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Adsorption of Phenol by a Biomass (Acacia Nilotica Branches) Based Activated Carbon for Water Purification.

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ABSTRACT

Phenol is a very toxic substance found in the effluents of various chemical and coke-oven industries. It requires to be treated with an economical adsorbent. Acacia nilotica is a native plant of India and of this region of Sangrur particularly. It has been found that this type of trees can be grown easily here which can, not only help in rehabilitation of dry land but can also enable us to provide the raw material for adsorption of phenol. Powdered branches of acacia nilotica (PBAN) were investigated as an adsorbent for phenol. The study presented here, consists of comparing effects of carbon dosage, pH, contact time, initial phenol concentration, and rpm of shaker on adsorption of phenol on PBAN and on activated PBAN in batch experimental studies. As adsorption is much more prominent in case of activated PBAN due to its much improved characteristics, adsorption studies were focused on it. Adsorption isotherm models of Langmuir, Freundlich, Temkin and Dubinin-Radushkevich were found to be fitting to experimental data for phenol adsorption for various concentration ranges between 0 and 832 µg/l. Kinetic models were also applied on kinetic experiments of adsorption of phenol on activated PBAN. It was found that pseudo-first-order explains the process appropriately.

Keywords: Acacia nilotica, Activated carbon, Adsorption, Biomass, Phenol.

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INTRODUCTION

Phenol is one of the common constituents from effluents of coke-oven industries, plastic manufacturing, oil purification, pharmaceutical and timber industries. Due to its good solubility in water it is one of the most hazardous constituent in industrial effluents. Presence of phenol in effluents is toxic to aquatic life also. Various methods are available in literature which is used for removal of phenol from waste water. The methods include steam stripping, solvent extraction, oxidation (O_3, H_2O_2 and ClO_2), ion exchange, biodegradation and adsorption methods [1-3]. Among the methods mentioned, adsorption methods are most popular in use all over the world [4-6]. However, generally adsorbents cost high. For last three decades much research has been reported towards investigating cheaper materials such as fly ash, peat, soil, rice-husk, saw dust, baggase, rice-straw, tendu leaf etc for their use as adsorbents [7-12].

In quest for new and low cost biomass as adsorbent for phenol, acacia nilotica branches have been considered for study. It is widely spread in subtropical and tropical Africa from Egypt to Mauritiana southwards to South Africa, and in Asia eastwards to Pakistan and India, in China, in the Northern Territory and Queensland in Australia, in Caribbean, Indian Ocean islands, Mauritius, United States, Central America, South America and the Galápagos islands [13-17]. This tree happens also to be native plant of Sangrur area of Punjab. Much have been reported in literature regarding its uses of bark, seed and stem for medicinal, fuel and furniture purposes [18]. Though acacia nilotica is slow growing specie but is moderately long lived tree. Its dried thin branches can be shredded again and again as a renewal raw material for its use as adsorbent.

Here adsorption studies have been done on powdered branches of acaia nilotica (PBAN) and its activated powder (powdered PBAN). After comparing the effects of carbon dosage, pH, contact time, initial phenol concentration, and rpm of shaker on adsorption, it is found that activated PBAN is much better adsorbent. Its reasonably higher value of fixed carbon content, phenol number, iodine number and BET surface area justifies this claim. Equilibrium and kinetic studies on activated PBAN are done and experimental data are fitted to various mathematical models. The present extensive study on activated PBAN is to evaluate it as new and appropriate adsorbent.

MATERIALS AND METHODS

Preparation of Powdered Branches of Acacia Nilotica (PBAN)

Dried branches of washed acacia nilotica were taken after removing leaves and thorns, cut into small pieces, put in the grinding machine. The powder was passed through 250 micron screen and stored for studying the physical characteristics and adsorption studies.

Preparation of Activated Powdered Branches of Acacia Nilotica (Activated PBAN)

The powder of acacia nilotica branches, preparation of which is explained in earlier section was taken to activate it. The powder was soaked in H_3PO_4 for 4 hrs with agitation at temperature of 35°C. The resultant slurry was filtered and washed thoroughly till pH of 7 was obtained. The sample was oven dried and kept for charring at 500°C in the muffle furnace for 3hrs. The sample thus obtained was passed through (mesh) screen 250 micron and stored for further studies.

Characterization of PBAN & Activated PBAN

Activated carbon obtained as above was subjected to various ways of characterization by adopting the standard procedures [19-22]. The moisture content of the samples was determined by heating the known weight of samples in an oven at 110 $^{\circ}$C for more than one hour till constancy of weight was obtained. For determining ash content the residues of both the samples were burned in the muffle furnace at 750^{o}C for more than two hours in silica crucibles till constancy of weight was obtained. The adsorption characteristics were studied in terms of phenol number (the amount of powdered carbon required for 90% removal of phenol) and iodine number of adsorbents. The surface area of the activated carbon was carried by BET (Brunauer Emmett Teller) nitrogen adsorption method. The properties of adsorbents under study are shown in Table 1.

