Adsorption of Chloride Ions from Aqueous Solution Using Activated Carbon Obtained by Chemical Activation of Jute Stick

S. Tarannum, M. N. Abir, A. F. Mita, B. K. Banik, M. A. Hoque, and M. I. Kabir

Abstract The coastal belt of Bangladesh is severely impacted by salinity intrusion. One way to address this problem is to use efficient and low-cost materials as adsorbents to remove saline ions from water. Activated carbon (AC) is the most efficient adsorbent in water treatment among those materials. This study demonstrates the effectiveness of activated carbon, derived from jute stick (JS), as an adsorbent and its adsorption performance in removing salinity. Both column and batch adsorption tests were carried out to evaluate the performance of AC on the synthesized saline water. As part of the column adsorption process, five experimental filters with a continuous flow were set up for five different initial concentrations of chloride ions. The best two AC samples obtained from the batch adsorption process (out of 32 samples), each having triplicates, were selected for further analysis. In batch adsorption, the removal efficiency of activated carbon was evaluated for chloride concentrations ranging from 500–900 mg/L. It was found that AC impregnated with $ZnCl₂ (1:1)$ at 600 $^{\circ}$ C and FeSO₄ (1:1) at 700 $^{\circ}$ C showed the maximum removal efficiency of 48% for optimum dosing of 4.4 g/L and 46% for optimum dosing of 4.8 g/L respectively. As for the column adsorption process with continuous flow having the same initial chloride concentration as the batch experiment, AC impregnated with $ZnCl₂ (1:1)$ at 600°C and FeSO₄ (1:1) at 700°C showed removal efficiencies of 58.13% and 56.91% when subjected to initial chloride ion concentration of 500 mg/L.

- A. F. Mita e-mail: mita-cee@sust.edu
- B. K. Banik e-mail: bijit-cee@sust.edu

M. A. Hoque e-mail: ahoque-cee@sust.edu

M. I. Kabir e-mail: imran.kabir-cee@sust.edu

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S. Tarannum · M. N. Abir (B) · A. F. Mita · B. K. Banik · M. A. Hoque · M. I. Kabir Department of Civil and Environmental Engineering, Shahjalal University of Science and Technology, Sylhet, Bangladesh

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1 Introduction

Bangladesh has an abundance of water, but almost one-fourth of the population lacks access to clean water for drinking. People in the coastal region are at risk of saline water due to climate change, rising sea levels, population growth, and decreasing freshwater supply. Direct medical issues, including hypertension and preeclampsia, skin conditions, acute respiratory infections, diarrheal disorders, and the transmission of mosquito-borne diseases, are all linked to an increase in water salinity [\[1](#page-9-0)]. According to a World Health Organization (WHO) study, the concentration of chloride in the main or second aquifer in most coastal areas ranges from 103 to 12,433 parts per million (ppm) during the dry months and 34 to 11,366 ppm during the wet season. Sodium chloride might taste salty; however, calcium or magnesium chloride is usually identifiable by taste up to 1000 mg/L [\[2](#page-9-1)]. Reverse osmosis, electrodialysis, ion exchange technology, and thermal technology are examples of conventional desalination methods that are effective in removing salt from drinking water but are unsuitable for rural communities [[3\]](#page-9-2).

Bangladesh is a country with a large agricultural sector and produces a lot of agricultural trash every year, especially jute sticks, which are made by separating the fibers [\[4](#page-9-3)]. Jute stick, a typical agricultural by product in most Asian countries, was used as a precursor for chemical and physical activation methods to make activated carbon [[5\]](#page-9-4). Activated carbon can be used to eliminate chlorine [[6](#page-9-5)]. Activated carbons are effective adsorbents for purifying drinking water and wastewater as well as extracting hazardous components from gases [[7\]](#page-9-6). Adsorption by activated carbon is the most popular method due to its highly developed interior surface area and porosity, as well as its straightforward design and simplicity of use [[8\]](#page-9-7). The fundamental goal of this work is to develop workable desalination methods utilizing adsorbents made from materials that are widely available locally to reduce the scarcity of safe drinking water. Moreover, the performance of utilized adsorbents is determined by two parameters in the adsorption process: maximum adsorption capacity and maximum elimination of contaminants; to examine the adsorbent for various parameters utilizing both batch and column adsorption techniques.

2 Materials and Methods

2.1 Sample Preparation

Producing activated carbon from jute stick with high cellulose and lignin content consists of steps like washing, pretreatment, impregnation, activation, and acid leaching to get carbons with micropores and high specific surface area. The steps of processing have been described accordingly.

2.2 Washing of Raw JS

At first, the jute sticks are washed thoroughly with water to remove dirt and other contaminants. Then they are oven-dried at about 110 \degree C for 24 h to eliminate excess water content.

2.3 Pre-treatment of JS

The JS are ground with a mixer grinder and sieved through ASTM #30 sieve to achieve a consistent particle size of 0.6 mm, which was later used to produce granular activated carbon.

