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Biosorption of Caffeine from Contaminated Water onto Sugarcane Bagasse Biomass

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ABSTRACT

Caffeine waste residue has been identified as a significant source of environmental caffeine contamination, primarily originating from the disposal of unfinished caffeinated beverages. Once released, caffeine can reach wastewater treatment plants (WWTPs) and eventually enter the water cycle, posing potential ecological risks. This study investigates the potential of sugarcane bagasse, a readily available and inexpensive material, as an activated carbon adsorbent for caffeine removal. The sugarcane bagasse was chemically activated using sulphuric acid to enhance pore development. Various operating parameters, including adsorbent dosage, contact time, and stirring rate, were examined to optimize caffeine adsorption. The activated carbon was characterized using Low Voltage Scanning Electron Microscopy (LVSEM) and Fourier Transform Infrared Spectroscopy (FTIR). Results demonstrated a maximum caffeine removal efficiency of 99.12%, highlighting the potential of sugarcane bagasse as a low-cost and eco-friendly solution for wastewater treatment applications.

1. Introduction

Caffeine (1,3,7-trimethylxanthine) contamination in water sources has emerged as a significant environmental issue, primarily due to the escalating global consumption of caffeinated products. The persistence of caffeine in aquatic ecosystems has been shown to adversely affect various organisms, including plants and animals, across different regions [1]. In particular, the increasing caffeine levels in water bodies, especially in agricultural soils, pose a threat to aquatic life, even at low concentrations. Research indicates that caffeine has been detected in over 50% of sampling sites

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across 258 rivers worldwide, raising alarms among scientists regarding its detrimental effects on aquatic organisms such as microalgae, corals, bivalves, sponges, marine worms, and fish [2,3].

In Malaysia, caffeine consumption is notably high, with an estimated intake of approximately 1.3 kg per capita, placing it among the top 50 countries for caffeine consumption [4]. The health implications of excessive caffeine intake are well-documented; for healthy adults, the recommended limit is 300-400 mg per day [5]. Exceeding this threshold can lead to various health issues, including sleep disturbances, anxiety, nervousness, headaches, and gastrointestinal problems [1,6]. Furthermore, high caffeine consumption during pregnancy has been linked to increased risks of miscarriage, preterm birth, and low birth weight [7].

Conventional methods for caffeine removal from contaminated water are often costly and energy-intensive, failing to effectively degrade certain toxic organic compounds while generating additional waste [8]. This necessitates the exploration of more efficient, eco-friendly, and cost-effective alternatives for water purification. Carbon-based adsorbents derived from renewable sources offer several benefits, including affordability, diverse forms, versatile porosity, and the ability to modify surface chemistry [9]. Agricultural waste materials, such as sugarcane bagasse, have shown promise as potential adsorbents due to their abundance, low cost, and favorable adsorptive properties when activated [10]. Additionally, this approach transforms waste into valuable products, helping to reduce waste-related issues.

On the other hand, an effective adsorbent typically possesses key attributes such as a large surface area, high pore volume, small pore size, and the presence of chemical functional groups like oxygen, nitrogen, and sulfur [11]. These properties can be tailored or enhanced using various preparation techniques, including physical activation, chemical activation, hydrothermal carbonization, combined physical-chemical activation, and biological activation [12,13]. Among these, chemical activation is the most widely used method for producing carbon-based adsorbents due to its simplicity and effectiveness in achieving the desired properties. Chemical activation of sugarcane bagasse, particularly using sulfuric acid, has been investigated to enhance its efficacy in removing caffeine from contaminated water [14].

The objective of this study is to analyze the effects of chemical activation on the pore structure and functional properties of activated carbon derived from sugarcane bagasse, utilizing Low Voltage Scanning Electron Microscopy (LVSEM) and Fourier Transform Infrared Spectroscopy (FTIR) for characterization. The caffeine removal efficiency will be assessed through batch adsorption experiments, examining various parameters such as adsorbent dosage, contact time, and stirring rate.