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Table 1: Properties of Activated PBAN and PBAN

Property	Activated PBAN	PBAN
Bulk Density (kg/m^3)	208	267
Moisture Content (%)	6.3	12
Ash Content (%)	1.6	2.73
Fixed Carbon (%)	71.5	23.80
Volatile Matter (%)	27	73.46
Solubility in Water (%)	0.0060	0.0151
pH of slurry	6.3 (1.75%)	6.3 (0.25%)
Phenol Number (g)	1.4	-
Iodine Number (mg.g^{-1})	680	493
Surface Area (m^2.g^{-1})	298	54

Batch Experiment

For adsorption study, 100 ml of phenol solutions (1g/l) were taken with various dosages of PBAN and activated PBAN at 25^{o}C. The adsorption study was done for effect of dosage, pH, contact time, initial phenol concentration and rpm of the shaker on both adsorbents. Determination of phenol was done using scott's method [22]**.** For study of effect of dosage, both the adsorbents were equilibrated with phenol solutions for 24 hours at 250 rpm. To study effect of pH, solutions of phenol were adjusted to the entire range of acid to base with the help of 0.1N NaOH or 0.1N H_2SO_4 and solution was equilibrated with fixed amount of each of adsorbents at the same rpm. To study effect of time on percentage phenol removal, optimum pH of 6.3 with fixed amounts of adsorbents were kept in the solution for different period of time till constancy of adsorption was observed. Similarly the study of initial phenol concentrations and effect of rpms were studied on adsorption. The adsorption data of activated PBAN were fitted in various mathematical models like Langmuir, Freundlich, Temkin and Dubinin Radushkevich isotherms. Study of effect of time on adsorption gave kinetic study. Here data were fitted in kinetic models of pseudo-first order and pseudo-second order.

RESULTS AND DISCUSSIONS

Effects of Various Parameters on Adsorption

Effect of Carbon Dosage

Fig.1 shows the amount of phenol removed as function of adsorbent dosage from the aqueous solution at pH of 6.3. Adsorbent dosages were varied between 100 mg to 2500 mg and the solution was left to be equilibrated for 24 hours. As shown in the figure to remove the entire amount of phenol (with initial phenol concentration of 1g / litre), the minimum amount of adsorbent dosage required was determined to be 1.75g for activated PBAN whereas adsorbent dosage of 0.25g removes the phenol at 33 % and values remain constant at this value till dosage value of 1.75 g and further till 2.5 g. Thus activated PBAN is far better adsorbent than PBAN.

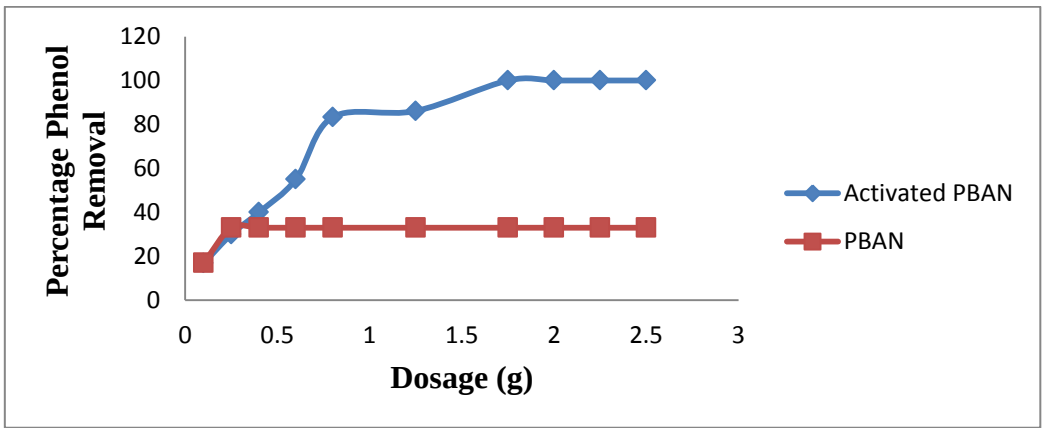

Figure 1: Comparison of effect of adsorbent dosage on percentage phenol removal by adsorbents

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Effect of pH

As shown in Fig. 2, PBAN and activated PBAN were studied at various pH values of the phenol solution at 25^{o}C for 24 hours. The amount of phenol removal distinctly increases as pH of solution increases, becomes maximum at pH of 6.3 and then decreases as pH value increases towards alkalinity for activated PBAN. But for PBAN, the change is not that distinct. Removal of phenol is maximum with value of 33 % at pH of 6.3. Fig. 2 shows that pH affects removal of phenol greatly for activated PBAN.

