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A Comparative Study of COD Removal Efficacy from Pharmaceutical Wastewater by Areca Nut Husk Produced and Commercially Available Activated Carbons

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Authors' contributions

This work was carried out in collaboration among all authors. Author SA designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors FS and MMK managed the literature searches. Author MKU supervised and managed the analyses of the study. All authors read and approved the final manuscript.

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ABSTRACT

Pharmaceutical industries in Bangladesh are considered as one major industrial as well as environmental pollution problems which discharge a significant amount of organic contaminants in the environment hence require advanced treatment technologies to decontaminate pharmaceutical wastewater. In the present investigation, areca nut husk treated activated carbon (ANHC) was used as an adsorbent to remove chemical oxygen demand (COD) from pharmaceutical effluent as well as a comparative adsorption efficiency with commercial activated carbon (CAC) was performed.

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The batch experiments were carried out in a laboratory scale. The materials also evaluated for different adsorbent dosages and contact times. The experiment revealed a removal percentage up to 70% for ANHC and 90% for CAC for 3g of adsorbents in 180 min. The adsorption processes were satisfactorily described by pseudo-second-order (PSO) kinetic model which shows a better fitting with the maximum regression coefficient for both adsorbents. The results show that Langmuir model best described the experimental data with a highest correlation coefficient (R^2=0.9856 for ANHC and 0.9993 for CAC) compared to Freundlich model and the experimental data showed asorption capacity of 36.549 and 64.935 mg/g for ANHC and CAC, correspondingly. According to the adsorption studies, the results revealed that COD adsorption process followed by the monolayer chemisorption mechanisms. The results revealed that ANHC adsorbent is potentially low cost and environmental friendly adsorbent for the removal of organic matter from pharmaceutical effluent.

Keywords: Adsorption; chemical oxygen demand; pharmaceutical effluents; isotherms; kinetics; Bangladesh.

1. INTRODUCTION

Use of pharmaceutical products has been increased and became problem worldwide. Over 3000 active elements are commercially available and every year new elements are coming to markets [1,2]. But wastewater coming from this industry carries a lot of organic and inorganic pollutants [3,4]. This water mixes with our regular mainstream water flow and badly affects marine biota and human life. Pharmaceutical wastewater is a real threat to environment.As a result, different types of problems such as mutagenesis, endocrine disruption, antimicrobial resistance etc. occurred [5,6].

Pharmaceutical wastewater affects the drinking water and other water resources directly and indirectly [7,8]. For this reason, water scarcity will happen soon [9,10]. For treating wastewater, different methods are available such as coagulation, flocculation, sedimentation, membrane filtration, sand filters, oxidation process etc [7,11,8]. But all of these methods are very costly, complex, slow and need skilled peoples to operate [11]. Sometimes, these methods are not able to separate the pollutants completely [12,13,14].

On the other hand, adsorption is an effective method for treating wastewater. Because it can remove organic and inorganic substances without the forming of hazardous materials and easy maintenance [15]. Globally activated carbon used as adsorbents for its excellent adsorption quality [16]. But commercially it is very costly and complicated to manufacture [17]. So researchers searched for new adsorbents such as date palm seeds [18], rice husk [19,20,21], paper mill sludge [22], *Moringaoleifera* Lam. seed husks [15], peanut shell activated carbon [23], betal nut activated carbon [20], animal horns [24], coconut shell activated carbon and sugarcane bagasse fly ash [25], for replacing activated carbon.

The utilization of waste material as bio-absorbent is an eco-friendly technique. It is a way of minimization of agricultural waste and its utilization for the removal of COD. The natural bio adsorbents are cheap and available of vast quantities. One of them is areca nut husk which is present in large quantities in all parts of Bangladesh.

The objectives of this study were to compare the adsorption efficiency of areca nut husk treated activated carbon (ANHC) and commercially activated carbon (CAC) for the treatment of a pharmaceutical industrial wastewater for the reduction of COD.

2. METHODOLOGY AND MATERIALS

2.1 Sample Collection

The wastewater sample was taken from a pharmaceutical industry in Mirpur, Dhaka. The wastewater samples were collected from an industrial outlet in plastic bottles and preserved at 4˚C. The effluent was characterized using standard methods for water and wastewater. The Department of Environmental Science and Wazed Miah Science Research Centre, Jahangirnagar University Savar, Dhaka, provided materials and apparatus for this analysis.