2. Methodology

2.1 Sample Preparation

The sugarcane bagasse used in this study was collected from a local market in Setapak, Kuala Lumpur, Malaysia. The raw sugarcane bagasse (RSB) was cut and washed thoroughly with distilled water to remove dirt and impurities. The cleaned bagasse then dried in an oven at 105°C for 24 hours to reduce the moisture content. After drying, the bagasse was ground into a fine powder using a grinder and sieved to obtain a particle size of 200 micrometers (µm).

2.2 Chemical Activation of Adsorbent

The activation of sugarcane bagasse involved chemical activation using sulphuric acid (H_2SO_4) as the activating agent. The pretreated sugarcane bagasse was impregnated with a concentrated

sulphuric acid solution at a ratio of 1:1 by weight. The mixture was soak for 3 hours and filtered, washed several times with distilled water until reached pH neutral. Activated sugarcane bagasse then dried in an oven at 105°C for 3 hours. The dried activated carbon sugarcane bagasse (ACSB) was then subjected to carbonization in a muffle furnace at 400°C for 4 hours [15].

2.3 Characterization of Adsorbent

The activated carbon was characterized using the Low Voltage Scanning Electron Microscopy (JEOL IT300LV) to analyse its surface morphology and pore structure. The functional groups present on the surface of the activated carbon were identified using Fourier Transform Infrared Spectroscopy (PerkinElmer Frontier 104968). These characterization techniques provided insights into the structural and functional properties of the activated carbon, which are crucial for understanding its adsorption capabilities.

2.4 Adsorption Test

Removal efficiency of caffein using the prepared activated carbon was evaluated through batch adsorption test. A series of adsorption tests were conducted by varying the adsorbent dosage, contact time, and stirring rate. The initial concentration of the caffeine solution was fixed at 0.2 M. For each test, a known amount of activated carbon (0.10, 0.15, 0.20, and 0.25 g) was added to 100 mL of caffeine solution in a conical flask. The mixture was agitated at a constant stirring rate (50, 100, 150, and 200 rpm) at 30 ℃. Samples were taken at time intervals (10, 30, 50, and 70 minutes) and filtered to separate the adsorbent from the solution. The residual caffeine concentration in the filtrate was determined using UV-Vis Spectrometry (PerkinElmer Lambda 25). The experiments were conducted in triplicate to ensure the accuracy of the results. The percentage of caffeine removal were calculated using Beer Lambert Law below:

$$A = \varepsilon c l \tag{1}$$

Where, $A, \varepsilon, c,$ and l are absorbance (unitless), molar absorptivity of compound or molecule in solution (M^{-1} cm^{-1}), concentration of solution (M), and path length (usually 1 cm) $Co - Ct$, respectively.

$$qt = \frac{(Co-Ct)V}{m} \tag{2}$$

$$\%Removal = \frac{(Co-Ct)V}{Co} \times 100 \tag{3}$$

Where qt, Co, Ct, V and m are adsorption capacity, initial adsorbate concentration (mg L^{-1}), final adsorbate concentration (mg L^{-1}), volume of adsorbate (L), and mass of adsorbate (mg), respectively.

3. Result

3.1 Low Voltage Scanning Electron Microscope (LVSEM)

The surface morphology of raw and activated sugarcane bagasse was analyzed using LVSEM, as depicted in Figure 1. The raw sugarcane bagasse displayed a dense skeletal structure with minimal visible pores, indicating a lack of significant porosity. In contrast, the activated sugarcane bagasse

exhibited a deformed, porous structure characterized by pores of various sizes. This transformation underscores the enhancement of the specific surface area and the adsorption capacity of the biochar, enabling it to effectively bind chemical compounds [16].

The development of microporosity in the activated sugarcane bagasse is attributed to the chemical activation and carbonization processes. These processes decompose the long-chain polymers and remove non-carbonaceous components through the release of volatile and moisture elements [14]. The use of sulfuric acid during activation plays a crucial role by facilitating the formation of active sites and significantly enhancing the porosity and surface area of the carbon. This improved surface morphology directly contributes to the material's performance as an efficient adsorbent.