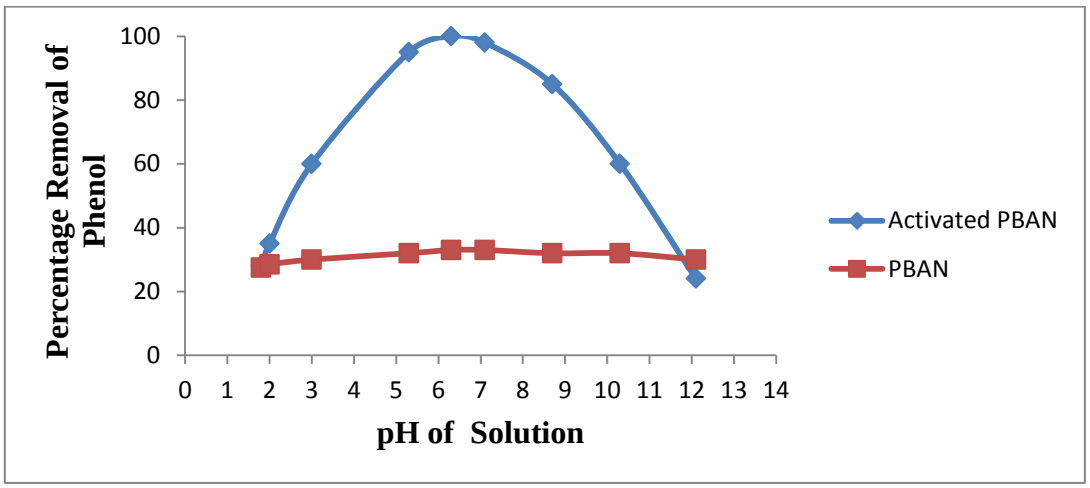

Figure 2: Comparison of effect of pH on percentage phenol removal by adsorbents

Effect of Contact Time

Fig. 3 shows the effect of contact time on the removal of phenol by activated PBAN and PBAN. As discussed in *Batch Experiment* in *Materials and Methods* section, percentage phenol removal was studied for different duration of time (ranging from 0.5 to 24 hours). The study reveals that 6 hour and 1hour as equilibrium time for adsorption of phenol for activated PBAN and PBAN respectively.

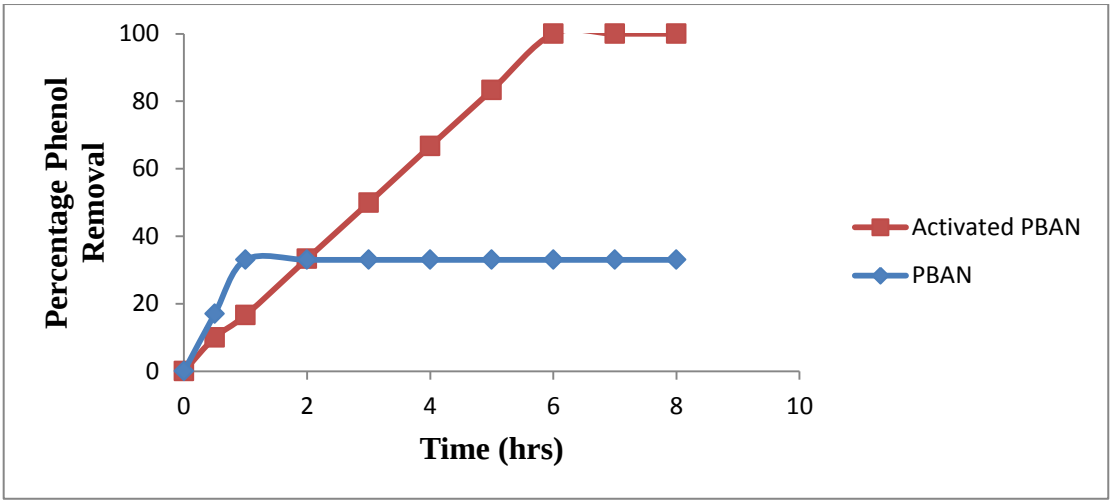

Figure 3: Comparison of effect of contact time on percentage phenol removal by adsorbents

Effect of Initial Phenol Concentration

Fig. 4 shows effect of initial concentration of phenol solution, on percentage removal of phenol by activated PBAN as well as PBAN. As mentioned in *Batch Experiment* in *Materials and Methods* section percentage phenol removal was studied for both the adsorbents for initial phenol concentration range of 96mg/l to1000 mg/l. For activated PBAN contact time was kept for 6hrs (equilibrium time), pH of solution kept

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at 6.3. It was found that percentage removal of phenol was constant at the value of 100%. For PBAN contact time was kept for 1hr (equilibrium time), pH of solution at 6.3.The percentage removal of phenol remained at 16.7% for concentration range 96mg/l to 769 mg/l. Percentage phenol removal increases to 33% from that of 16.7 % for concentration range 769mg/l to 1000mg/l.

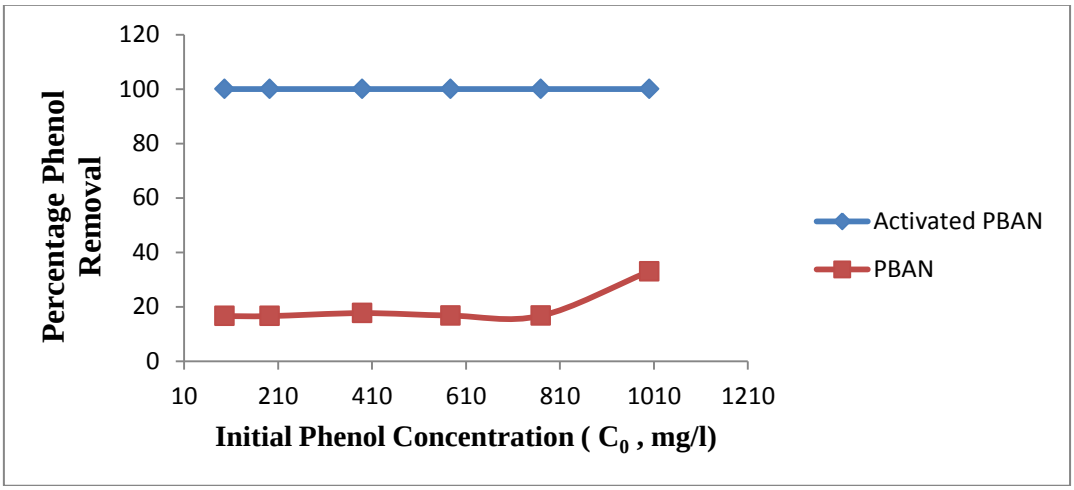

Figure 4: Comparison of effect of initial phenol concentration on percentage phenol removal by adsorbents

