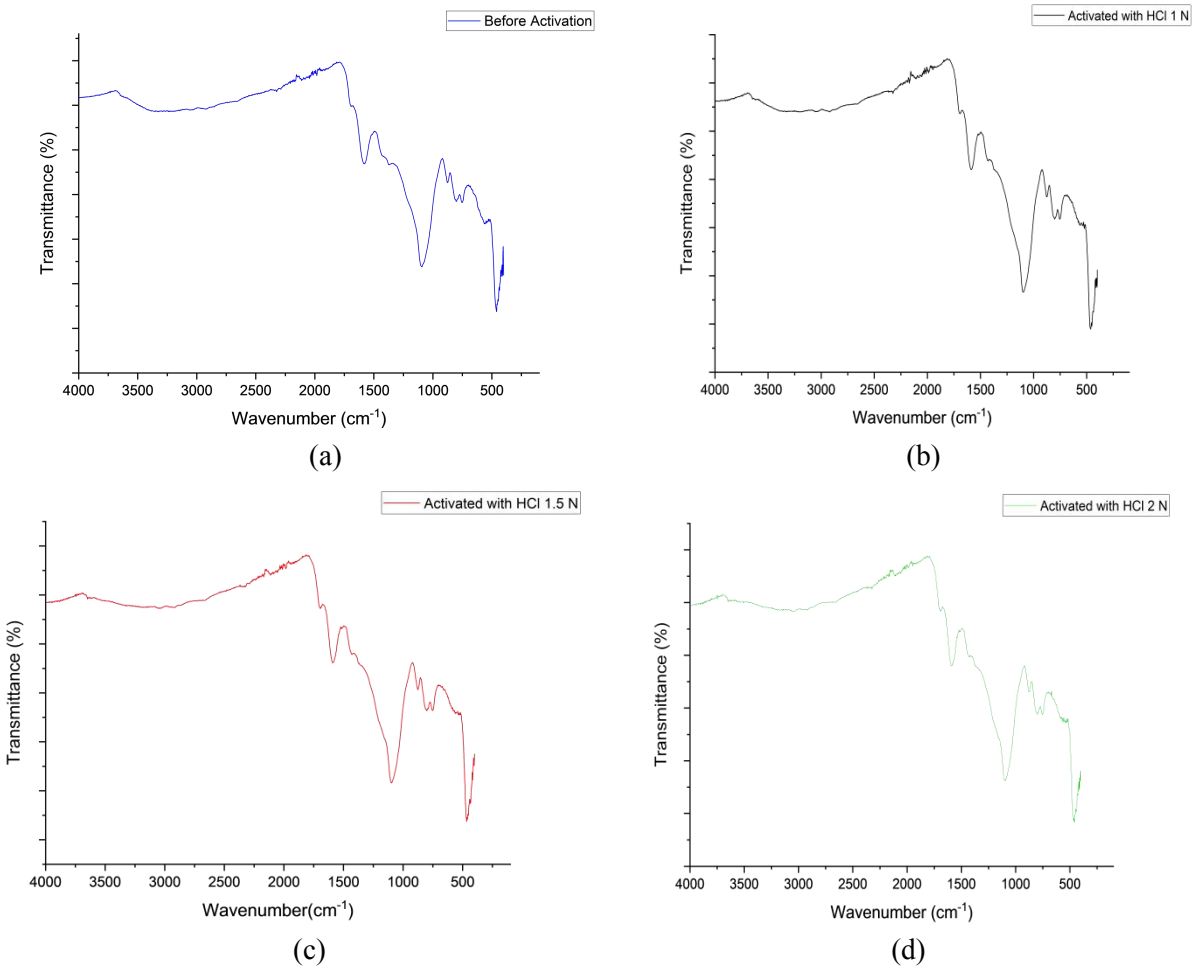

Fig-2: Results of FTIR Analysis on Corn Husk Waste Adsorbent (a) Before Chemical Activation, (b) After Chemical Activation with 1 N HCl, (c) 1.5 N, and (d) 2 N