2.2 Adsorbent Preparation

The husks of areca nut were collected from a tea stall and a stationary store. The areca nut husk (ANH) was collected in approximately 3 kg in amount and kept in a dust-free clean airtight polythene bag. The husks of the areca nut were cut into small pieces then washed several times with distilled water to remove dust.Then the areca nut husks were sun dried for seven days to remove moisture. To remove any remaining moisture, it was dried in a hot air oven at 60°C for 24 hours. This was confirmed by taking its initial and final weights. The husks were ground to powder using a blender after they had dried completely. To obtain fine particles, it was sieved with a 1mm sieve. After sieving, the powder was put in a crucible and burned for 1 hour at 350°C in a muffle furnace. The powder was cooled in desiccators for 1 hour before being collected and placed in an airtight clean glass container. Before being used as a source material for the preparation of adsorbent, this dried material was kept in desiccators. For the adsorption analysis, ANHC was used directly. As an adsorbent, only fine ash was used. Finally, the carbon powder was sieved to obtain a very fine carbon powder, which was then used as an adsorbent. Particles with a diameter of less than 1 millimeter were used.

2.3 Batch Adsorption Study

The adsorption studies were completed by looking at the adsorption of COD with AHNC and CAC. In which, CAC had powerful adsorption capacity of COD than ANHC.Adsorption of COD to its adsorbents was performed in batch operating condition. In a laboratory scale, all the experiment of Batch Adsorption study (BAS) were executed inside a conical flask of 250 ml including a solution of 100 ml COD solution. Initially, the analyses were carried out in 100 mL of pharmaceutical wastewater and the selective adsorbent dosages were (1.5 to 3 g/L) after the time interval (30-240 min). All flasks were put in a shaker incubator for 180 min at 120 rpm before they reached equilibrium. After refluxing digestion and titration, the COD of the sample was calculated after each pre-determined time period. Before applying adsorbent, the COD of the wastewater sample was determined. Since the solution of small test tubes contained some adsorbent particles, it was centrifuged at 1500 rpm for 2 minutes to separate the adsorbent particles. After that, whatman 44 filter paper was used to filter the sample. After centrifugation, a micro pipette was used to take 2 mL of solution from a small test tube and prepare it for digestion.

2.4 Efficiency Analysis of Adsorbents

Percentage of removal of COD concentration calculated by following equation:

$$\text{Removal \%} = (C_o - C) / C_o \times 100 \quad \ldots\ldots\ldots\ldots (1)$$

Where, C_o is the initial concentration of COD (mg/ L) of the wastewater sample and C is the final concentration of COD (mg/ L) after addition of adsorbent.

2.5 Statistical Analysis

Each set of the experiment was repeated for three times (in batch sorption experiment). To establish statistical significance, the data were analyzed using the Student's t test in with a p value below 0.05. Graphpad Prism, Version-8.0.2 software was used for all statistical analysis of operating parameters (Inc. CA USA). Isotherm and kinetic model both were evaluated to determine the correlation between experimental and simulated results through Origin pro 2019b.

3. RESULTS AND DISCUSSION

In this study, ANHC were used as adsorbent for removal COD from pharmaceutical industry due to it is cheap, efficient, fibrous. For assessing COD removal efficiency thermally treated areca nut husk has been used. In this chapter, adsorption capacity was analyzed based on contact time and adsorbent dosage. Then adsorption capacity of treated areca nut husk was compared with commercially activated carbon. In order to establish the optimum conditions of operating in full-scale batch process for removal of COD from pharmaceutical industry by adsorption onto areca nut husk.

3.1 Initial Characteristics of Pharmaceutical Wastewater

Pharmaceutical wastewater has a diverse composition, with a high content of organic matter, microbial toxicity, high salt, and a challenging degradability [26]. Furthermore, most pharmaceutical factories operate on a batch method, with diverse raw materials and manufacturing processes, resulting in a wide range of wastewater. The properties of pharmaceutical wastewater are summarized in Table 1.