Effect of agitation speed

Effect of agitation speed was studied on percentage removal of phenol for both the adsorbents as shown in Fig.5. Here contact time was kept at 6hrs, pH at 6.3 and temperature at 25°C for activated PBAN. Contact time for PBAN was kept at 1hr, pH at 6.3 and temperature at 25°C .It was found that agitation speed had no effect for adsorption of phenol on PBAN. Phenol removal remained at 33% in the supernatant solution. When the agitation speed was studied between 50 to 250 rpm for activated PBAN, percentage phenol removal increased to 100 % at 200 rpm from that of 33 % at 50 rpm. Percentage removal of phenol remained constant at 100 % till 250 rpm.

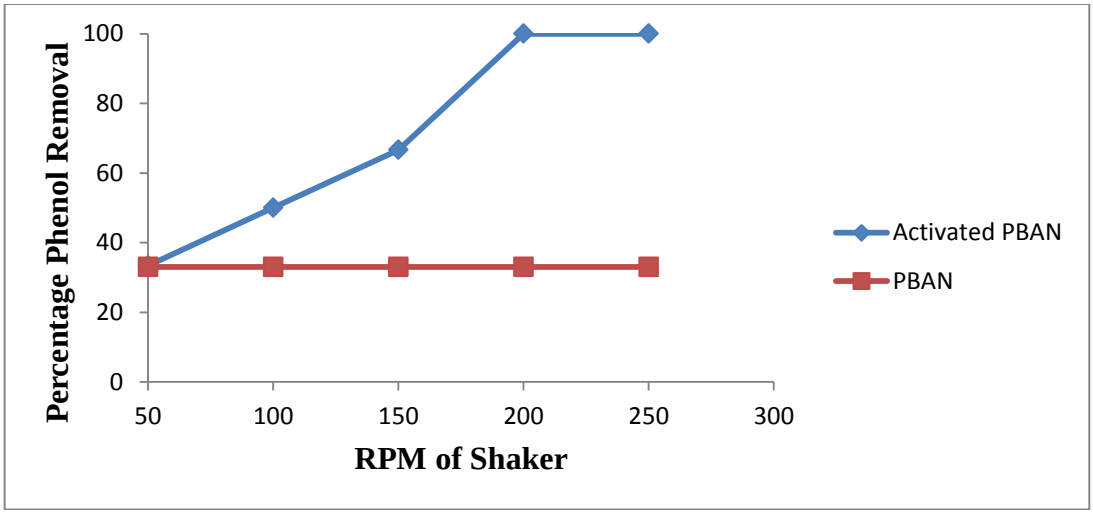

Figure 5: Comparison of effect of agitation on percentage phenol removal by adsorbents

Adsorption Isotherm

By comparing effects of various parameters namely adsorbent dosage, pH, contact time, initial phenol concentration, agitation speed on percentage phenol removal, it was found that activated PBAN is much better adsorbent than PBAN. It was focussed to study adsorption isotherm of activated PBAN . Adsorption isotherm data (as shown in Fig.6) were analysed by fitting into Langmuir, Freundlich, Temkin and

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Dubinin Radushkevich mathematical models [23-24]. Adsorption kinetics were also analysed by fitting into pseudo-first and pseudo-second order kinetic models.

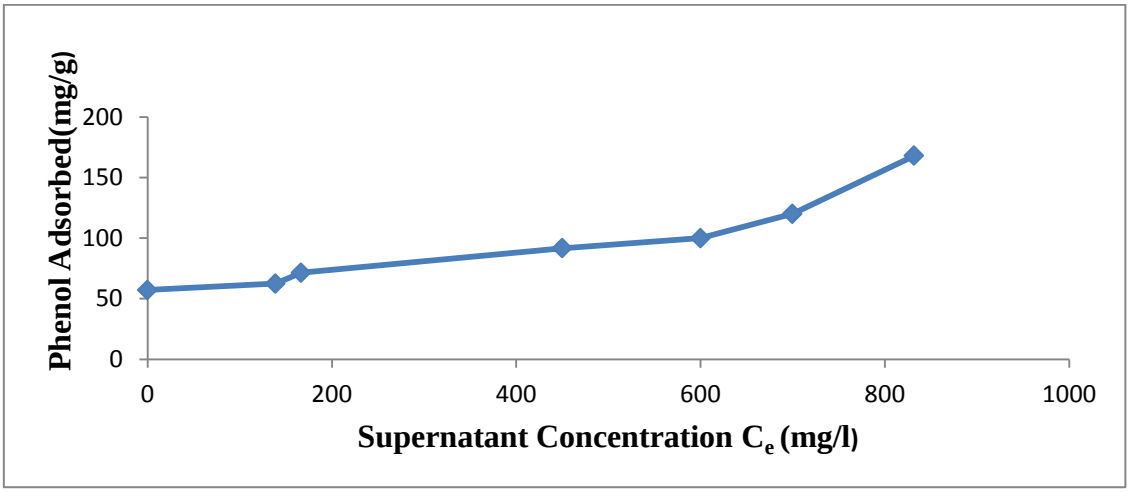

Figure 6: Adsorption Isotherm of Activated Carbon

Langmuir Isotherm

The mathematical description of adsorption isotherm by Langmuir is given as:

$$1/q_e = \left(\frac{1}{Q_0 b C_e}\right) + \left(\frac{1}{Q_0}\right) \quad \text{(Eq.1)}$$