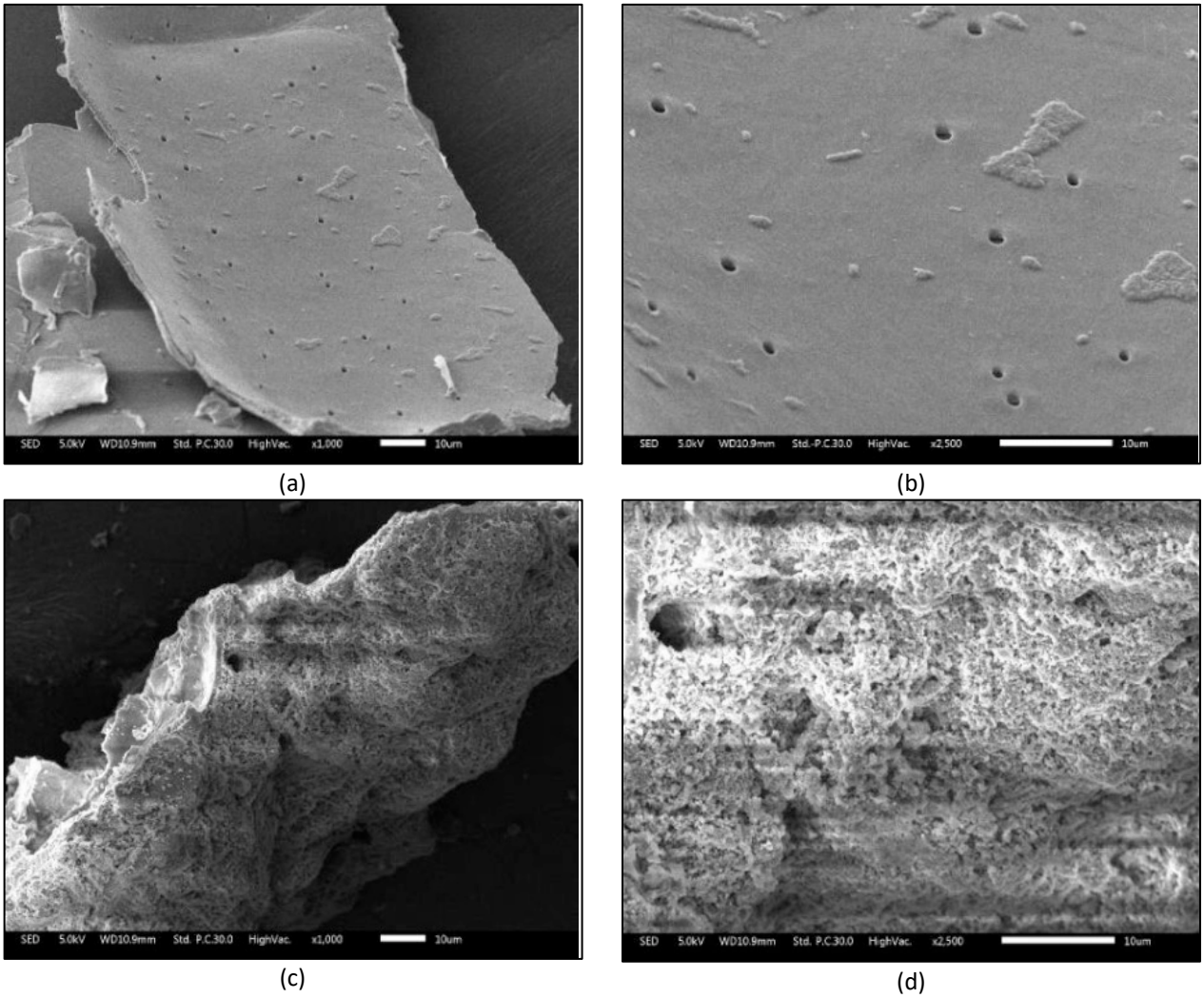

Fig. 1. Image LVSEM of at different magnifications: (a) RSB x 1,000; (b) RSB x 2,500; (c) ACSB x 1,000; (d) ACSB x 2,500

