ISSN 0973-3450 (Print)

JUC Vol. 16(6), 83-93 (2020). Periodicity 2-Monthly

ISSN 2319-8036 (Online)

Estd. 2005

JOURNAL OF ULTRA CHEMISTRY

An International Open Free Access Peer Reviewed Research Journal of Chemical Sciences and Chemical Engineering

website:- www.journalofchemistry.org

Characteristic evaluation of adsorption efficiency of activated wood charcoals in adsorbing acetic acid

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http://dx.doi.org/10.22147/juc/160602

Acceptance Date 02nd November, 2020, Online Publication Date 10th November, 2020

Abstract

Activated wood charcoal (Carbonized) acts as an excellent adsorbent and it finds wide applications. The present study was carried on four different activated wood charcoals Salix (Sac), Pinus (Pac), Anacardium occidentale (AOac) and Calophyllum inophyllum (CIac) found in Karwar district, Karnataka, India. The activated charcoal was prepared by carbonizing in a muffle-furnace at 800 ^{0}C and the yield of carbonized carbon was about 50%. SEM morphology and EDX spectrum indicates the homogeneity and purity of the activated wood charcoals containing 65-86% carbon to that of commercial charcoal (89% C) and hydrogen is absent. Langmuir and Freundlich adsorption isotherms are well-correlated and verified. The regressive coefficient (R^2) of isotherms recorded a higher value above 0.92 which proves the homogeneous and even adsorption phenomenon by the activated wood charcoals. Sac and AOac recorded highest percentage of removal of acetic acid of about 40-60% and 20-26% with Cac (20-25%) where Pac and CIac recorded 17-23% which is relatively near to the commercially activated charcoal(Cac). On comparison of relative percentage of removal of acetic by activated wood charcoals with commercial charcoal, the activated wood charcoals serve as an efficient adsorbent for acetic acid. The cost and availability of the wood is cheaper.

Key words : Wood charcoal, SEM, Isotherms, Acetic acid, Efficiency.

Introduction

Wood charcoal is a less expensive adsorbent for adsorbing liquids and gaseous molecules and ease of method of preparation. Wood charcoal can be converted to activated carbons which becomes versatile adsorbent. Adsorption capacity of the activated carbon of wood charcoal is due to their high surface area, a microporous structure, and a high degree of surface reactivity [1]. The textural property of activated carbon depends on the method of preparation and starting material[2]. Many research studies reported the preparation of activated carbon using nutshells[3,4], Bamboo[5], rice husk[6], plant stem[7] etc. as a raw material. Activated carbon is the commonly used as an adsorbent for the removal of dyes and phenolic compounds[4]. The renewable resources, such as wood and coconut shell are dominant in the preparation of activated carbon[9]. Rajeshwar Man Shrestha[10] suggested different activated carbons prepared by variable parameters with optimal conditions like Lapsi seed stone particles to Phosphoric acid ratio (1:1), temperature of carbonization (400 °C), and time required for carbonization (4 hours). The utilization of cheaper wastes and agricultural by- products like apricot stones[11], guava seeds[12], black stone cherries[13], peach stones[14, 15], orange peel[16], Peanut shell[17], coconut shell and wood[18], rubber seeds[19], molasses[20] are used for the preparation of activated carbon. Commercial activated carbons are commonly produced from naturally occurring carbonaceous materials such as coal, wood, and peat[21]. A. A. Attia *et al.,*[22] proposed that activated carbon produced from olive stones was chemically activated using sulfuric acid and utilized as an adsorbent for the removal of Cr(VI) from aqueous solution in the concentration range 4-50 mg/L. Adsorption results obtained for activated carbon was compared with the acid-treated commercial activated carbon. The favourable efficiency indicates that the Cr(VI) adsorption is obtained at pH 1.5 and equilibrium adsorption data was better fitted to the Langmuir adsorption model. Urbain Kouakou, *et al.,*[23] reported that the commercial activated carbon from a local wood was investigated as a suitable adsorbent for the removal of heavy metal ions such as Zn and Fe from synthetic and industrial wastewater by batch adsorption technique. The initial and final concentrations were determined by absorption atomic spectrometer (AAS). The models of Langmuir and Freundlich were applied to describe adsorption, and Langmuir model is more appropriated to represent the experimental equilibrium.