Based on FTIR analysis, there were significant differences in the spectrum between charcoal before and after chemical activation with variations in HCl concentration (1, 1.5, 2 N). In charcoal before activation, the presence of –OH groups was detected at 3323 cm^{-1} as a result of hydrogen bonds in cellulose, hemicellulose, or lignin. The peak at 1582 cm^{-1} indicated the presence of C=C bonds, which were characteristic of aromatic lignin, while the range of 1092 cm^{-1} was related to the C-O group of polysaccharides. Furthermore, the range of 460-410 cm^{-1} was related to aromatic C-H vibrations and inorganic minerals, showing that the material still contained many lignocellulosic components and impurities.[21] After chemical activation with HCl, there was a change in intensity and a shift in the absorption wavenumber, indicating the modification of functional groups. In 1 N HCl activation (Figure 2b), the –OH group shifts to 3203 cm^{-1} with a lower intensity, showing the reduction of hydroxyl groups. The aromatic peak around 1584 cm^{-1} was still detected, but its intensity decreased because some of the lignin began to decompose. Activation with 1.5 N HCl (Fig.-2c) showed the –OH group located at 3048 cm^{-1} with a decreasing wavenumber, as well as the aromatic peak at 1589 cm^{-1}, which continued to

decrease. This showed that lignin and hemicellulose experienced a decrease, while at 801 cm^{-1}, there was an aromatic C-H vibration. At a concentration of 2 N HCl (Fig.-2d), the changes were more obvious, where –OH was detected at 3044 cm^{-1} with the lowest intensity, while the 1589 cm^{-1} peak became weaker. At wave number 1099 cm^{-1}, the peak appears more dominant, where the C-O groups of cellulose were present due to the loss of non-cellulose components. These results were consistent with studies on young coconut shell charcoal activated with NaOH, where the –OH peak in the 3500–3200 cm^{-1} range decreased in intensity after activation, indicating a reduction in hydroxyl content. Increasing the HCl concentration showed a stronger effect in reducing the hydroxyl and aromatic groups of lignin and hemicellulose, leading to a cleaner and more open material structure. This result was consistent with the literature stating that chemical activation with HCl and NaOH could remove organic impurities, reduce bound oxygen content, and increase porosity and the availability of active sites for adsorption.[22,23] However, there were differences in the results between corn husks and young coconut shells. HCl activation on corn husks was more effective in degrading lignin and hemicellulose, leading to a purer surface, while NaOH activation on young coconut shells tended to retain aromatic groups (C=C) and increase the stability of the carbon skeleton. Therefore, HCl-activated corn husks had the potential to produce materials with cleaner and more porous surfaces, while NaOH-activated young coconut shells were superior in maintaining their aromatic structure for long-term applications.

SEM (Scanning Electron Microscopy) Analysis

Adsorbent characterisation using SEM was conducted to observe the morphological structure and pore size on the adsorbent surface. The results of SEM analysis for corn husk waste adsorbent, both before and after chemical activation with HCl at various concentrations, are shown in Fig.-3.

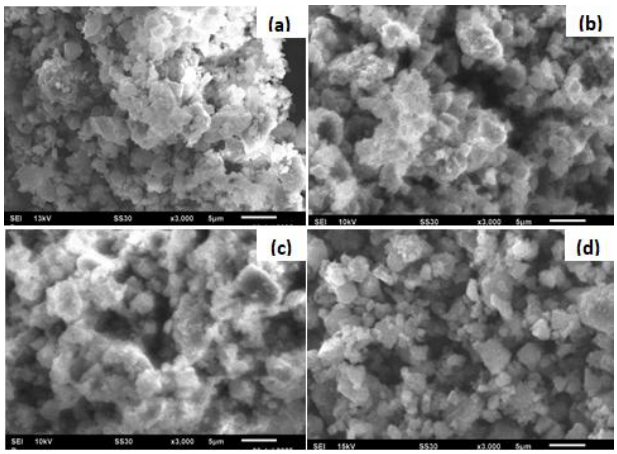

Fig-3: Results of SEM Analysis with 3000x Magnification on Biosorbent
(a) Before Chemical Activation and (b) After Chemical Activation with 1 N HCl (c) 1.5 N (d) 2 N