Where, q_e represents the amount of adsorbate adsorbed per unit mass of adsorbent, C_e represents equilibrium concentration (µg/l) of supernatant solution, Q_0 monolayer adsorption capacity (mg/g) of adsorbent, and b surface energy (g/l) corresponding to the process of adsorption. From adsorption isotherm data of activated PBAN, a plot of $1/q_e$ versus $1/C_e$ is shown in Fig. 7. Here '1/ (b Q_0)' and '$1/Q_0$'respectively shows slope and intercept of normal equation of straight line of the form of 'y = mx+c'

The Langmuir adsorption isotherm can be expressed in terms of separation factor R_L, which is given by the equation [25]

$$R_L = 1/ (1+b\ C_i) \quad \text{(Eq.2)}$$

Where, C_i is the initial concentration of phenol in µg/l and b is the Langmuir constant in g/l as indicated in (Eq.1). The separation factor R_L indicates the nature of the adsorption process as given in Table2 [9].

Fig.7 indicates that adsorption data fitted quite well in Langmuir isotherm model with R^2>0.99. As per values of intercept and slope, value of R_L lies between 0 and 1, confirming that the adsorption process to be favourable. Also value of R_L is very near to 0 showing that the process is almost irreversible. Here b value comes out to be 4g/l which represents low surface energy, indicating stronger bonds between phenol and adsorbent. [26]. Fairly low to moderate b values have been reported in many of sorbent-phenol systems [9,10,23]. Value of Q_0, monolayer adsorption capacity is calculated to be 250 mg/g which seems significantly high for phenol-activated PBAN system.

Table 2: The Process Nature of Separation Factor

RL Value	Type of Process
$R_L = 0$	Irreversible
$0<R_L<1$	Favourable
$R_L=1$	Linear
$R_L>1$	Unfavourable

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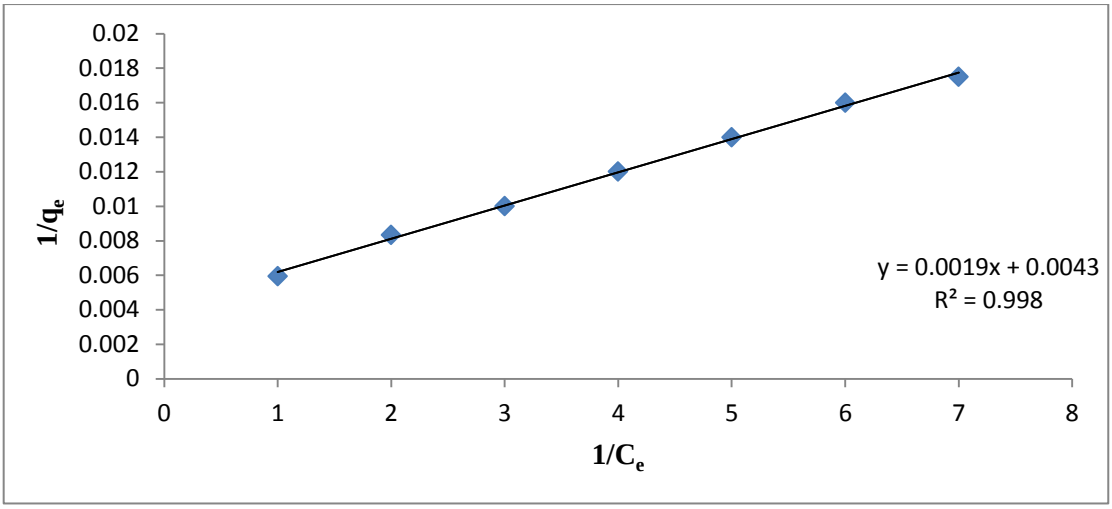

Figure 7: Langmuir Adsorption Isotherm

Freuendlich Isotherm

The mathematical equation for adsorption isotherm given by Freundlich is given as:

$\frac{x}{m} = q_e = kC_e^{1/n}$ (Eq.3)

Where, k [(mg/g). (l/g)$^{1/n}$] and n are the measures of adsorption capacity and intensity of adsorption. q_e is the amount of phenol adsorbed per unit mass of adsorbent and C_e is the equilibrium concentration in µg/l. The logarithmic form of Freundlich Equation can be expressed by

$\ln q_e = \ln k + 1/n \ln C_e$ (Eq.4)

Plot of ln q_e vs ln Ce is shown in Fig.8.The results indicated that adsorption of phenol on activated carbon obeys Freundlich isotherm from concentration range of 166.67 µg/l to 600 µg/l as indicated by very high R^2 values (>0.99).Here values of k and n can be calculated from slope and intercept of Fig. 8.Value of k=23.48[(mg/g). (l/g)$^{1/n}$] indicating very greater affinity for phenol towards adsorbent in the concentration range of 166.67µg/l to 600µg/l. Also value of n comes out to be 4.46 indicating it is satisfying the condition of heterogeneity ie, 1<n<10 as well as 0<1/n<1 [27]