Several researchers[24-31] have proposed the effectiveness wood type material and seeds, peels, leaves from plant source activated carbon was used in treating industrial wastewater. Ademiluyi *et al.,*[32] investigated the adsorption and treatment of organic contaminants using activated carbon from waste Nigerian bamboo by carbonizing at 400 °C-500 °C and activated with acid at 800 °C forming granular activated carbon (GAC). They investigated the adsorption of organics from the refinery waste on the activated carbon at 28 °C. The experimental data was correlated by Freundlich, and Langmuir adsorption isotherms. The adsorption data fitted well into the Freundlich isotherm with breakthrough time of 1.5 hours for the fixed bed adsorption phenomenon. Ibaraj and Sulochana[33] used activated carbon from Jack fruit peel to treat and remove malachite green from polluted water from a dye industry. Equilibrium data derived follows Freundlich, Langmuir and Redlich Peterson adsorption isotherms. Researchers[34,35] reported in their research the use of FTIR, SEM-EDS, XRD, and BET to characterize the tamarind seed activated carbon prepared. The proximate determinations like, percent yield, iodine number, methylene blue number, and preliminary test of Fe(III) adsorption were also studied for activated carbon.

The present work deals with the carbonization and activation of wood charcoals by physical method. SEM- EDX has been used to study the morphology, elements present and purity of the activated carbon. Langmuir and Freundlich adsorption isotherms were used to explain the efficiency of removal of acetic acid.

Materials and Methods

Chemicals :

Double distilled water, Con H_2SO_4, Commercial activated charcoal, Analytical grade NaOH, Glacial acetic acid

Materials :

Dry wood of Salix (Willow tree) belonging to

Solicaceae family, Pinus (Pine tree) belonging to pinaceae family, Anacardium occidentale (Cashew nut tree) belonging to anacardium family, and Calophyllum inophyllum belonging to calophyllacea family was used to get carbonized carbon.

Preparation of activated carbon:

Slash of fresh dry wood was weighed accurately and soaked in concentrated sulfuric acid for 24 hours. Then, the excess of sulfuric acid was drained off and dried. The dried wood slashes were carbonized in muffle-furnace at 130 – 150 ^{O}C. The carbonized slashes were ground to fine powder and activated in muffle-furnace at 800 ^{O}C. Powdered material was thoroughly washed with doubled distilled water till it reaches the pH of the distilled water and drain. The activated carbon was dried by heating in the oven at 110 ^{O}C for one hour and cooled to room temperature for further study. Percentage of carbonization was determined as,

$$\text{Percentage of cabonization} = \frac{W_c\ 100}{W_b}$$

Wc = Weight of carbonized wood, Wb = weight of wood before carbonization

Scanning electron microscopy (SEM) analysis:

The surface morphology of activated charcoal of wood samples was obtained by SEM. SEM images were recorded with Scanning Electron Microscope (JEOL; JSM-IT500) equipped with an electron probe analyzer system having the following conditions.

Items	Value
Measurement conditions	
Acceleration voltage	20.00 kV
Probe current	0.00 nA
Magnification	x 12000-14000
Process time	T3
Measurement detector	First
Live time	30.00 seconds
Real time	30.25 seconds
Dead time	1.00
Count rate	250 -750.00 CPS

Determination percentage removal of acetic acid:

Exact normal solutions of acetic acid was prepared ranging from 0.5 N to 0.1 N. These solutions were standardized with potassium hydrogen phthalate (PHP) and the normality was fixed. Five different 250 ml reagent bottles containing one gram activated carbon, and each were added 100 ml of acetic acid of normality from 0.5 N to 0.1 N. The bottles were occasionally shaken and allowed for one hour exactly. Then, the solution was filtered by discarding 5 ml of the filtrate in the beginning, and 5 ml of the filtrate was titrated against standard 0.1 N NaOH solution using phenolphthalein as an internal indicator. The concentration of acetic acid was determined before and after adsorption by activated charcoal of the samples and the commercially available standard activated charcoal. The percentage of removal of acetic acid was calculated using the formula below.

