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Optimization of Preparation Conditions of Activated Carbon from Millet Stalk Agricultural Waste

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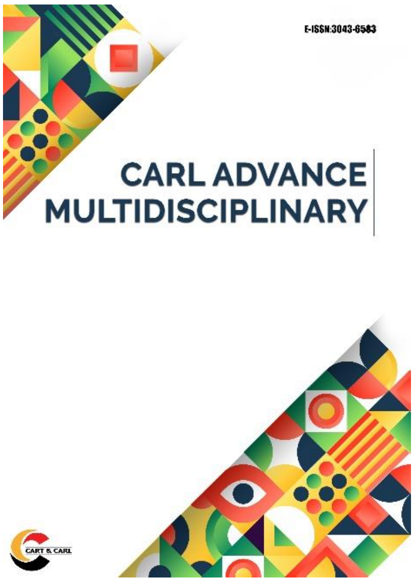

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Abstract

The use of Activated Carbon (AC) has a long history dating back to ancient times. In recent years, there has been growing interest in utilizing agricultural by-products as a potential resource for water pollution control. The study investigates and explore the optimization of preparation conditions of activated carbon from millet stalk agricultural waste. To obtain millet stalk activated carbon with the best performance, three parameters that influence the adsorption capacity of the activated carbon: iodine number, cation exchange capacity (CEC), and bulk density were examined by systematically varying the preparation process variables including phosphoric acid (H_3PO_4) acid concentration, impregnation ratio, and carbonization temperature. From the result, higher concentration of H_3PO_4 leads to a decrease in bulk density while a decrease in bulk density may improve adsorption capacity. From the result, it can be seen clearly that the CEC of the adsorbent begins to increase with an increase of the temperature from 350 to 550 °C, reaches a maximum of 2.5 meq/g at 550 °C and subsequently decreases when the temperature exceeds 550 °C. Agricultural wastes such as millet stalk considered as suitable raw materials for preparation of activated carbon by using different chemical activating agents.

Keywords : Activated Carbon, Agricultural By-product, Adsorption Capacity, Millet Stalk

Introduction

The use of Activated Carbon (AC) has a long history dating back to ancient times. The Egyptians used charcoal for reduction of ores in the manufacture of bronze and for medicinal applications as early as 3750 BC (Inglezakis and Poulopoulos, 2006). On the other hand, the Hindu documents dating from 450 BC show the use of sand and charcoal filters for the purification of drinking water. Activated carbon in form of char is also reported in keeping drinking water fresh in recent studies of the wrecks of Phoenician trading ships (University of Kentucky, 2012). In the time of Hippocrates (ca. 460 - 370 BC) and Pliny the Elder (AD 23 - 79) wood chars were employed for medicinal purposes (Hassler, 1963). During the time of Columbus (15th century) sailors put drinking water in wooden barrels that were blackened in the insides with fire to keep the water fresh. Water pollution caused by organic pollutants poses a significant threat to ecosystems and human health. Organic pollutants, including pesticides, pharmaceuticals, and industrial chemicals, are commonly found in water bodies due to agricultural, industrial and domestic activities. Conventional water treatment processes often struggle to effectively remove these complex and persistent compounds, leading to their accumulation and potential adverse effects on aquatic life and human populations. Therefore, there is an urgent need to explore sustainable and cost-effective solutions for the remediation of organic pollutants from water systems (Bashir et al., 2020).

In recent years, there has been growing interest in utilizing agricultural by-products as a potential resource for water pollution control. Agricultural by-products, such as crop residues, food waste, and animal manure, are abundantly generated worldwide as a result of agricultural activities and food production processes. These by-products, which are often considered as waste materials, possess inherent characteristics that make them suitable for the remediation of organic pollutants (Hampel et al., 2015). The utilization of agricultural by-products for water remediation offers several advantages (Okoya et al., 2020). Firstly, it provides an environmentally friendly approach by repurposing waste materials and reducing their potential negative impacts on landfills or natural ecosystems. Additionally, agricultural by-products are cost-effective alternatives compared to traditional treatment methods, as they are readily available and can be obtained at low or even no cost. Moreover, their utilization promotes sustainable practices by reducing the reliance on synthetic adsorbents or chemical treatments, thereby minimizing the carbon footprint associated with water remediation processes (Blachnio et al., 2020; Karić et al., 2022).