In this study, about 20 g of granular jute stick is added with 660 ml of 0.25 M, 0.5 M and 1 M sodium hydroxide solution. A portion of the sample has not been mixed with sodium hydroxide to further investigate the effect of base leaching on the produced AC. According to studies done on similar lignocellulosic materials, this solution provides a minimum amount of NaOH to produce silicate with $SiO₂/NaOH$ ratio $= 1$ [[9\]](#page-9-8). JS powder is subjected to immersion in NaOH solution for 1 h with constant stirring on a magnetic stirrer hotplate. After the base solution is drained, JS powder is rinsed with deionized water until the pH reaches 7. Since the $Na₂SiO₃$ formed is soluble in water, it can be easily removed by water washing. After washing, the leached jute stick is oven-dried at 110 °C for 24 h. to remove excess water.

2.4 Impregnation of JS for Activation

To activate the leached JS, two different samples have been prepared by mixing 200 ml of 10% (w/w) ZnCl₂, and 10% (w/w) FeSO₄ as activating agents with 20 g base leached JS in each case. Impregnation is carried out at 70 °C in a thermostatic water bath for all two samples until excess water gets evaporated.

2.5 Carbonization of Impregnated JS

For carbonization, the impregnated JS is inserted into a stainless-steel tubular reactor 6 inches in length and 2 inches in diameter. After placing JS into the reactor, an inert atmosphere is created using nitrogen gas. Then the reactors are placed into the furnace where carbonization is performed at 300 °C, 400 °C, 500 °C, 600 °C and 700 °C for 1 h.

2.6 Acid Washing of AC

For acid washing, the AC is immersed in a 3 M HCl solution. Then the slurry is magnetically stirred for 30 min at ambient temperature. After the acidic solution is drained, the samples are washed with hot distilled water several times until neutralization. The washed samples are dried at 100 °C for 24 h. and stored in zip-locked bags for further use.

At the end of this step, thirty types of activated carbons are obtained, characterized by three concentrations of the base (0.25 M, 0.5 M, and 1 M), two activating agents (ZnCl₂ and FeSO₄), and five activation temperatures (300 °C, 400 °C, 500 °C, 600 °C and 700 °C). These samples are then used as adsorbents in the preparation of filter media.

2.7 Adsorption Process

As a part of the experimental work on a laboratory scale, two types of adsorption techniques have been adopted:

- . Column adsorption with continuous flow
- . Batch adsorption in suspension

2.8 Experimental Setup for Column Adsorption

For the removal of salinity, the performance of produced AC as an adsorbent is checked on an experimental basis. Five different filtration units have been established for column adsorption, having chloride concentrations of 500, 600, 700,800, and 900 mg/L respectively. Each of the filters is set up using a total of 10 types of AC samples containing triplicate for each to conduct the filtration process in laboratory scale. The filtration is carried out for 21 days to check the efficiencies of the adsorbents.

2.9 Experimental Setup for Batch Adsorption

The adsorption capacities of 32 types of activated carbons are checked in suspension for dosing of 2 g/L, 4 g/L, 6 g/L and 8 g/L. Among them, the top two activated carbons obtained from using different activating agents have been selected for further analysis. Suspension batches are made by mixing the required amount of sample with 1L saline solution of initial chloride concentrations of 500 mg/L, 600 mg/L, 700 mg/ L, 800 mg/L, and 900 mg/L ensuring a contact time of 180 min.

Figure [1](#page-4-0) shows the flow chart of the significant steps regarding activated carbon production and the adsorption process.

3 Results and Discussions

The quality of the JS derived activated carbon is influenced by pre-carbonization conditions, type of activating agent, impregnation ratio, inert atmosphere, activation temperature, and activation time. As such, these factors need to be carefully controlled to obtain activated carbon with the desired adsorption properties.

In this section, the adsorption capacities of different adsorbents have been summarized. Also, the effect of activating agent, concentration of base, different concentrations of ion, and temperature have been discussed. In addition to this, the effect of dosing and effect of temperature on suspension have also been described.

Table [1](#page-5-0) represents the notation and salinity removal efficiency of derived ACs using column and batch adsorption. From Table [1,](#page-5-0) it has been found that AC with

Fig. 1 Overview of the study

Notation	Initial Salinity (g/L)	Final salinity (g/L)		Removal efficiency $(\%)$	
		Column adsorption	Batch adsorption	Column adsorption	Batch adsorption
$FS(7)-5$	0.82	0.35	0.44	56.91	46.34
$FS(7)-6$	0.99	0.44	0.53	55.89	46.46
$FS(7)-7$	1.15	0.50	0.62	56.52	46.09
$FS(7)-8$	1.32	0.60	0.72	54.80	45.45
$FS(7)-9$	1.48	0.68	0.81	54.05	45.27
$ZC(6)-5$	0.82	0.34	0.42	58.13	48.78
$ZC(6)-6$	0.99	0.44	0.51	55.89	48.48
$ZC(6)-7$	1.15	0.51	0.60	55.94	47.83
$ZC(6)-8$	1.32	0.60	0.71	54.80	46.21
$ZC(6)-9$	1.48	0.69	0.80	53.83	45.95

Table 1 Removal efficiencies of derived activated carbons

FS(7)-5, FS(7)-6, FS(7)-7, FS(7)-8, and FS(7)-9 represents AC with FeSO₄ (1:1) at 700 °C for chloride concentration of 500, 600, 700, 800, and 900 mg/L respectively.