Pharmaceutical wastewater comes in a variety of forms, each with its own set of characteristics. Biopharmaceutical wastewater has a high chromaticity, a low C/N ratio, a high BOD, COD concentration, a high sulfate concentration, a complex structure and biological toxicity. Chemical pharmacy is low in protein, difficult to biodegrade, and harmful to microbes, as well as having a high salt content [27].

3.2 Comparative Study of COD Removal by ANHC and CAC

Fig. 1(A) compares the removal efficiency of COD by ANHC and CAC. The percentage removal of COD reached 75% and 50% in 30 minutes and reached equilibrium in 240 minutes, indicating both high removal efficiency for both ANHC and CAC respectively. At equilibrium, the ANHC and CAC had the highest COD removal efficiency of 71.08% and 91.67%, respectively.

In Fig. 1(B) show that the highest removal was found for 3g dosage.The commercially activated carbon had higher removal capacity than thermally treated areca nut shell adsorbent with increasing contact time CAC (91.67%) > ANHC (71.08%).The lowest the organic matrix, the larger the surface area. Organic matrix was present in large amount in the raw of adsorbent, so that, the surface area became low which caused less adsorption capacity to remove COD. Thermal treatment (ash) of areca nut shell destructs the organic matrix that causes an increase in the surface area for adsorption of COD.

Various studies reported that the finer the ashes of natural products, causes decreasing particle sizes which caused less adsorption capacity to remove COD (Rayhanet al., 2016); [28]. On the other hand, activated carbonate are free from organic matrix which makes it better adsorbent than any other natural adsorbents product [29]. CAC effectively raising the overall specific surface area, making it possible for contaminants to engage with active adsorption sites (Kara et al., 2006); [30].

3.3 Removal of COD at Different Experimental Conditions

3.3.1 Effect of adsorbent dosage

In sorption studies, the amount of adsorbent also matters a lot with changing the concentrations of adsorbent in a reaction mixture while maintaining the other parameters constant. The experiment was carried out with various levels adsorbent doses from 1.0 to 3.5 g/L at 28°C (301K) for 180 minutes and initial COD concentration 109.74 and 35.4 mg/L to test the effects of ANHC and CAC dosage on COD adsorption.

Fig. 2(A) depicts that increase in the amount of the adsorbent facilitated a rapid increase in the percentage removal of the COD. This is due to the availability of more sites which aided the adsorption; hence the optimum removal was achieved at 3g of the adsorbent. Another study shows that increasing adsorbent dosage more than the optimum the increase in adsorption is very less and it becomes cost ineffective it is due to aggregation of adsorbent particles that leads to decrease in effective surface area of adsorbent available for adsorption (Samiksha et al. 2013); [31]. Fig. 2(B) shows the reduction of COD concentration with adsorbent dosages.

3.3.2 Effects of contact time

The current study focused at the removal of COD by ANHC and CAC as a factor of contact time (0–240 min) and adsorbent dosage (3 g/L) (Fig. 2C). From the beginning to 30 minutes, the adsorption was extremely rapid. The adsorption kinetics decreased as time passed, and in all situations, the adsorption reached equilibrium within 180 minutes. There were no remarkable changes in COD removal efficiency after equilibrium time for each adsorbent types.

This phenomenon can be attributed to the fact that a large number of vacant surface sites are available for adsorption at the initial stage, and after a lapse of time, the remaining vacant surfacesites are difficultto be occupied due to repulsive forces between the solute molecules on the solid and bulk phases. It also showed that removal efficiency increased with increasing contact time. This result is similar to (Samikshaet al. 2013); Rayhan et al., 2016; [32,33,34].