$$\text{Percentage of removal} = \frac{(C_0 - C_e)\ 100}{C_e}$$

Ce=Equilibrium concentration, Co=Initial concentration

Adsorption isotherms :

Adsorption isotherms are useful for correlating the efficiency of activated carbon of the wood as adsorbent to the find feasibility in the utilization. Langmuir and Freundlich isotherms were constructed to analyze the experimental equilibrium data. Langmuir assumes mono layer adsorption into a surface consist of a finite number of adsorption sites of uniform strategies of adsorption with immigration of adsorbate within the plane of surface. The Langmuir isotherm is modeled by the equation

$$Ce/Qe = (Ce/Qo) + (1/Qob)$$

The Freundlich isotherm is an empirical equation which is used for heterogeneous system with interaction between the molecules adsorbed. Freundlich isotherm assumes heterogeneous surface energies and Freundlich isotherm is modeled by the equation.

$$\text{Log}\,(Qe) = \text{Log}\,Kf + 1/n\ \text{Log}(Ce)$$

Where Qe = Amount adsorbed per unit weight of

Table 1. Percentage of carbonized charcoal

Name of the wood charcoal (activated)	Wb(g)	Wc(g)	$\% \text{ of Carbonization} = \frac{Wc \times 100}{Wb}$
Salix (Sac)	20.05	9.41	46.9
Pinus (Pac)	20.21	9.67	47.8
Anacardium occidentale (AOac)	20.13	10.20	50.7
Calophyllum inophyllum (CIac)	20.08	9.82	48.9

Cac – Commercial activated charcoal

adsorbent, mg/g at equilibrium, Ce= Equilibrium concentration of adsorbate in solution, mg/l, Qo = Mono layer capacity of adsorbate, mg/g, b = Langmuir constant, Kf, n = Freundlich constants.

Results and Discussion

Percentage of carbonization :

The charcoal of the woods was prepared using impregnated sulfuric acid method. The charcoal was thoroughly washed with double distilled water to remove impregnated sulfuric acid and dried in an oven at 110 ^{O}C. The percentage of yield was determined.

Table 1 report the percentage of carbonized charcoal against the dry weight of wood. Anacardium occidentale shown a maximum of 50.7 % and salix wood gives a minimum of 46.9 % of carbonized charcoal. Pinus and calophyllum inophyllum recorded 47.8 and 48.9 % of carbonization respectively.

SEM images of carbonized charcoal of the woods:

Fig. 1 show the SEM images of surface activated carbonized charcoal of Salix, Pinus, Anacardium occidentale, Calophyllum inophyllum, and Commercial charcoal. The SEM images were recorded for 1μm size with a magnification of 12000 to 14000 times. Carbonization of wood samples were well-comparable with commercially available charcoal. The morphology of experimentally determined wood charcoal samples was significantly distinguished in the adsorption capacity for acetic acid as adsorbate.