ZC(6)-5, ZC(6)-6, ZC(6)-7, ZC(6)-8, and ZC(6)-9 represents AC with ZnCl₂ (1:1) at 600 °C for chloride concentration of 500, 600, 700, 800, and 900 mg/L respectively.

ZnCl₂ (1:1) at 600 °C has the best salinity removal efficiency (58.13%). Hence the most efficient activating agent is $ZnCl₂ (1:1)$ for producing AC in Column adsorption.

3.1 Effect of Temperature for Different Dosing in Suspension

The samples carbonized at five different temperatures (300 °C, 400 °C, 500 °C, 600 °C, and 700 °C) show different thermal responses. ZnCl₂ (1:1) and FeSO₄ (1:1) produces AC most efficiently at 600 °C and 700 °C respectively. Figure [2](#page-6-0) exhibits that both $ZC(6)$ and $FS(7)$ have the best salinity removal efficiency at a dosing of 4 g/L. When the temperature is lower than 700 \degree C, the active reaction increases with the increase of temperature and a large number of pores are formed, so the specific surface area increases. However, when the temperature rises higher than 600 °C, the destruction of pores plays a principal role to decrease the specific surface area [\[10](#page-9-9)]. Despite using three samples for each case having a standard deviation value close to 0.01, in Fig. [2,](#page-6-0) FS(7) shows an increasing trend for 4 g/L and 6 g/L dosing, which is irregular. Further experiments are required using higher activation temperatures to evaluate the effect on removal efficiency for temperatures greater than 700 °C.

Fig. 2 Comparison of the effect of activation temperatures

3.2 Effect of Dosing for FeSO4 (1:1)

The best adsorption capacity of AC impregnated with $FeSO₄$ (1:1) at 700 °C has been found for a dosing of 4 g/L.

From Fig. [3,](#page-6-1) In case of 2 g/L dosing, the amount of adsorbent is not sufficient to remove the saline ion in a greater proportion. Thus, the removal efficiency becomes lower. Conversely, for a dosing of 8 g/L, the amount of adsorbent is more than required which causes the agglomeration of adsorbent particles. This results the decreased removal efficiency of AC with $FeSO₄$ (1:1). From Fig. [3,](#page-6-1) the optimum dosing is found to be 4.8 g/L.

Dosing (g/L)

3.3 Effect of Dosing for ZnCl₂ (1:1)

The best adsorption capacity of AC impregnated with $ZnCl₂ (1:1)$ at 600 °C has been found for a dosing of 4 g/L.

From Fig. [4,](#page-7-0) In case of 2 g/L dosing, the amount of adsorbent is not sufficient to remove the saline ion in a greater proportion. Thus, the removal efficiency becomes lower. Conversely, for a dosing of 8 g/L, the amount of adsorbent is more than required which causes the agglomeration of adsorbent particles. This results in the decreased removal efficiency of AC with $ZnCl₂$ (1:1). From Fig. [4](#page-7-0), the optimum dosing is found to be 4.4 g/L.

3.4 Analysis of Carbon Yield

The yield of AC with different activating agents at three temperatures are calculated by Eq. (1) (1) .

$$
Carbon yield (\%) = (Mass of activation / Mass of dried ricehusk) \times 100
$$

$$
\tag{1}
$$

For two chemical activating agents and three base concentrations, the yields of carbon decreases with the increase of reaction temperature as shown in Fig. [5](#page-8-0). By increasing the reaction temperature, the gasification becomes severe, and ultimately leads to a reduction in the yield.

Figure [5](#page-8-0) also shows that the carbon yield for 0.25 M base concentration is higher than 0.5 base concentration. Similarly, 0.5 M base concentration has a higher value of yield compared to 1 M base concentration. This is because the lignin tissue is removed after base-leaching of the JS, and this causes a decrease in the carbon yield.

4 Conclusion

The porous structure of AC can be optimized with the alteration of the activation parameters such as the type of activation (physical and chemical), choice of activating agent, activation temperature, impregnation ratio, and activation sequence. The optimization of the activation parameters yields the desired physicochemical properties and consequently the highest adsorption capacity. AC impregnated with ZnCl₂ (1:1) at 600 °C has the best salinity removal efficiency (58.13%) for 500 mg/L in column adsorption process with continuous flow. With increasing concentrations of saline water, removal efficiency slightly decreases. AC impregnated with ZnCl2 $(1:1)$ at 600 °C shows the maximum removal efficiency of 48% can be obtained for an optimum dosing of 4.4 g/L in suspension. 1 M NaOH is the best for base leaching of granular JS among the concentration of 0.25 M, 0.5 M, and 1 M NaOH.

AC impregnated with $ZnCl₂$ and $FeSO₄$ represents the best adsorption capacity at 600 °C and 700 °C among the five activation temperatures.

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