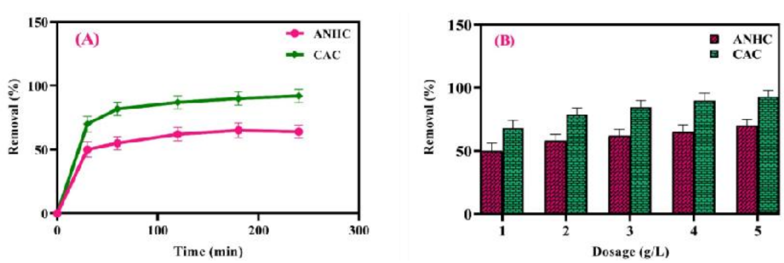

Fig. 1. Comparative study of COD removal efficiency with contact time
(A) and adsorbent dosages (B) for ANHC and CAC respectively

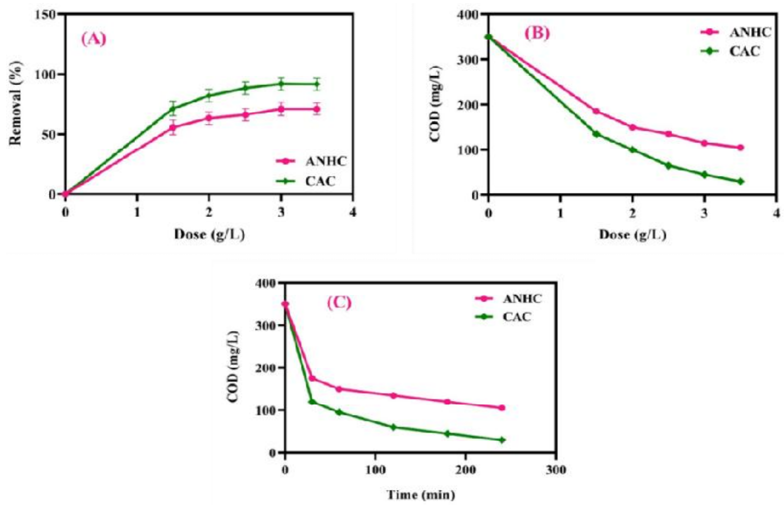

Fig. 2. Effect of ANHC and CAC on removal (%) and reduction concentration of COD

(A) Removal efficiency of COD with adsorbent dosages; (B) Reduction concentration of COD with adsorbent dosage; (C) Effect of contact time on reduction of COD(Temperature: 28^0C, contact time: 180 min, dosage: 3 g/L, initial concentration: 109.74 and 35.4 mg/L)

Table 1. Physicochemical properties of raw pharmaceutical wastewater before adsorption

COD (mg/L)	TDS (mg/L)	DO (mg/L)	Temperature (^{0}C)	P^H
354.67	173.3	3.4	29.3	7.2

3.4 Adsorption Modelling

3.4.1 Adsorption isotherm models

The equilibrium relationship between COD and adsorbents (ANHC and CAC) was described using the Langmuir and Freundlich isotherm models. The COD sorption model's parameters onto both adsorbents are reported in Table 2.

3.4.1.1 Langmuir isotherm model

The Langmuir model equation in linear form is stated in Fig. 3(A, B). The experimental results and Langmuir isotherm models for COD have a strong correlation, R^2, indicating that both ANHC (0.9856) and CAC (0.99929) respectively, which demonstrating the monolayer surface coverage of COD sorption. The maximum adsorption capacities (q_{max}) for ANHC were 36.95 mg/g and 64.94 mg/g for CAC.

The key feature of the Langmuir equation is the equilibrium parameter (R_L), which is a non-dimensional constant that indicates the form of isotherm represented as-

$$R_L = \frac{1}{1 + C_i K_L}$$

Here, Ci is the initial concentration, and K_L is the Langmuir constant. Unfavorable adsorption is indicated by a value of R_L> 1, linear adsorption is indicated by a value of R_L = 1, ideal adsorption is indicated by a value of $0<R_L<1$, and irreversible adsorption is indicated by a value of R_L = 0. ANHC had R_L values of 0.489, while CAC had R_L values of 0.276. This shows that COD adsorption on ANHC and CAC is feasible. Because of the high correlation coefficient values, the Langmuir isotherm model was better fitted with the experimental results.