Based on the results of SEM analysis with a magnification of 3000x, changes in the morphology of the biosorbent were observed before and after chemical activation with HCl at various concentrations. The charcoal before activation (Fig.-3a) showed a surface that was still dense and aggregated, with pores that were not clearly developed. Therefore, the active surface area available for the adsorption process was relatively low. After chemical activation with HCl at a concentration of 1 N (Fig.-3b), the biosorbent surface began to show pore formation and a looser structure, which indicated the release of impurities and the opening of cavities by the activation process. At a concentration of 1.5 N (Fig.-3c), the biosorbent surface showed an increase in the number of pores with a rougher texture, resembling small and broken grains, leading to a more open structure and potentially increasing the surface area and adsorption capacity. Meanwhile, chemical activation with HCl at a concentration of 2 N (Fig.-3d) showed larger pores and a rough surface, but the structure appeared less stable due to the more intensive corrosion process. This showed that the higher the HCl concentration, the greater the porosity formed, although at excessive concentrations it can cause structural degradation and potentially reduce the stability of the biosorbent. The results of this analysis were in accordance with the study of Abegunde *et al*.,[24] stating that

chemical activation with acid could increase the number of pores and the surface area of the adsorbent, thereby increasing the adsorbent capacity. A study by Bouanzi *et al*.,[25] Also stated that the biosorbent resulting from chemical activation showed a more open morphology compared to the material without chemical activation. This was consistent with the increase in adsorption performance. Furthermore, Soliman *et al*.,[26] observed that chemical activation using a combination of HCl and an oxidiser could change the surface structure of the sludge, leading to a higher surface area and porosity, and improving adsorption performance. Raji *et al*.,[27] also stated that chemical activation treatment on waste produced activated carbon with more developed pores, which directly contributed to the adsorption capacity of heavy metals. Therefore, it could be concluded that chemical activity using HCl was effective in improving the surface structure of the biosorbent, although selecting the optimum concentration was very important to avoid excessive damage to the material.

XRD (X-Ray Diffraction) Analysis

XRD analysis aimed to identify the molecular shape and crystal structure of the adsorbent material. The crystal shape of this material greatly affected the adsorption process. Figure-4 showed the XRD pattern of corn husk charcoal before and after chemical activation with HCl at a concentration of 1.5 N. Both patterns showed a broad peak around $2\theta \approx 22^{o}$; the absence of a sharp peak indicated the presence of a crystalline phase.

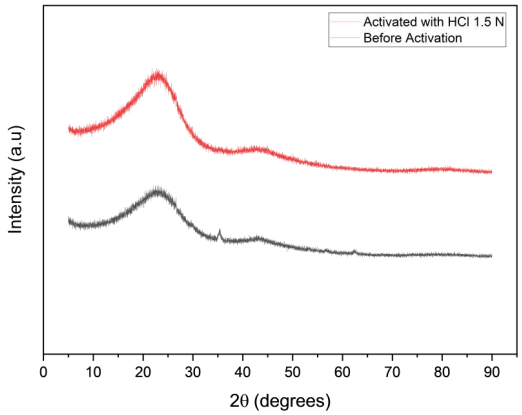

Fig-4: Results of XRD Analysis on Corn Husk Waste Adsorbent Pyrolysis and After Chemical Activation

This showed that the sample had an amorphous nature, or irregular structure, which was the main characteristic of activated carbon derived from biomass. Furthermore, this broad and blunt peak indicated the presence of an imperfect graphite structure, which was usually found in non-crystalline carbon produced from the pyrolysis process.[28] After activation with HCl, the peak intensity increased, indicating changes in the microstructure, such as increased local order or the formation of microcrystalline domains. The activation process could also dissolve inorganic compounds, such as silica or heavy metals, trapped in the pores, resulting in a more open structure and increased surface area.[29] This pattern was consistent with the results of Kishibayev *et al*.,[30] who showed that activated carbon produced from biomass waste generally had a semi-amorphous nature because the carbonisation and activation processes did not create a perfect crystalline order. Therefore, the observed diffraction pattern remained consistent with the characteristics of amorphous carbon, which had high efficiency in adsorption applications due to its large surface area and the presence of active functional groups.