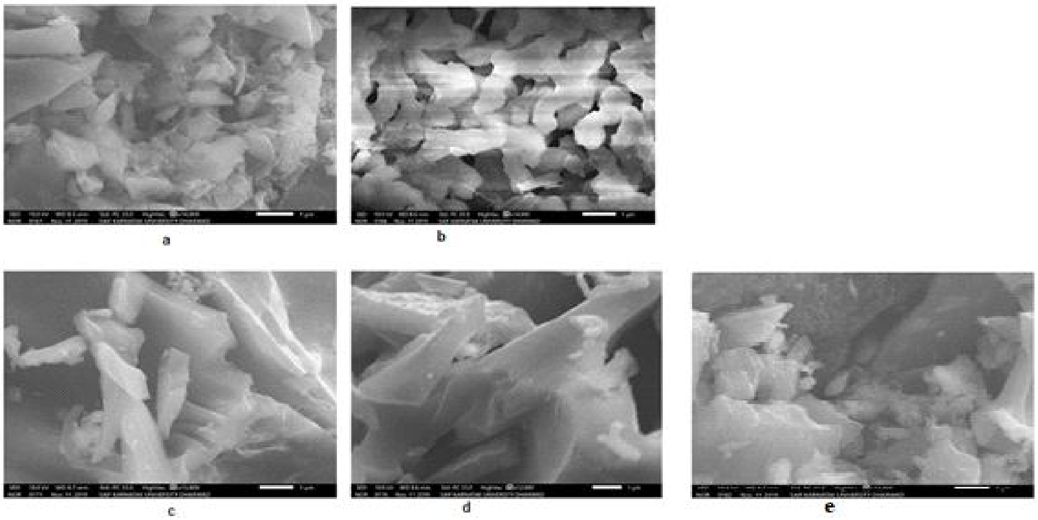
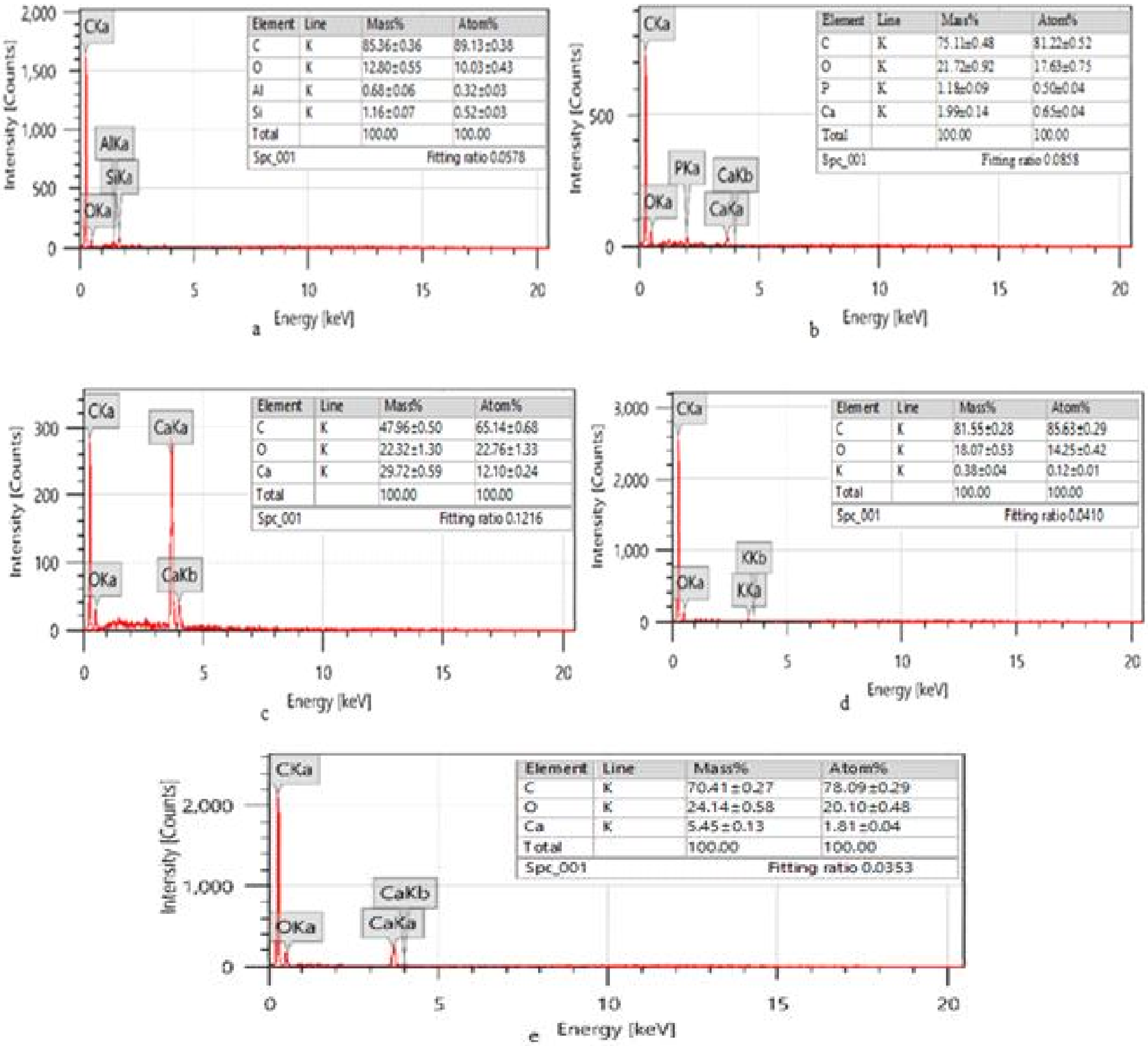