3.4.1.2 Freundlich model

Fig.3 (C, D) reveals the Freundlich isotherm in its linear form on plots of ANHC and CAC. Table 2 shows that the Freundlich model was not suitable for COD adsorption for both adsorbents due to lower correlation coefficient values (R^2 = 0.95421for ANHC and R^2 =0.97362 for CAC) compare to Langmuir correlation co-efficient values. Assume that 1/n denotes the adsorption process type: An irreversible process is 1/n = 0, an adsorption equilibrium state is $0<\frac{1}{n}<1$, and a collaborative adsorption state is 1/n > 1. 1/n = 1.8159 for ANHC and 1/n =0.9183 for CAC, which indicating both collaborative and preferential adsorption respectively. The Freundlich constant (K_F) for ANHC was determined to be 0.0012 mg/g and 0.1897 mg/g for CAC. These findings suggest that the Freundlich isotherm was ineffective for both adsorbent for COD adsorption.

3.4.2 Adsorption kinetic model

The study of adsorption kinetics, which describes the adsorption capacity, is the most important component used during the development of a process. The kinetics of adsorption are influenced by the adsorbent's physical and chemical properties, as well as the mass transfer process [35,36]. The kinetic models of COD adsorption were predicted using experimental data. The adsorption statistical analyses were performed by pseudo-second-order (PSO) kinetic models throughout this research.

3.4.2.1 PSO kinetic model

The PSO kinetic model largely represents the chemisorption mechanism of the adsorption process. In Fig. 4 (A, B) a linear equation was plotted against time with a slope of $1/k^2q_e^2$ and an intercept of $1/q_e$. The plot's experimentally determined the constants (q_e and k_2) are summarized the theoretical data implied by this model in Table 3. The PSO kinetics model shows a higher regression coefficient (R^2> 0.99) for all concentrations and close values between q_e, exp and q_e, cal, according to the findings.

In order words, the data also show good compliance with the proposed pseudo-second order equation. Indeed, the regression coefficients for the linear plots were higher than 0.999 for both systems in these studies. As a result, the PSO kinetic model performed better than the other two models in terms of fitting the experimental data and it confirms the chemical functionalization of COD removal by ANHC and CAC adsorbents. This result is similar to Emmanuel et al. (2008).