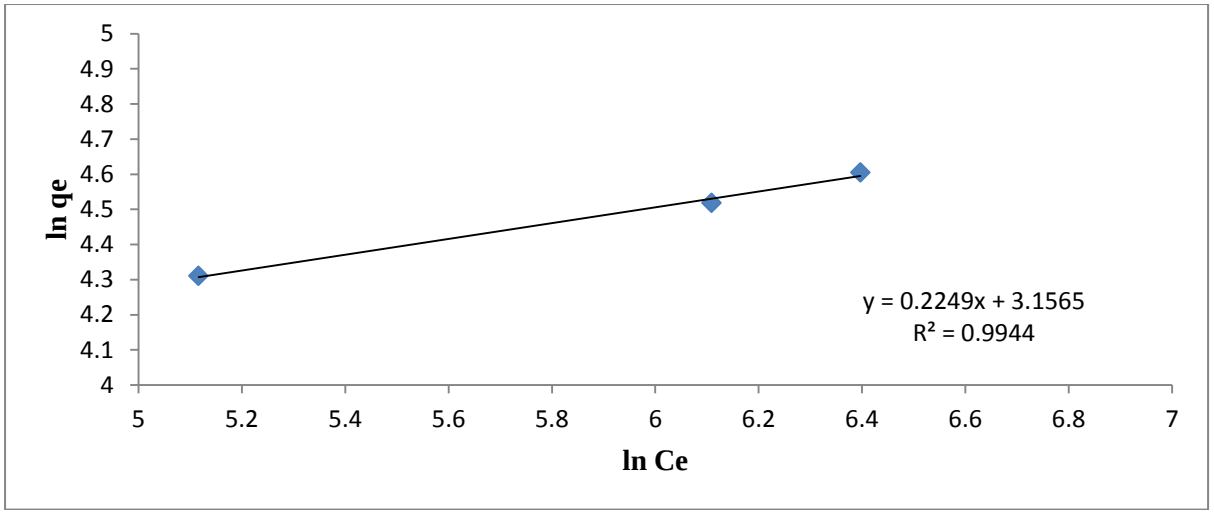

Figure 8: Freundlich Adsorption Isotherm

Temkin Isotherm

Mathematical equation for adsorption isotherm given by Temkin is:

$q_e = B \ln A + B \ln C_e$ (Eq.5)

Where A (l/g) and B (dimensionless) = RT/b_1 are Temkin constants which can be found from slope and intercept of the plot q_e versus ln C_e. Here b_1(Jmol^{-1}) is constant related to heat of sorption.

From Fig. 9 value of B = 21.80; A=0.16 l/g; b_1=113.65J mol^{-1}; R^2= 0.994

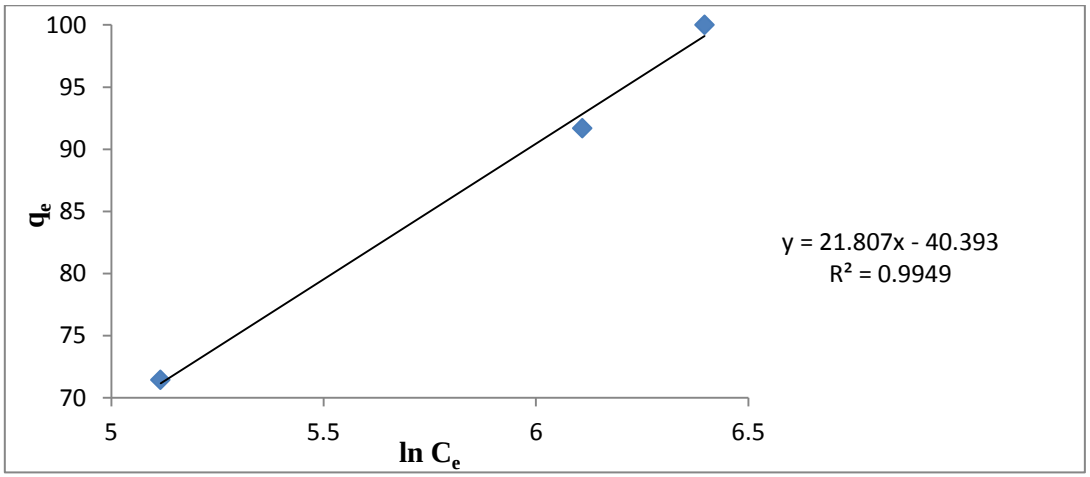

Figure 9: Temkin Adsorption Isotherm

The values of R^2, A and b_1 showed that model favoured the adsorption of phenol on activated PBAN in the concentration range of 166.7 µg/l to 600 µg/l of phenol.

Dubinin Radushkevich Isotherm

To estimate the characteristic porosity of the biomass and apparent energy of adsorption, the Dubinin Radushkevich model [28] was used.

Mathematical equation for adsorption isotherm given by Dubinin Radushkevich is:

$\ln q_e = \ln q_m - \beta \varepsilon^2$ (Eq.7)

Where β (mmol^{2}J^{-2}) is D-R constant; ε (Jmmol-1) is Polanyi potential and $\varepsilon = RT \ln(1+1/C_e)$

Where R (8.314 J mol^{-1} K^{-1}) is universal gas constant, T (K) is temperature.

Here β is related with free energy of adsorption per mole of adsorbate as it migrates to the surface of biomass from infinite distance in the solution. Fig.10 shows plot of ln q_e versus ε^2 for phenol concentration range of 600 µg/l to 832 µg/l. The graph gives values of q_m and β from slope and intercept values.

Value of β = 0.063 mmol^{2}J$^{-2.}$ Porosity parameter value β for activated PBAN towards phenol was less than unity indicating sorption of phenol was significant. Value of q_m = 282 mg/g supports the experimental value. R^2 value of 0.94 indicated that this model fitted experimental adsorption data under study.