3.2 Fourier Transform Infrared Analysis (FTIR)

Figure 2 illustrates the surface functional groups of raw and activated sugarcane bagasse analyzed using FTIR spectroscopy, highlighting the chemical transformations that occur during the activation process. In Figure 2(a), the broad band around 3300 cm^{-1} corresponds to O-H stretching vibrations, likely originating from hydrogen bonding in alcoholic, phenolic, and hydroxyl groups present in the lignocellulosic biomass [17,18]. The peak near 2900 cm^{-1} is attributed to C-H stretching

vibrations of aliphatic compounds, commonly found in organic materials [19,20]. The reduction in intensity and disappearance of these peaks in Figure 2(b) can be attributed to the removal of moisture and the degradation of cellulose and lignin during chemical activation and carbonization [17].

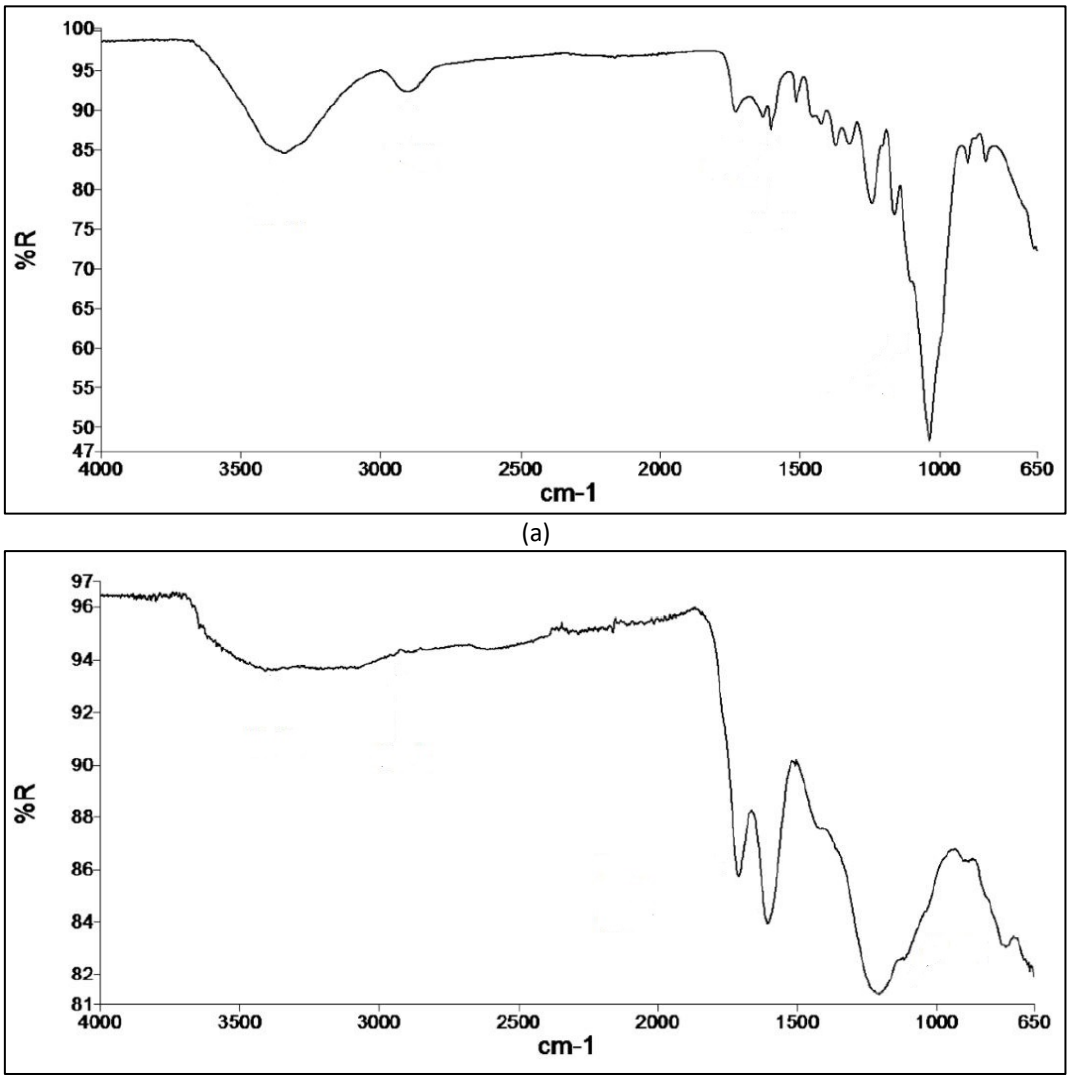

Fig. 2. FTIR spectra for: (a) raw sugarcane bagasse; (b) activated sugarcane bagasse

The sharp peaks observed between 1600–1700 cm^{-1} in Figure 2(b) correspond to C=O stretching vibrations, indicates the conjugated and unconjugated carbonyl groups, such as carboxylic acids or ketones. These functional groups are formed through the breakdown of hemicellulose and cellulose [15]. The bands in the range of 1300–1500 cm^{-1}, associated with C-H bonds from amorphous lignin and cellulose polysaccharides, are absent in the spectrum of activated sugarcane bagasse. This absence indicates the decomposition or alteration of lignin and other non-carbonaceous organic compounds due to sulfuric acid treatment and high-temperature carbonization [14].