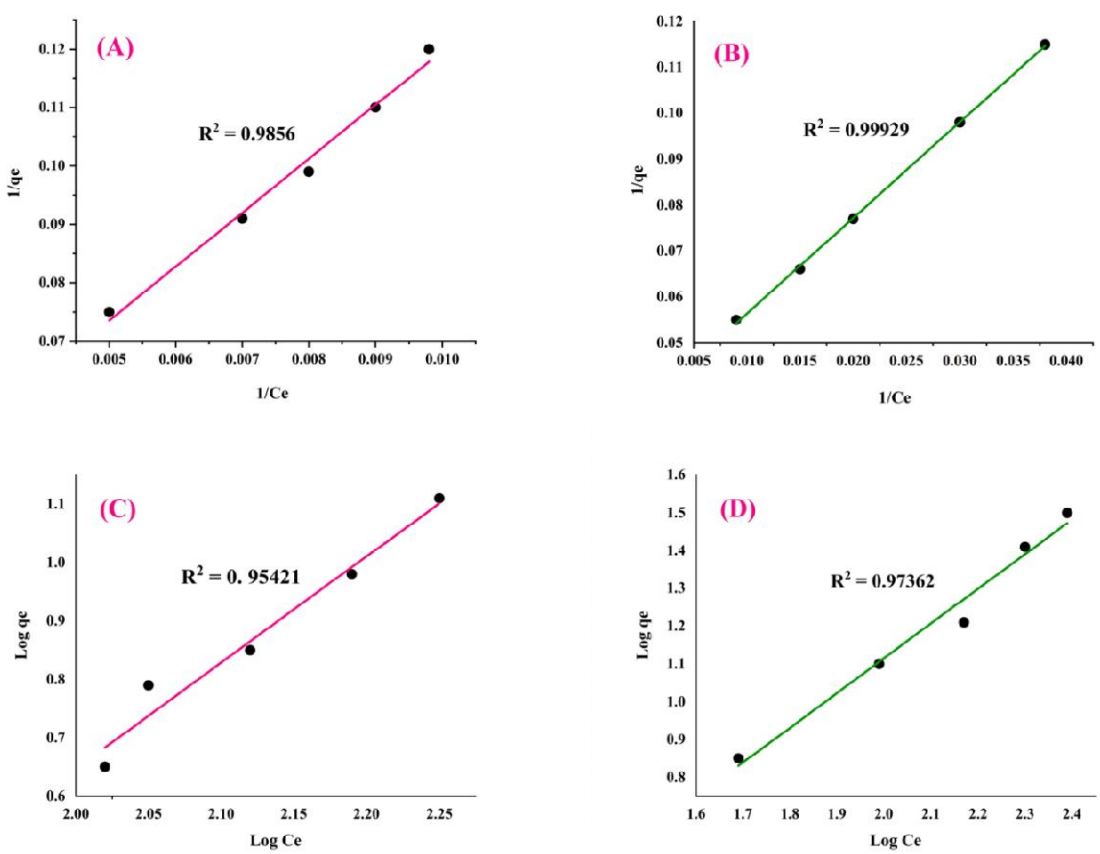

Fig. 3. Isotherm plots for adsorption of COD onto (B, D) ANHC and (A, C) CAC at 109.74 and 35.4 mg/L COD concentrations and 3 g/L adsorbent dosage; (A, B) Langmuir model; (C, D) Freundlich model

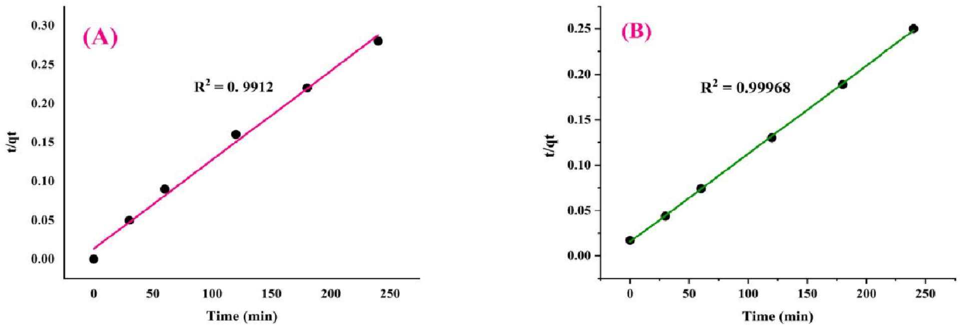

Fig. 4. Pseudo second order (PSO) kinetic plot for adsorption of COD onto (A) ANHC and (B) CAC at 109.74 and 35.4 mg/L COD concentrations and 3 g/L adsorbent dosage

Table 2. COD adsorption isotherm constants on ANHC and CAC

Adsorbents	Isotherm Model							
	Langmuir				Freundlich			
	K_L (cal) (mg/g)	R_L (L/mg)	q_{max} (mg/g)	R^2	K_F (cal) (mg/g)	n	1/n	R^2
ANHC	0.00296	0.4875	36.549	0.9856	0.0014	0.5506	1.8159	0.9542
CAC	0.00738	0.2762	64.935	0.9993	0.1897	0.0889	0.9189	0.9736

Table 3. COD adsorption Kinetic constants on ANHC and CAC

Adsorbents	Kinetic Model				
	Experimental data		Pseudo second order (PSO)		
	COD (mg/L)	q_e(exp) (mg/g)	qe (cal) (mg/g)	K_2 min-^{1}	R^2
ANHC	109.74	35.085	40.783	0.04724	0.99116
CAC	35.4	62.973	66.667	0.01452	0.99968

4. CONCLUSION

Experimentally, the reduction of COD from wastewater using commercially activated carbon and areca nut husk treated activated carbon as adsorbents was determined. The wastewater of pharmaceutical industry is highly contaminated having higher COD value, as adsorbent dose increases adsorption increases due to the availability of free sites of the particles. Despite the fact that CAC has higher reduction efficiency overtime than ANHC and it is more costly.COD removal by ANHC has a trend of up to 70% which is comparable to CAC's 90% performance. The problems that occur with the disposal of exhausted adsorbent can be solved by activation, incineration, or disposal following proper treatment. The results revealed that ANHC was produced from agricultural wastes for removing COD from industrial wastewater could be a promising sustainable, cheap and environment friendly adsorbent with profound implications in water and wastewater treatment contaminated with COD.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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