Figure 1: SEM images of the carbonized charcoal of a) Sac b) Pac c) AOac d) CIac e) Cac

*Energy dispersive X-ray spectrum (EDX)***:**

Fig. 2 represents Energy Dispersive X-ray spectra of the wood carbonized charcoals. The elementary analysis of the carbonized charcoal reports the percentage of carbon and the elements present in comparison to commercial activated charcoal as standard.



Element	Line	Mass%	Atom%
C	K	85.36±0.36	89.13±0.38
O	K	12.80±0.55	10.03±0.43
Al	K	0.68±0.06	0.32±0.03
Si	K	1.16±0.07	0.52±0.03
Total		100.00	100.00
Spc_001			Fitting ratio 0.0578

Element	Line	Mass%	Atom%
C	K	75.11±0.48	81.22±0.52
O	K	21.72±0.92	17.63±0.75
P	K	1.18±0.09	0.50±0.04
Ca	K	1.99±0.14	0.65±0.04
Total		100.00	100.00
Spc_001			Fitting ratio 0.0858

Element	Line	Mass%	Atom%
C	K	47.96±0.50	65.14±0.68
O	K	22.32±1.30	22.76±1.33
Ca	K	29.72±0.59	12.10±0.24
Total		100.00	100.00
Spc_001			Fitting ratio 0.1216

Element	Line	Mass%	Atom%
C	K	81.55±0.28	85.63±0.29
O	K	18.07±0.53	14.25±0.42
K	K	0.38±0.04	0.12±0.01
Total		100.00	100.00
Spc_001			Fitting ratio 0.0410

Element	Line	Mass%	Atom%
C	K	70.41±0.27	78.09±0.29
O	K	24.14±0.58	20.10±0.48
Ca	K	5.45±0.13	1.81±0.04
Total		100.00	100.00
Spc_001			Fitting ratio 0.0353

Figure 2. Energy dispersive X-ray spectrum of a) Cac b) Sac c) Pac d) AOac e) CIac

The EDX spectrum gives 89 % carbon atom, 10 % oxygen atom, 0.3 % Aluminium and 0.5 % silicon in commercial activated charcoal, 81% of carbon atom, 17.6 % oxygen atom, 0.5 % phosphorus and 0.7 % calcium in Salix activated charcoal, 65 % carbon atom, 22 % oxygen atom, 12 % calcium in pinus activated charcoal, 86 % carbon atom, 14 % oxygen atom, 0.2 % potassium in Anacardium occidentale activated charcoal and 78 % carbon atom, 20 % oxygen atom, 1.8 % calcium in Calophyllum inophyllum activated charcoal. The Salix and Anacardium occidentale activated carbon shown a relatively high percent of carbonization (81 % and 86 %) to that of pinus and Calophyllum inophyllum (65 % and 78 %). Carbonization Salix and Anacardium occidentale was nearly close to that of commercially activated charcoal (89 %) as compared to pinus and Calophyllum inophyllum. A relative percentage of oxygen was more with wood charcoals as compared to commercially activated charcoal. The trace percentage of other atoms in wood charcoals were phosphorus, calcium, and potassium to that of commercially activated charcoal having aluminium and silicon. The EDX spectra conspicuously indicates the absence of hydrogen atom and this could confirm the completion of charring of the wood. This indicates the percentage of purity of wood charcoals for the utilization as an effective adsorbent for acetic acid.

Verification of Langmuir's adsorption :

To verify Langmuir's adsorption isotherm, a graph was plotted 1/ Qe versus 1/ Ce. The plot gives a straight line whose slope is equal to 1/ Qo b and the intercept of the straight line is equal to 1/ Qo. The plot for the samples were as shown in the Fig. 2.