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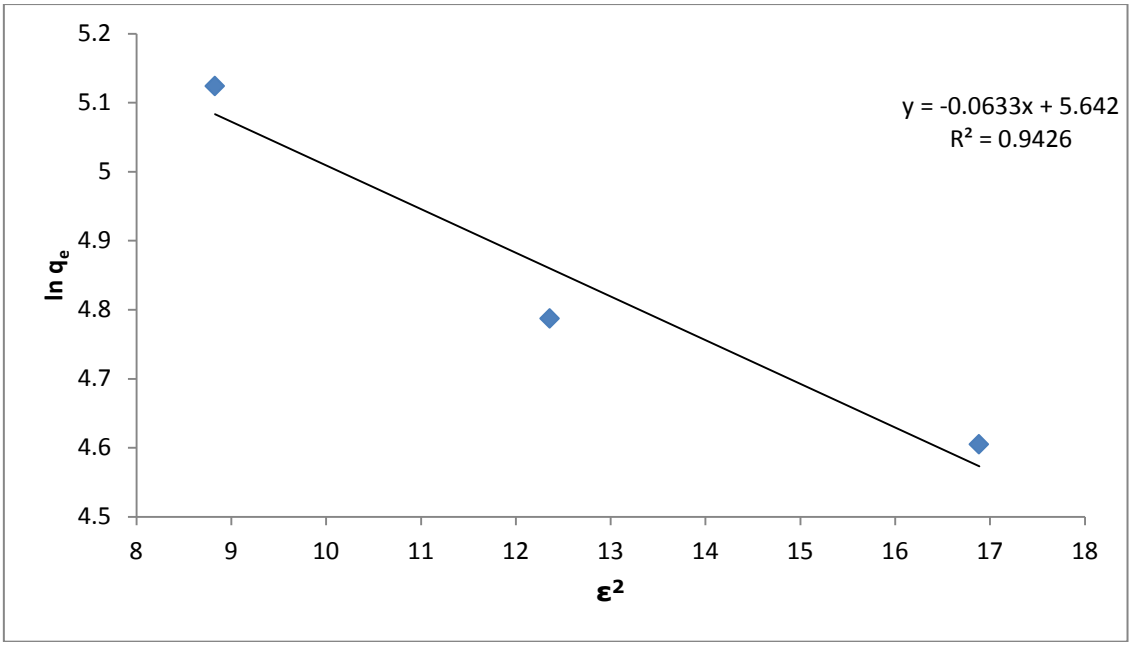

Figure 10: Dubinin Radushkevich adsorption isotherm

Adsorption Kinetics

For measuring adsorption efficiency kinetics of adsorption is required to be studied. This study gives solute uptake rate. The dosage of 1.75 g of activated PBAN was taken in phenol solution (1g/l) of 100 ml. The adsorbent was separately exposed to phenol solution for different times till equilibrium was achieved. The amount of phenol adsorbed was estimated between 60 minutes to 300 minutes.

Pseudo-First-Order Kinetics: Lagergren model was used to study the pseudo first order kinetics [29-31]:

$dq_t/dt = k_1(q_e - q_t)$ (Eq.8)

Where q_e (mg/g) and q_t(mg/g) refers to the amounts of phenol adsorbed on the activated PBAN at equilibrium time and time, t (min) respectively. Here k_1 (min^{-1}) refers to rate constant. Integrating Eq.8:

$\log (q_e - q_t) = \log q_e - k_1\, t/2.303$ (Eq.9)

Thus the rate constant (k_1) can be obtained from the slope of plots of log (q_e-q_t) vs t. Value of R^2>0.9 confirming the fitting of data in Lagergren model. Values of constants as per calculations (shown in Table 4) are q_e = 79.80mg/g and k_1= 0.0046min^{-1}.

Pseudo-Second-Order Kinetics: Adsorption data were also studied for second order kinetics [29].The mathematical model is as given below:

$dq_t/dt = k_2(q_e - q_t)^2$ (Eq.10)

Where k_2 (g/mg.min) refers to the rate constant. After integration of Eq.10:

$t/q_t = 1/(k_2 q_e^{\,2}) + t/q_e$ (Eq.11)

Eq.11 can be expressed in linear form as below:

$t/q_t = 1/h + t/q_e$ (Eq.12)

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The plot of t/q_t vs t (Fig.12) gives linear relationship with correlation coefficient R^2=0.711. Hence the data did not fit Pseudo-Second-Order Kinetics quite well.

