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Characterization studies of activated carbon prepared from agricultural wastes of *Vachellia nilotica*

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Key words: *Vachellia nilotica*, Adsorption, Langmuir, Freundlich isotherm, Activated carbon

Abstract

In this study, *Vachellia nilotica* was utilized to formulate initiated activated carbon (AC), which was then incubated with potassium hydroxide at 600°C for two hours. FTIR, TGA/DTA, and SEM/EDAX were used to characterize the functional groups, surface shape, thermal stability, and elemental identification of the activated carbon. Furthermore, the efficacy of the AC remediation was evaluated using dye (Congo Red) under different conditions, such as pH and dose, and the maximum amount of Congo Red that the adsorptive material could absorb was measured using the Langmuir and Freundlich adsorption isotherm principles. The Langmuir isotherm model fits the maximal dye adsorption capacity well, which was attained at 24.35 g/L. In order to improve (raise) the water's potability level, the research's findings suggest that *Vachellia nilotica* may be used as a cost-effective and capable raw material to create initiated carbon for wastewater treatment.

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Introduction

In recent years, the dye industry has been rapidly developed. According to the United States" Color Index", commodity dyes have reached tens of thousands. About 60,000 tons of dyes are discharged into the environment in the form of waste each year worldwide (Cheng *et al*., 2003) 80% of which are azodyes. Toxic substances present in dye water enter into the food chain of humans and several diseases are caused. This became a worldwide concern, because of emerging various diseases. Toxic substances liberated from industries in the form of heavy metals can damage the central nervous system, energy level, and vital organs. Long-term effects will lead to neurological and degenerative processes. Some toxic compounds in dye can damage human cells and leads to cancer (Jaishankar *et al*., 2014).

Various treatment methods have been used for the removal of dyes from industrial wastewater, including membrane filtration, electrochemical degradation, bioremediation, biodegradation, coagulation/flocculation, chemical precipitation, chemical oxidation, ozonation and photocatalysis (Rashid *et al*., 2018; Ozer *et al*., 2012; Lina *et al*., 2018; Inthapanya *et al*., 2019). However, adsorption process is a well-established and powerful technique for treating industrial wastewater due to high efficiency, flexibility and simplicity of design, convenience of operation and low cost of appropriate adsorbent materials (Rashid *et al*., 2018; Ozer *et al*., 2012; Lina *et al*., 2018; Inthapanya *et al*., 2019; Uner *et al*., 2016).

Recently activated carbon has been used in many purification and adsorption procedures. Water is no exception. Activated carbon is a carbonaceous, highly porous substance that can attract and retain organic chemicals. It is meant to have small and low-volume pores that would increase the surface area existing for adsorption or chemical reactions (Hagemann *et al*., 2018).

There are two processes for the preparation of activated carbon physical activation and chemical activation. In chemical activation, a raw material is impregnated with an activating reagent such as $ZnCl_2$, H_3PO_4, KOH, etc., and the impregnated material is heated in an inert atmosphere. Chemical activation is preferred over physical activation owing to the higher yield, simplicity, lower temperature and shorter time needed for activating material, and good development of the porous structure (Li *et al*., 2010). The main objective of the present work is to study experimentally and theoretically the equilibrium isotherms of Congo red adsorption onto *Vachellia nilotica*-based activated carbon prepared by KOH activation.

Materials and methods

Materials

The *Vachellia nilotica* were collected from the place Thirunelveli. After collected, *Vachellia nilotica* was washed with distilled water to get rid of impurities, dried at sun light for seven days then in oven at 110^{o}c for 1 h. After drying the sample were crushed. Then the dried sample was placed in muffle furnace for 1 h at 600^{o}c, then the sample stored in plastic containers for further use.

Preparation of activated carbon

Carbon prepared from tapioca peel is further activated by using reagent KOH. The sample was soaked in 1M KOH in 1:1 ratio for 24 hrs followed by weighing the sample in order to know the impregnation of 1M KOH to the sample and is followed by activation in muffle furnace at temperature 350°C for 2 hrs. The carbonized material was washed with distilled water to remove alkalis and dried at 110°C for 1 h. Finally it was stored in tightly closed bottles.