The peak observed at 1000–1200 cm^{-1} in Figure 2(a), attributed to C-O stretching vibrations typical of cellulose, hemicellulose, and lignin, broadens in Figure 2(b). This broadening suggests the formation of more complex oxygenated functional groups, such as carboxyl or carbonyl groups, introduced during the chemical activation process [15].

These spectral changes signify the effective removal of non-carbonaceous components and the introduction of oxygen-containing functional groups, contributing to the development of mesopores [21]. The appearance of new functional groups confirms the success of the chemical activation process, improving the material's potential to interact with caffeine through adsorption mechanisms [22].

3.3 Adsorption Test

The adsorption process is regulated by factors such as absorbent dosage, stirring rate, and contact time. These parameters are used by the full-scale caffeine elimination therapy procedure. The calibration curve in Figure 3 depicts the relationship between absorbance and caffeine concentration, showing a linear trend with a correlation coefficient (R^2 = 0.9784). This high R^2 value confirms the reliability of the spectrophotometric method for quantifying caffeine concentrations in the solutions.

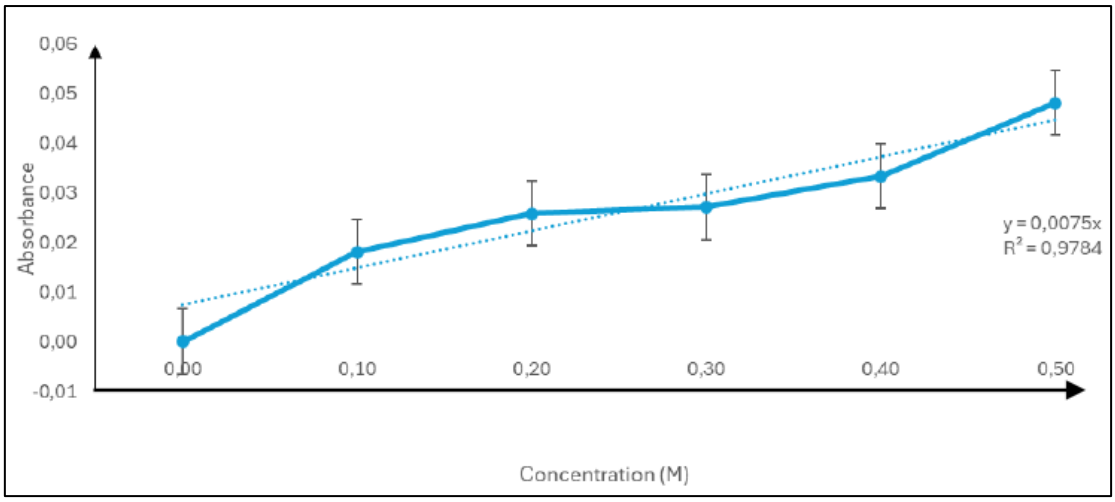

Fig. 3. Standard calibration curve of initial absorbance versus initial concentration of caffeine solutions