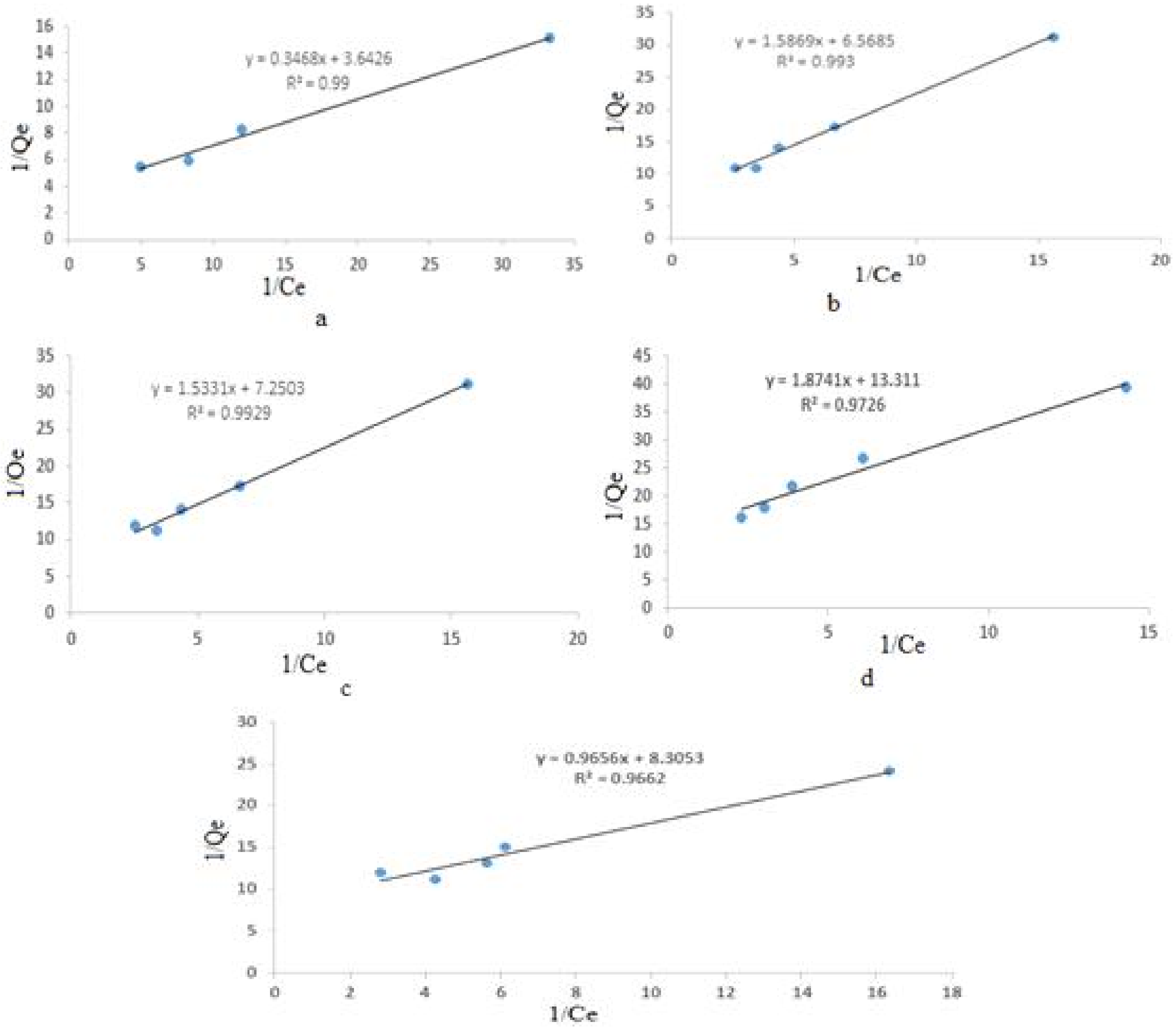


Figure 3. Langmuir adsorption isotherm of a) Sac b) Pac c) AOac d) CIac e) Cac

Verification of Freundlich's adsorption :

To verify Freundlich's adsorption isotherm, a graph was plotted logQe versus logCe. The plot gives a straight line whose slope is equal to 1/n and the intercept of the straight line is equal to logkf. The plot for the samples were as shown in the Fig. 3.