Characterization of activated carbon

The phase state of sample determined by X-ray Diffraction (XRD), which indicated the amorphous nature of the prepared material. The surface functional groups were detected on Fourier transforms spectrometer (FT-IR). The morphology of the AC was obtained with a field emission scanning electron microscope coupled with the EDX analyzer. Thermogravimetric analysis was performed in a thermo gravimetric analyzer.

Preparation of adsorbate

A stock solution of 500 mg of adsorbate Congo red dye was made by dissolving the dye in 1000 mL of water. For the adsorption research, dilutions ranging from 10 to 70 mg/L were used to create the stock solution's necessary concentrations.

Adsorption studies

In order to perform tests on batch equilibrium, Congo red dye was made by dissolving dye in deionized water. The water and dye are added in the right proportion to get the desired concentration. In a conical flask, 1g of Tapioca peel activated carbon was added. Following that, ACVN was applied to dye concentrations ranging from 10 to 70 mg/L. Studies on equilibrium reveal that it takes 24 hours to reach the equilibrium point. The effect of an adsorption isotherm was ascertained by running a number of isotherms at different pH values (3, 7.8, and 9.6) and adsorbent dosages (1.0, 2.0, and 3.0 g/l). A UV/visible spectrophotometer are used to measure the dye concentrations. Using the mass balance relationship equation the amount of dye absorbed onto the activated carbon, q (mg g-1), was calculated. At regular intervals of time, the samples in an aqueous medium were collected to determine their dye concentration. The amount of dye adsorption in a particular period at time t, qt (mg g-1), was determined by an equation given below.

$$qt = (C_o - C_t)xV/M$$

Liquid-phase concentrations of solutes at initial at any time (t) are denoted by C_o (mg L-1) and C_e (mg g-1). The volume of the solution is denoted by V, and the dosage of adsorbent in the solution (g L-1) is denoted by M. Relations between the mass of dye adsorbed at a particular dosage, pH, and liquid phase of dye concentration are determined by Langmuir (1918) and Freundlich (1906) isotherm model (Jayarajan *et al.*, 2011).

Results and discussion

Characterization of Vachellia nilotica activated carbon

FTIR analysis

To find the functional groups present in the *Vachellia nilotica* activated carbon (VNAC) Fourier Transform Infra-Red spectra were recorded shows characteristic peaks at 3384 cm^{-1} are attributable to the O–H stretch (Fig. 1). Peak at 2925 and 2846 cm-1attributed to C-H stretch. Also, the peak at 1574and 1383cm−1corresponds to the C=C stretching of aromatic ring. The peaks at 900-700 ascribed to C-H stretch of aromatic rings. The presence of hydroxyl, carbonyl and aromatic compounds confirms the lignocellulosic biomass and it is also evident that availability of these functional groups is more favorable to adsorb dyes on the surface of activated carbon.

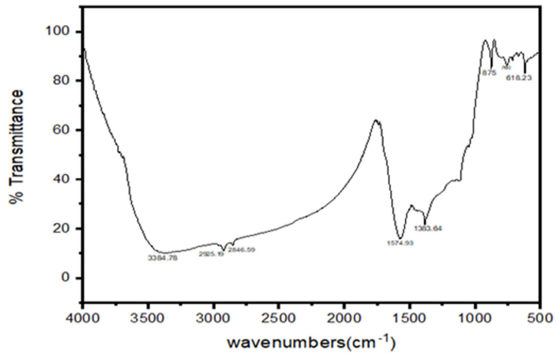

Fig. 1. FTIR spectra of *Vachellia nilotica* activated carbon (VNAC)

The synthesized *Vachellia nilotica* biomass was examined using TGA/DTA analysis to determine the thermal stability of the material. Fig. 2 shows the TGA/DTA curve of *Vachellia nilotica* activated carbon.