Figure 4 reveals the effect of adsorbent dosage on caffeine removal efficiency. For both coffee and tea solutions, increasing the dosage from 0.1 g to 0.25 g leads to enhanced caffeine removal. However, the tea solution exhibits significantly higher removal rates, achieving nearly 99% efficiency even at lower adsorbent dosages. In contrast, caffeine removal from coffee gradually increases, reaching approximately 82.86% at 0.25 g. This difference may stem from the variations in the chemical composition of coffee and tea, where tea potentially has fewer competing organic molecules or inhibitors, allowing more effective adsorption. The accumulation and clustering of caffeine within the porous structure of activated carbon are facilitated by hydrogen bonding and the presence of hydrophobic methyl groups [22].

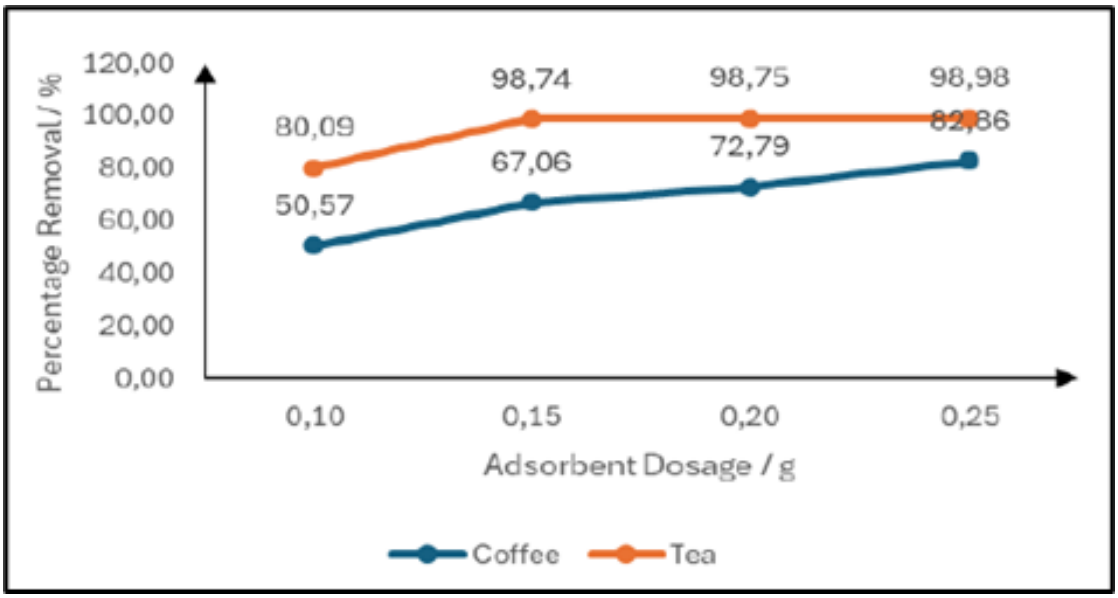

Fig. 4. Percentage of caffeine removal in coffee and tea against adsorbent dosage

The caffeine removal efficiency in tea become plateau which might be due to the absence of additional active sites for more adsorption [23]. The improved efficiency with higher adsorbent dosages can be attributed to the increased availability of active adsorption sites and greater surface area provided by the sugarcane bagasse-based activated carbon [17,24].

Figure 5 shows the effect of contact time on caffeine removal efficiency. Tea solution exhibits consistently high removal rates at 99% across all time intervals, indicating rapid adsorption equilibrium. On the other hand, coffee solution shows an initial increase, highest at around 30 minutes with 80.5% removal, followed by a slight decline at 70 minutes. Rapid increase in the caffeine removal efficiency for tea and coffee within the first 10 min because as the time of the adsorption process increases, it creates high interaction and contact chance between the adsorbent and caffeine molecules [23]. The adsorption process is dynamic, and as contact duration increases, so does the adsorption rate.