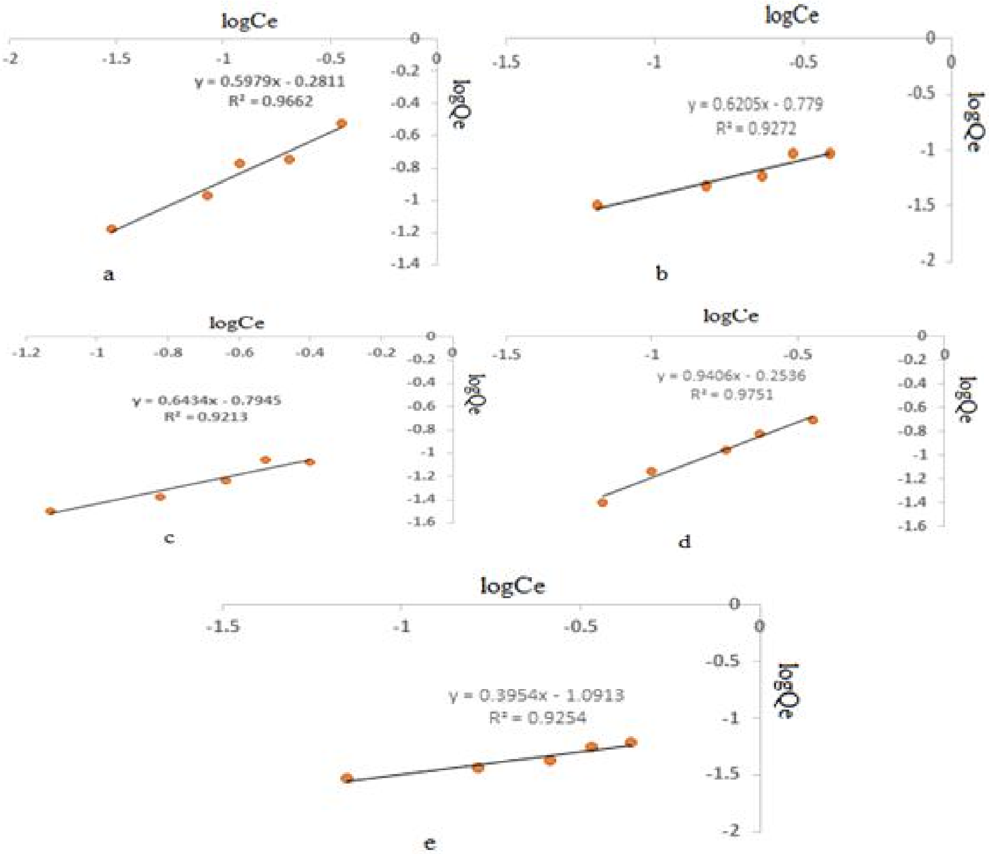


Figure 4. Freundlich adsorption isotherm of a) Sac b) Pac c) AOac d) CIac e) Cac

Table 2, gives the record of adsorption isotherm parameters of Langmuir and Freunlich adsorption. These data explain the correlation of the adsorption of samples with the commercially available activated charcoal.

Table 2. Adsorption isotherm parameters

Adsorbent	Adsorbate	Langmuir adsorption parameters			Freundlich adsorption parameters		
		Qo	b	R^2	Kf	1/ n	R^2
Sac	Acetic acid	0.28	10.5	0.9901	0.52	0.60	0.9662
Pac	Acetic acid	0.15	3.5	0.9930	0.12	0.62	0.9272
AOac	Acetic acid	0.14	4.7	0.9929	0.16	0.64	0.9213
CIac	Acetic acid	0.08	7.1	0.9726	0.56	0.94	0.9721
Cac	Acetic acid	0.12	8.3	0.9662	0.12	0.40	0.9254