The remediation potential of agricultural by-products lies in their unique properties. They possess high surface areas, abundant porous structures, and diverse functional groups that facilitate the physical adsorption, chemical interactions, and biodegradation of organic pollutants. Moreover, the presence of active compounds, enzymes, and microorganisms within these by-products contributes to their pollutant removal capabilities. However, further research is needed to understand the mechanisms involved in pollutant removal and optimize their performance (Akuso et al., 2020; Okoya et al., 2020). The study aimed to investigate and explore the optimization of preparation conditions of activated carbon from millet stalk agricultural waste.

Materials and Methods

To obtain millet stalk activated carbon with the best performance, three parameters that influence the adsorption capacity of the activated carbon: iodine number, cation exchange capacity (CEC), and bulk density were examined by systematically varying the preparation process variables including H_3PO_4 acid concentration, impregnation ratio, and carbonization temperature. Each preparation test was conducted as follows: 40 g of the powdered millet stalk was mixed with H_3PO_4 solution having different concentrations (10 to 80 % H_3PO_4 in weight). The impregnation ratio, defined by the weight ratio of impregnant (H_3PO_4) to millet stalk powder, was 1; 1.25; 1.5; 1.75; 1.85 and 2. The impregnation was carried out in a three-neck round bottom flask on a hot plate magnetic stirrer. The temperature and the duration of the reaction were 104 °C and 2 hours, respectively. The carbonization of the impregnated material was conducted in a muffled furnace at temperatures ranging between 350 to 650 °C, while activation time was maintained at 2 hours. After cooling down to room temperature, the obtained activated carbon was thoroughly washed with hot distilled water until a neutral pH of 7 was obtained. The sample was then dried at 105 °C overnight, ground (until a particle size ranging between 100 and 160 µm) and finally kept in a closed bottle for subsequent uses.

Result and Discussion

The iodine number is directly related to the surface area of an adsorbent as it gives an idea of the micro-porosity of the activated carbon (Joshi et al., 2021; Nunes & Guerreiro, 2011). As it can be seen from Figure 4.1, increasing the impregnation ratio resulted in a sharp increase in iodine number and was highest at an impregnation ratio of 1.75. However, the iodine number decreased when the impregnation ratio is above or below 1.75. The effect of H_3PO_4 concentration on bulk density is presented in Figure 4.2. The bulk density of an adsorbent is closely related to the adsorption capacity. Adding different H_3PO_4 concentrations to millet stalk activated carbon resulted in different bulk densities as seen in Figure 4.2. The activation process with H_3PO_4 increases surface area and influences the pore distribution. From the result, higher concentration of H_3PO_4 leads to a decrease in bulk density. While a decrease in bulk density may improve adsorption capacity, adsorbent with very low bulk density may be ineffective due to decreased mechanical strength and an increase in pressure drop when adsorbate is passed through it (N'Tsoukpoe et al., 2014). A balance between increasing surface area through higher H_3PO_4 concentrations and maintaining a suitable bulk density is needed. From the result, the optimal H_3PO_4 concentration that has a bulk density of 1 was selected. This is because adsorbent having a bulk density between 0.8 – 1 g/cm^3 adsorbs organic contaminants effectively (Ali et al., 2012; Zhao et al., 2023).

The carbonization temperature has a high impact on cation exchange capacity (CEC) of an adsorbent, this influences its ability to adsorb organic contaminants. Increase in carbonization temperature results to the loss of oxygen-containing functional groups which are responsible for adsorbing contaminants (Cao et al., 2019). Conversely, lower carbonization temperatures may result in lower surface area development and higher ash content which might block the pores resulting in a reduction in the CEC of the adsorbent. This reduction in CEC is less suitable for adsorbing organic contaminants.

As such, there is a need to assess the temperature of carbonization by evaluating its effect on the CEC of the adsorbent. The effect of carbonization temperature on the CEC of the adsorbent was investigated and presented in Figure 4.3. From the result, it can be seen clearly that the CEC of the adsorbent begins to increase with an increase of the temperature from 350 to 550 °C, reaches a maximum of 2.5 meq/g at 550 °C and subsequently decreases when the temperature exceeds 550 °C. The CEC of 2.5 meq/g agrees with the works of (Baccar et al., 2009; Duguet et al., 2006) which reported values of 2.42 and 2.33 meq/g respectively. This suggest that the carbonization temperature of leads to a better development of the adsorbent's CEC which in turn improves adsorption capacity to trap organic contaminants effectively. Several studies have established that the optimal temperature of carbonizing agricultural waste adsorbent after acid activation is within the temperatures of 500 – 550 °C (Alothman et al., 2011; Bouzid et al., 2023; Köseoğlu & Akmil-Başar, 2015).