BET Analysis (Brunauer-Emmett-Teller)

This analysis was conducted using the BET method for adsorbent characterisation to measure its surface area and pore size. The results of BET analysis showed that chemical activation with HCl at a concentration of 1.5 N was able to improve the textural properties of activated carbon compared to only pyrolysis treatment. Furthermore, the specific surface area increased from 7.70 m^2/g to 22.48 m^2/g, while the total pore volume increased from 0.0555 cm^3/g to 0.0683 cm^3/g. The average pore diameter decreased from 28.8 nm to 12.1 nm, indicating the formation of smaller pores after chemical activation.

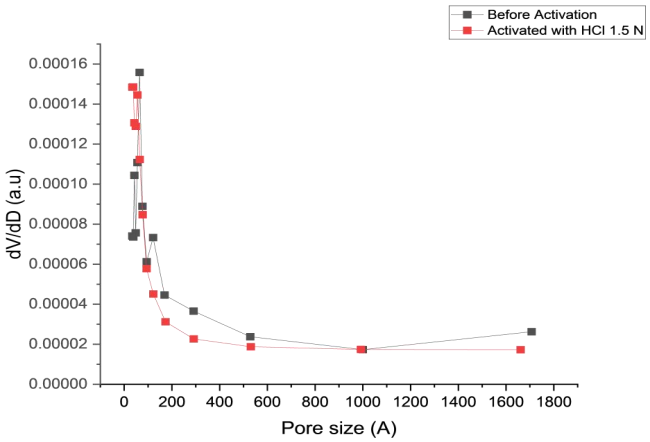

Fig.-4: BET Analysis Results on Corn Husk Waste Adsorbent Pyrolysis and After Chemical Activation

The pore size distribution also shifted, where before chemical activation, the peak was at 64.5 Å with a value of 1.558×10^{-4} cc•Å^{-1}•g^{-1}. Meanwhile, after activation, the peak shifted to 37.9 Å with a higher value, namely 5.179×10^{-4} cc•Å^{-1}•g^{-1}. The increase in surface area and the number of small pores strengthened the ability of activated carbon for the adsorption process.[19,31] The physicochemical improvements achieved after HCl activation, including increased specific surface area, enhanced pore development, and a more open surface morphology, represent material attributes widely recognised as influential for adsorption-based treatment in water and wastewater applications.[32,33] Accordingly, although adsorption performance toward specific target contaminants was not quantified in the present study, the resulting characteristics provide a robust materials basis for further development of low-cost adsorption technologies that support progress toward SDG-6, especially Target 6.3. [20]

CONCLUSION

In conclusion, this study demonstrates that corn husks can be processed into activated carbon through a pyrolysis process followed by chemical activation using HCl solution. Variations in HCl concentration can significantly affect the chemical structure and morphology of the material. FTIR characterisation results show a reduction in hydroxyl groups, aromatics, and non-cellulosic compounds after activation, while SEM analysis indicates the formation of a more porous surface. XRD data show changes in the degree of crystallinity, and BET results confirm an increase in the specific surface area at higher activator concentrations. Therefore, HCl activation can modify the functional group properties and texture of activated carbon from corn husks, thereby increasing its potential as an adsorbent. Overall, these findings underscore the study's relevance to SDG-6 (Clean Water and Sanitation), particularly Target 6.3 by providing a biomass-derived activated carbon candidate with potential for further development as a low-cost adsorbent for water and wastewater treatment applications.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean Water and Sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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