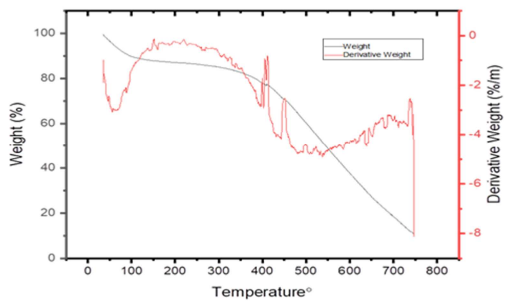

Fig. 2. FTIR spectra of *Vachellia nilotica* activated carbon (VNAC)

A loss in mass was obtained in the preliminary stage and on increasing the temperature the synthesized activated carbon can withstand up to 400°C. After reaching 400°C a sudden increase at 420°C and

450°C can be noticed. The activated carbon starts to degrade after 450°C. TGA shows that the degradation of activated carbon at 450°C is lower when compared with its properties. It is evident from the results that the synthesized activated carbon from *Vachellia niltica* will be a promising material for the active removal of organic pollutants, also it that can resist even at elevated temperatures.

Surface morphology and elemental analysis

Scanning electron microscopy (SEM) technique was used to observe the surface morphology of the activated carbon samples. Fig. 3 shows the SEM image of the activated carbon. It was evident that the surface of the activated carbon contained a lot of big pores. The huge surface area and porous structure of the activated carbon are the result of the well-developed pores. The numerous micropores that are produced on the particle's surface by chemical stimulation favour the carbon's adsorption properties. As the Selective AC pores formed from tapioca peel are possible; these morphological traits are essential (Selvarajan *et al.*, 2021; Abisha *et al.*, 2021).

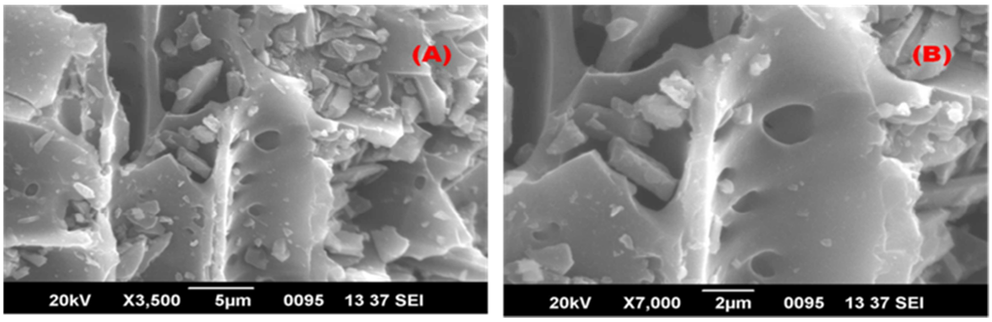

Fig. 3. SEM image of VNAC (A) and (B) various magnification

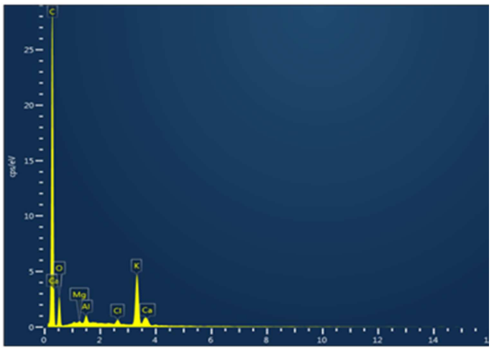

Fig. 4. EDAX spectrum of VNAC

Table 1. Elemental analysis of VNAC

Element	Wt%	Atomic %
C	75.11	83.09
O	16.96	14.09
Mg	0.17	0.09
Al	0.51	0.25
Cl	0.42	0.16
K	6.1	2.07
Ca	0.72	0.24
Total	100	100

EDAX analysis

Elemental composition of the synthesized activated biomass was determined by using Energy dispersive X-ray spectroscopy (EDAX). Fig. 4 displays the energy dispersive X-ray (EDX) spectra of VNAC. EDAX spectra illustrates that the elements such as C, O, Mg, Al, Cl, K and Ca peaks are obtained. This confirms the distribution of elements on synthesized VNAC. Table 1 displays the weight percentages of C (75.11), O (16.96), Mg (0.17), Al (0.51), Cl (0.42), K (6.1) and Ca (0.72). From the obtained results EDX spectra confirms the purity of the synthesized material.