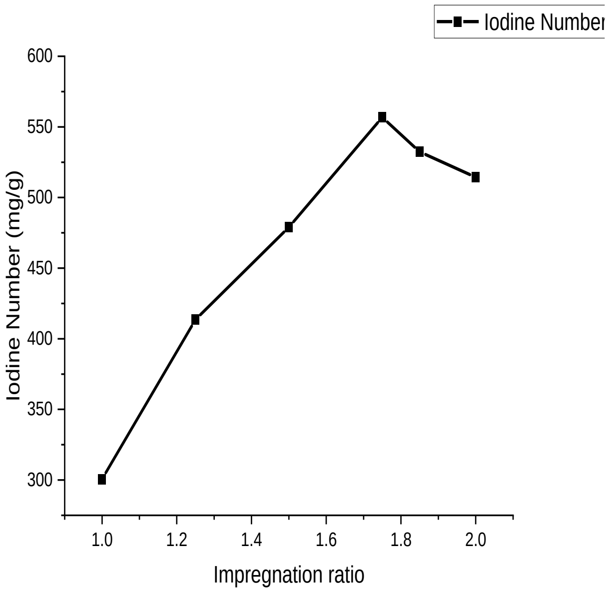

Figure 4.1. Effect of impregnation ratio on the Iodine number of millet stalk activated carbon (H_3PO_4 concentration: 40%; carbonization temperature: 500 °C;)

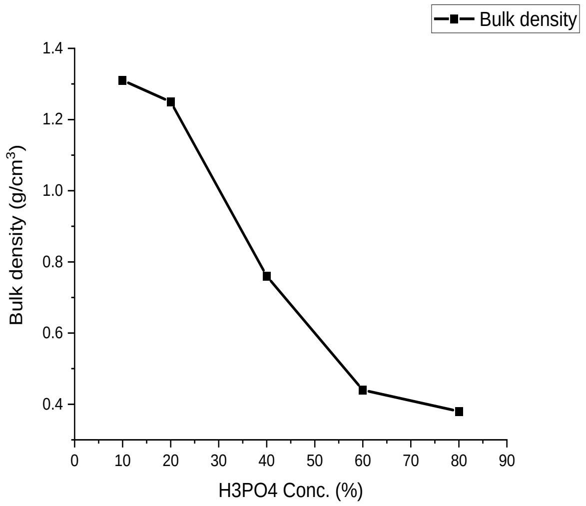

Figure 4.2. Effect of H_3PO_4 concentration on the Bulk density of millet stalk activated carbon (impregnation ratio: 1.75; Pyrolysis temperature: 500 °C;)

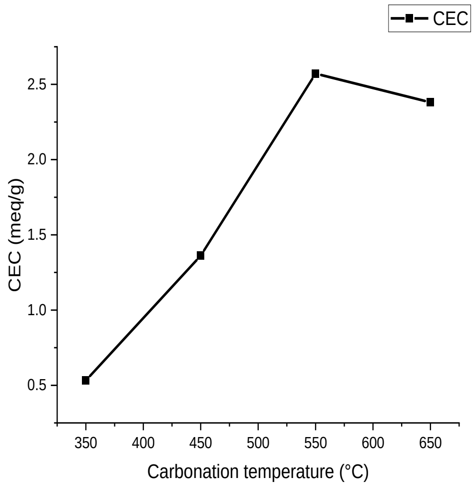

Figure 4.3: Effect of Carbonization temperature on the CEC of millet stalk activated carbon (H_3PO_4 concentration: 30%; impregnation ratio; 1.75)

Conclusion

Agricultural wastes such as millet stalk considered as suitable raw materials for preparation of activated carbon by using different chemical activating agents. The physical and chemical properties of activated carbon vary with the temperature of activation also it was depending on the carbonious row material and contact time during the impregnation and activation. Influence of activation temperature, time and impregnation ratio on development of porosity of activated carbon. The activation was performed using phosphoric acid under different operating conditions of temperature.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Credit Authorship Contribution Statement

Sanni, D.A: Conceptualization, Methodology, Formal analysis, Investigation, Resources, Data curation, Visualization, Project administration, Writing - original draft. **Woke, G.N.** and **Gbarakoro, T.N.**: Supervision, Methodology, Validation, Formal analysis, Data curation, Visualization, Review & Editing.

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