The determination of regressive coefficient (R^2) of Langmuir isotherms for Sac, Pac, AOac, CIac and Cac are 0.9901, 0.9930, 0.9929, 0.9725 and 0.9662 respectively. The regressive coefficient (R^2) of Freundlich isotherms for Sac, Pac, AOac, CIac and Cac are 0.9662, 0.9272, 0.9213, 0.9711 and 0.9254 respectively. It is conspicuous that the correlation of regressive coefficients for the Langmuir isotherms are significantly higher than that of Freundlich isotherms, which clearly indicates that the accumulation of adsorbate occur on a homogeneous surface by mono layer adsorption and could be explained regarding chemisorption through the formation of an ionic, or covalent bonds between adsorbent and adsorbate[36]. The Langmuir isotherm is often evaluated by a separation factor, R_L, named as a dimensionless separation factor, and it is calculated to determine whether the adsorption system is favourable or not[37]. The following equation defines R_L.

$$R_L = \frac{1}{1 + C_o b}$$

Where, R_L is a dimensionless separation factor, Co is the initial carboxylic acid concentration and **b** is the Langmuir constant. The value of separation factor shows the type of isotherm and nature of the adsorption process. Feasibility of the reactions is explained using the value of R_L ($R_L > 1$: unfavourable; RL = 1: linear; $0 < R_L < 1$: favourable; $R_L = 0$: irreversible). Values of R_L were found in the range of 0.14-0.43 for Sac, 0.36-0.76 for Pac, 0.15-0.46 for AOac, 0.17-0.50 for CIac and 0.15-0.49 for Cac respectively. These values clearly indicate the favourable adsorption of all wood charcoals.

Percentage of removal of acetic acid :

Table 3, indicates the percentage of removal of acetic acid by different activated wood charcoal in relation with the commercially available activated charcoal.

Table 3. Percentage removal of acetic acid by wood charcoal with commercial charcoal

Adsorbate	Cac	Sac	Pac	AOac	CIac
% of removal of acetic acid	20-25	40-60	20-23	20-26	17-20

Sac and COac show relatively higher percentage of removal of acetic acid of 40-60% and 20-26% with the commercially activated charcoal Cac having 20-25%. The samples Pac and CIac shown slightly lower percentage of 20-23% and 17-20% with Cac. It is observed a marginal difference 2-5%. Thus, all the wood charcoals under the experiment contributes very efficient adsorption for acetic acid.

Conclusion

The woods in the experiments were cheaply available in Karwar district Karnataka, India. The woods were carbonized about 50% per dry weight ratio. The EDX spectra confirmed the elements present the activated wood charcoal samples. Wood samples Sac and COac contained 80-85% to that of Cac having 89 % carbon whereas Pac and CIac contains 65- 78% carbon. The elements present were Al, Si, Ca, and oxygen. Absence of hydrogen shows the purity of wood charcoal. Acetic acid was adsorbed on the wood charcoal homogeneously, and follows Langmuir and Freunlich adsorptions. The regressive coefficients R^2 recorded were more than 0.92 showing availability of uniform and even surface of adsorbent for the adsorption of acetic acid. The adsorption isotherm of Langmuir records higher regressive coefficient R^2 as compared with Freunlich isotherms. This confirms the mono layer adsorption. Sac and COac shown relatively higher percentage of removal of acetic acid of 40-60% and 20-26% with respect to the commercially activated charcoal Cac having 20-25%. The samples Pac and CIac shown slightly lower percentage of 20-23% and 17-20% with Cac. Thus, this experiment proves that the activated carbon of the wood Salix (Willow tree), Pinus (Pine tree), Anacardium occidentale (Cashew nut tree) and Calophyllum inophyllum are an alternative for commercially available charcoal.

Scope of future work :

The application of charcoal as an absorbent for environment pollutants finds lot of scope in future research. It is used in the water treatment by determining BOD and COD. Charcoal can also be used to heavy metal ions factories effluents.

Acknowledgement

I would like to acknowledge the support of our beloved Principal and group members for their invaluable cooperation to work on this topic

Abbreviations

BHT	Butylated Hydroxy Toluene
SL	Soya Lecithin
SEM	*Scanning Electron Microscopy*
EDX	Energy Dispersive X-Ray Analyzer

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