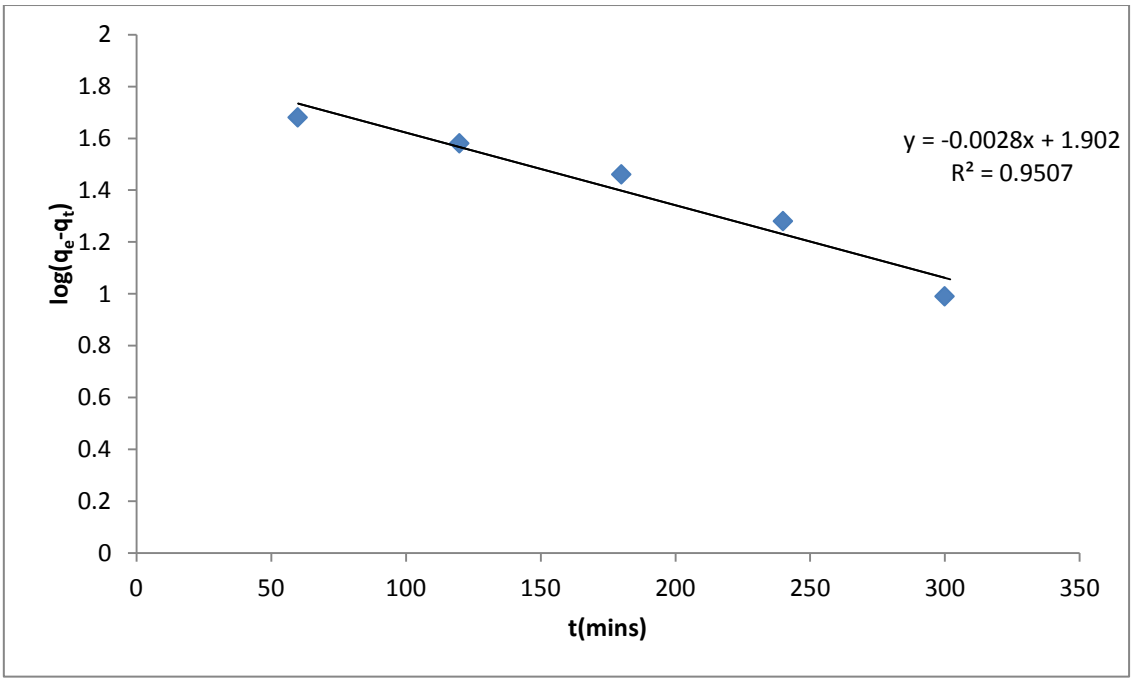

Figure 11: Pseudo-First-Order Kinetics for activated PBAN

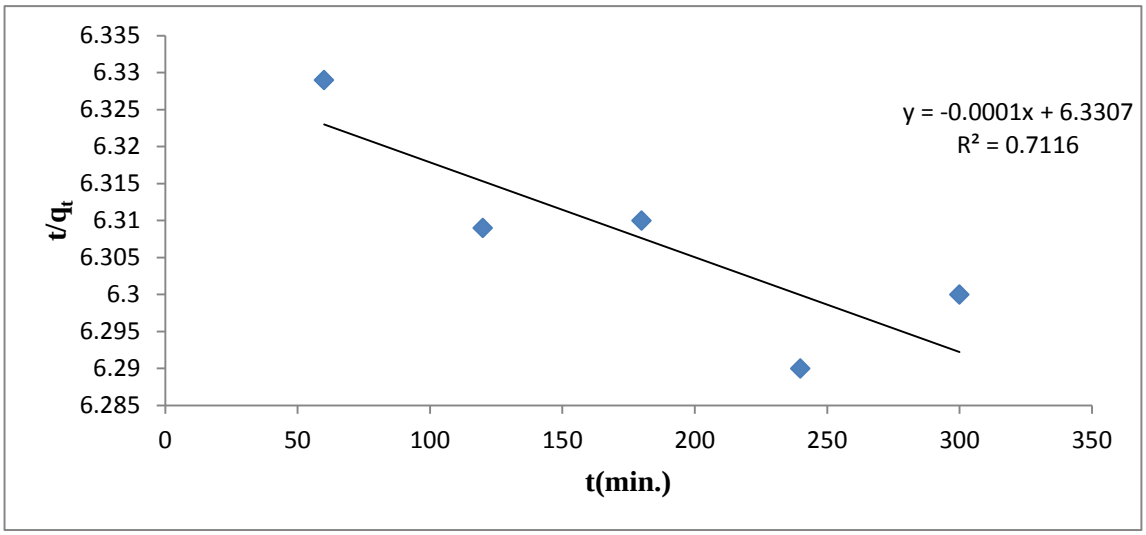

Figure 12: Pseudo-second-Order Kinetics for activated PBAN

Table 3: Various isotherm constants and correlation coefficients

Langmuir isotherm Constants			Freundlich isotherm constants			Temkin isotherm constants			Dubinin Radushkevich isotherm constants		
Q_0	b	R^2	k	n	R^2	A	b_1	R^2	β	q_m	R^2
250	4	0.998	23.48	4.46	0.994	0.16	113.65	0.994	0.063	282	0.942

Table 4: Kinetic rate constants for phenol removal by activated PBAN

Pseudo-First-Order model			Pseudo-Second-Order model		
q_e	k_1	R^2	k_2	h	R^2
79.80	0.0046	0.950	-	-	0.711

CONCLUSION

The present studies indicate activated PBAN is a quite good adsorbent for adsorption of phenol compared to PBAN (without activation). Langmuir isotherm fitted quite well the experimental data for entire concentration range of phenol under study. This confirmed that activated PBAN had monolayer adsorption of phenol. Value of R_L was between 0 and 1 indicating favourable adsorption of phenol towards activated PBAN. Adsorption isotherm data fitted quite well to various concentration ranges under study for Freundlich, Temkin and Dubinin-Radushkevich. All adsorption isotherms confirmed that phenol had very good affinity towards activated PBAN. Kinetics of adsorption was best explained by pseudo-first-order-kinetics. Hence by activating the powder of acacia nilotica which is renewable in nature and freely available, it proves to be new and quite appropriate adsorbent for removal of toxic substance, phenol from effluents and water bodies.

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