Adsorption studies

Effect of dosage and pH

Adsorption of dye using environmentally benign *Vachellia nilotica* (VNAC) at various dosages and pH are shown in Fig. 5(a) and (b). The concentration of dye causes an abrupt increase in adsorption during the initial stage (Yadav *et al.*, 2021). It eventually arrived at a constant value. Adsorption slightly increases as the concentration rises. pH controls the electrostatic contact between the adsorbent and the adsorbate during the adsorption process. The sorbent's surface is affected by positive charge, which causes the adsorbent's rate to vary. This enhances the rate of adsorption by allowing the negatively charged dye molecule to be accepted. Adsorption increases in response to an increase in adsorbent dosage (Jain *et al.*, 2013). The effect of pH is described in Fig. 5(b). This indicates that the adsorption rate is higher at pH 3 and pH 7.8. Lower pH levels promote this, and as a result, the surface of the prepared activated carbon has more H+ ions. Because of their electrostatic attraction, dye molecules acquire a negative charge on one another (Sushmita Banerjee *et al.*, 2017).

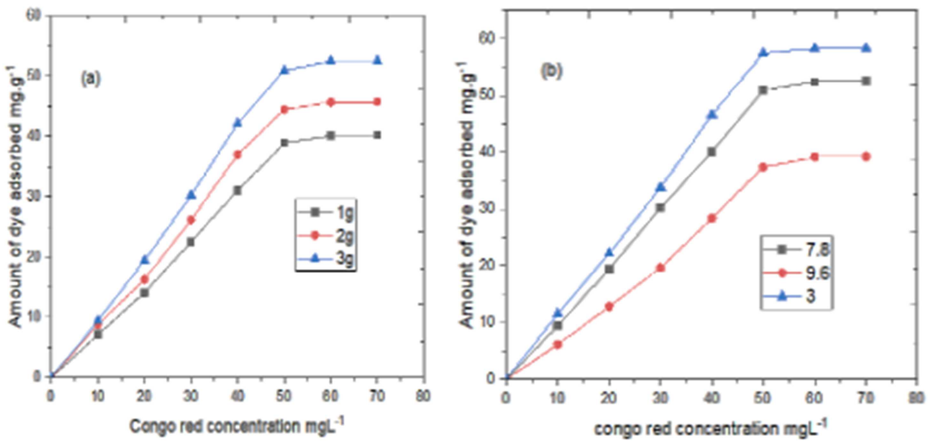

Fig. 5.a-b. Effect of adsorption of Congo red on VNAC (a) at different dosages with dye concentration and (b) at different pH with dye

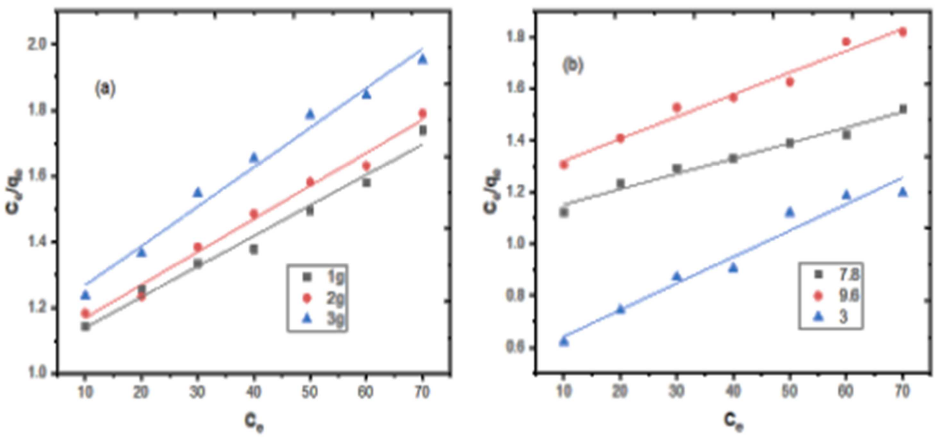

Fig. 6a-b. Langmuir isotherm for the adsorption of Congo red on *Vachellia nilotica* (a) at different adsorbent dosages with dye concentration; and (b) at different pH with dye concentration

Langmuir isotherm

The adsorption isotherm is of major importance in elucidating the interactive behavior between the adsorbate and adsorbent. The saturation monolayer can be represented by the expression.

$$qe = \frac{KbCe}{(1 + bCe)}$$

$$\frac{1}{qe} = \frac{1}{k} + \frac{1}{kbce}$$

As seen in Fig. 6a and b, the adsorption isotherm is also used to determine the monolayer capacity (Qo) of the adsorbent for the dye. The Langmuir isotherm plot provided a good fit for the dye's adsorption at these various concentrations. The statistical plots substantially exhibited a linear relationship (at the 95% confidence level), demonstrating the application of the isotherm and the surface adsorption efficiency, as indicated by the R^2 values, which are close to unity. The adsorption isotherm's shape makes it abundantly evident that it falls into the L2 group of isotherms, indicating that it is either the normal or Langmuir type of adsorption. The Langmuir isotherm constants and correction factors are presented (Egbosiuba *et al.*, 2020; Bergaoui *et al.*, 2018).