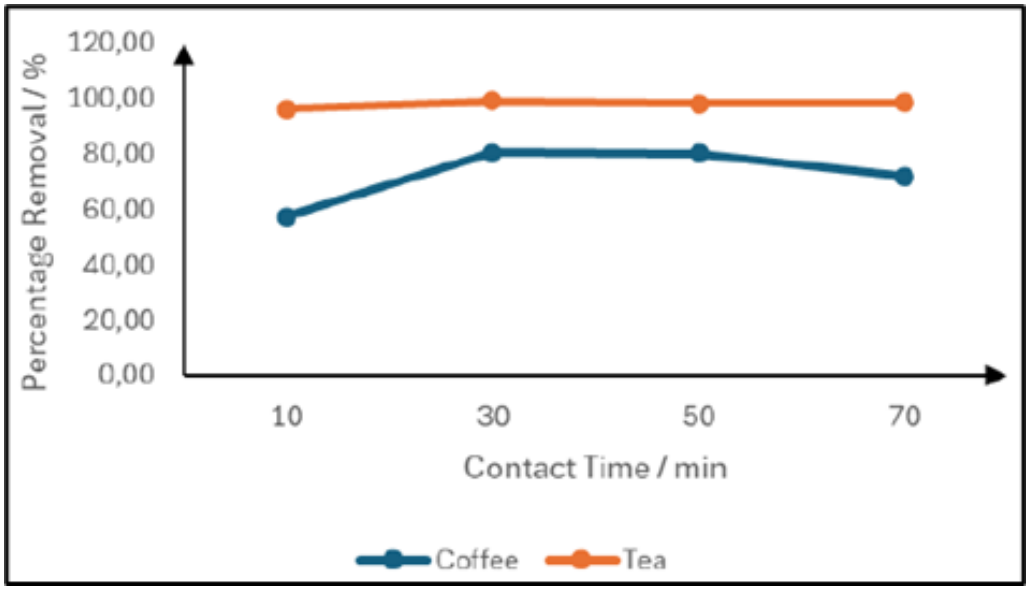

Fig. 5. Percentage of caffeine removal in coffee and tea against contact time

The observed decline in coffee solution might be due to desorption phenomena or the saturation of adsorption sites by competing compounds in the coffee matrix [25]. For tea solution, equilibrium was established at 30 min, the removal efficiency vs. time has shown a plateau which means that the system has reached the equilibrium. The rapid equilibrium in the tea solution suggests that the adsorbent's surface properties and the caffeine's molecular interaction are highly favorable. Beyond this stage, extending the contact time does not result in a significant improvement in the removal percentage of caffeine molecules [26].

Figure 6 illustrates the effect of stirring rate on caffeine removal efficiency for both coffee and tea solutions. For the tea solution, caffeine removal remains consistently high at 98% regardless of stirring speeds. This indicates that mass transfer resistance is minimal under the tested conditions, suggesting efficient adsorption kinetics and stable adsorbate-adsorbent interactions regardless of agitation intensity [25]. In contrast, the coffee solution demonstrates an optimal stirring rate of 100 rpm, where highest caffeine removal efficiency at 72%. Beyond this point, caffeine removal efficiency decreases at higher stirring speeds, such as at 200 rpm. This decline can be attributed to turbulence effects that disrupt the adsorption equilibrium, potentially causing desorption of adsorbed caffeine molecules or weakening adsorbate-adsorbent interactions [26]. These observations highlight a balance between agitation intensity and adsorption efficiency, where excessive turbulence may hinder the adsorption process.