Freundlich isotherm

The possibility of multilayer development and the uniform energy of the Freundlich adsorption isotherm represent the adsorption process for heterogeneous surface energy systems. The most practical technique to show the experimental data at different adsorbent dosages and pH levels is to use the sorption isotherm, as shown in Fig. 7a and b. The pictures also show the isothermal batch data matched to the linear shape of the Freundlich isotherm.

$$qe = {k_F cce^1}/{n}$$

$$lnq_e = \frac{lnK_F}{(1/n)lnC_e}$$

It has been stated that the magnitude of the exponent 1/n indicates the favorability and capacity of the adsorbent system (Egbosiuba *et al.*, 2020; Bergaoui *et al.*, 2018). The values n> 1 represent favorable adsorption conditions. In most cases, the exponent between 1< n< 10 shows the beneficial adsorption. It can be seen from Fig. 7 that the correlation coefficients (R^2) were satisfactory and strengthened the fact that the sorption process was very favorable.

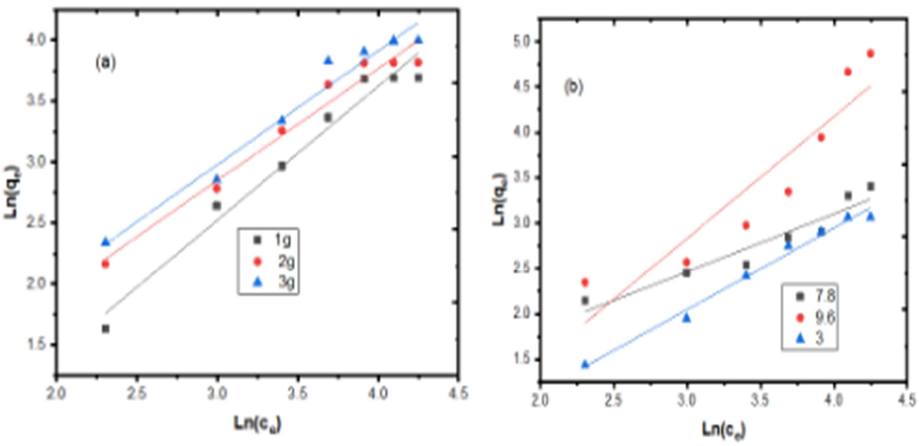

Fig. 7a-b. Freundlich isotherm for the adsorption of Congo red dye using *Vachellia nilotica* (a) at different adsorbent dosages with dye concentration and (b) at different pH with dye concentration.

Conclusion

The adsorption of Congo red dye from an aqueous solution was investigated in batch and equilibrium modes under different reaction conditions using activated carbon derived from *Vachellia nilotica* waste. The Langmuir model's fitness in the current system shows that the adsorbate's monolayer coverage is created at the adsorbent's outer space. An analysis of the isotherm of the Freundlich model demonstrated a rather high capacity for monolayer adsorption. For Congo red on ACVN at 70 mg L^{-1} concentration, the greatest percentage removal of 24.3 g L-1 was achieved with an adsorbent dose of 3.0 g L^{-1}. The increase in adsorption of dye with adsorbent dosage was due to the availability of more surface area of the adsorbent for adsorption. It can be seen that the dye uptakes in acidic solutions are significantly higher than those in neutral and alkaline situations. The pH of the aqueous solution significantly affects how well the dye adheres. The utilization of *Vachellia nilotica* activated carbon, which is produced from agricultural waste, opens up new possibilities for affordable wastewater treatment options.

The information provided here should be useful in designing and constructing a treatment process that is both practically and economically feasible that makes use of batch (or stirred tank) reactors.

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