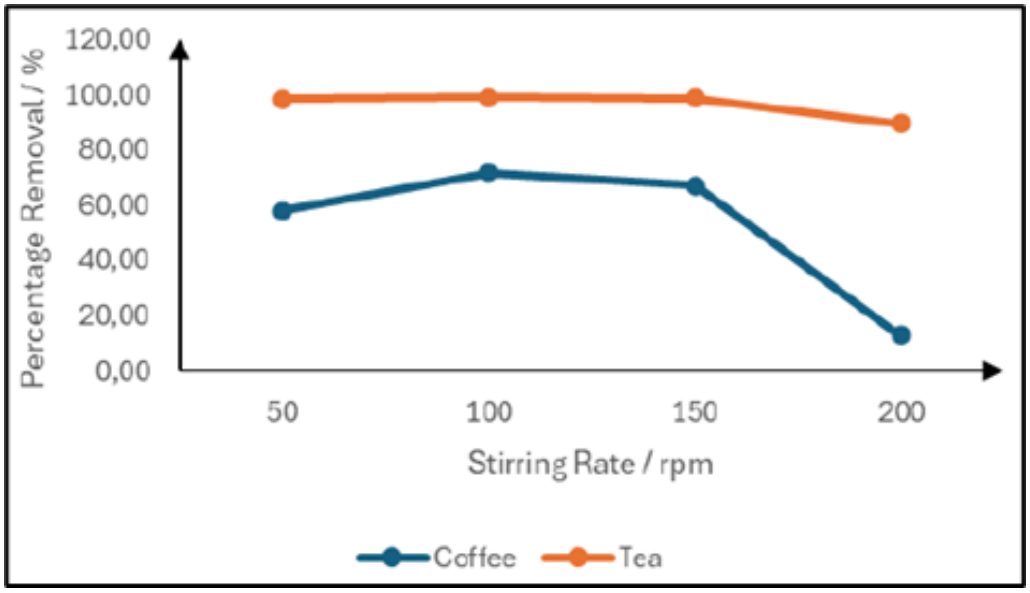

Fig. 6. Percentage of caffeine removal in coffee and tea against stirring rate

The increasing removal efficiency with stirring rate up to the optimal point can be explained by enhanced mixing and homogenization of the solution [27,28]. Higher stirring rates promote mass transfer of caffeine molecules to the adsorbent surface, increasing the likelihood of adsorption. Additionally, improved mixing results in more frequent collisions between caffeine molecules and the adsorbent, facilitating greater adsorption. However, beyond the optimal rate, the diminishing returns for coffee suggest that further agitation does not enhance adsorption and may, instead, interfere with the stability of the adsorption process [17,29].

4. Conclusions

This study successfully demonstrated the potential of sugarcane bagasse as an efficient and sustainable biosorbent for caffeine removal from contaminated water. Chemical activation using sulphuric acid significantly enhanced the adsorbent's performance by improving its porous structure and introducing functional groups conducive to adsorption, as evidenced by LVSEM and FTIR characterization. Batch adsorption experiments revealed the activated sugarcane bagasse's high efficiency, achieving a maximum caffeine removal of 99.12% for the tea solution within 30 minutes. However, for the coffee solution, the removal efficiency was lower compared to tea solution, with the lowest percentage observed at 12.77% under a stirring rate of 200 rpm, likely due to excessive turbulence disrupting adsorption equilibrium. The findings underline the feasibility of using agricultural waste materials like sugarcane bagasse as low-cost, eco-friendly alternatives to conventional adsorbents for water purification. This approach not only offers a sustainable solution for waste management but also contributes to environmental conservation and public health protection.

Future studies should investigate the effects of varying activation conditions, such as acid concentration, activation temperature, and duration, to further enhance the adsorbent's efficiency and tailor it for different pollutants. Detailed kinetic and isotherm studies for various pollutants should also be conducted to better understand the adsorption mechanisms and optimize operational parameters for industrial-scale applications. Finally, an economic feasibility and environmental impact assessment, including a lifecycle analysis, would provide valuable insights into the advantages of utilizing sugarcane bagasse compared to conventional methods. By addressing these areas, future research can validate and expand the applicability of sugarcane bagasse as a sustainable material for advanced water treatment technologies.

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