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Preparation and characterization of activated carbons from *Sterculia alata* nutshell by chemical activation with zinc chloride to remove phenol from wastewater

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Abstract Nutshells of *Sterculia alata*, a forest waste, were used to prepare activated carbons by zinc chloride activation under four different activation atmospheres, to develop carbons with substantial capability, and to adsorb phenol from wastewater. Experiments were carried out at different chemical ratios (activating agent/precursor). Effect of carbonization temperature and time are the important variables, which had significant effect on the pore structure of carbon. Developed activated carbon was characterized by SEM analysis. Pore volume and surface area were estimated by Hg porosimetry and BET surface area analyses. The carbons showed surface area and micropore volumes of around 712 m^2/g and 0.542 cm^3/g, respectively. The activated carbon developed shows substantial capability to adsorb phenol from wastewater. The kinetic data were fitted to the models of intraparticle diffusion, pseudo-second order, and Lagergren model and followed more closely the pseudo-second-order chemisorption model. The isotherm equilibrium data were well-fitted by the Langmuir and Freundlich models. The maximum uptake of phenol was found at pH 3.5.

Keywords Wastewater treatment · Activated carbon · *Sterculia alata* nutshell · Surface area · Chemical activation · Phenol removal

1 Introduction

Liquid-phase adsorption is a very potent method for the abatement of trace pollutants present in dissolved state in industrial and domestic wastewater. The adsorption process has not been used extensively in wastewater treatment, but demands for a better quality of treated wastewater effluent, including toxicity reduction, have led to an intensive examination and use of the process of adsorption. Adsorption is usually thought of as a polishing process for wastewater that has already received normal biological treatment to remove a portion of the remaining organic as well as inorganic matter. The relative advantages of adsorption over other conventional advanced treatment methods are: (1) it can remove both organic as well as inorganic constituents even at very low concentrations, (2) it is relatively easy and safe to operate, (3) both batch and continuous equipment can be used, (4) no sludge formation, and (5) the adsorbent can be regenerated and used again. Moreover the process is economical because it requires low capital

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cost and there are abundant low-cost materials available which can be used as adsorbents.

Activated carbon has been the water industry's standard adsorbent for the reclamation of municipal and industrial wastewater for potable use for almost three decades (Fornwalt and Hutchins, 1996). Activated carbons have the advantage of exhibiting a high adsorption capacity for organic pollutants due to their high specific surface area, adequate pore size distribution, and relatively high mechanical strength (Perrich, 1981; Zumriye et al., 2001). Despite its prolific use in the water and waste industries, activated carbon remains an expensive material. In view of the high cost and tedious procedures for the preparation and regeneration of activated carbons, there is a continuing search for low-cost potential adsorbents.

In practice, coal and waste biomasses of lignocellulosic materials are two main sources for the production of commercial activated carbons (Goud et al., 2005; Mohanty et al., 2005). Waste biomass, which includes forest as well as agricultural byproducts have emerged as a better choice. Though these raw wastes can be used as adsorbents without further treatment, activation could enhance their adsorption capacity. The production of activated carbons from such wastes converts unwanted, surplus waste, of which billions of kilograms are produced annually, to useful valuable adsorbents. There are a quite large number of studies regarding the preparation of activated carbons from agricultural wastes (Kadirvelu et al. 2003; Nag et al., 1999), nuts (Mohanty et al., 2005), nutshells (Ahmadpour and Do, 1997; Goud et al., 2005), fruit stones (Lussier et al., 1994), bagasse (Mohan et al., 2002), coirpith (Kadirvelu et al., 2001), oil palm waste (Lua and Guo, 1998) and agricultural residues from sugarcane (Blanco Castro et al., 2000), rice (Shrinivasan et al., 1998) and peanut (Periasamy and Namasivayam, 1996), sawdust (Marquez-ontesinos et al., 2001) and canes from some easy-growing wood species (Basso et al., 2002; Maci'as et al., 2003).

Chemical activation of the waste biomass is an effective process to prepare activated carbons with good surface area and pore volume. Different activating agents like potassium hydroxide, phosphoric acid (Maci'as et al., 2003) and zinc chloride have been used by many researchers. However, it has been found that chemical activation with $ZnCl_2$ produces better surface area and pore volume than other activating agents (Ahmadpour and DO, 1997; Mohanty et al., 2005).

Phenol is a very common water pollutant. The major sources containing phenols are the wastewaters from processing manufacturing industries engaged in oil refining, coal tar processing, petrochemical production, coke oven byproducts, plastic industry, textile processing, leather processing, insecticides production, manufacture of dyes and dyeing, glass production, etc. (Patterson, 1997; Zumriye et al., 2001; Banat et al., 2004). The discharge of phenolic waste into waterways, even in low concentration may adversely affect human health as well as that of flora and fauna. It is therefore necessary to remove phenol completely from wastewater, before being discharged in to waterways.

In this work we report the results obtained on the preparation of activated carbons from *Sterculia alata* nutshell with zinc chloride activation and their ability to remove phenol from wastewater. Different preparation variables on the characteristics of activated products were studied to find the optimum conditions for making activated carbons with a reasonable surface area and well-developed porosity. The activated carbons thus produced were used for the removal of phenol from dilute aqueous solutions. Kinetic models were used to identify the possible mechanisms of such adsorption process. The Langmuir and Freundlich models were used to analyze the adsorption equilibrium.

2 Experimental technique

2.1 Preparation of activated carbon

Sterculia alata nutshells collected from nearby locality was first washed with distilled water to remove the water-soluble impurities and surface adhered particles and then dried at 60°C to get rid of the moisture and other volatile impurities. Then the precursor was grounded in the ball mill and sieved to particle size range of 150–200 μm. The proximate analysis of the precursor yielded, moisture 6.4%, volatile matter 31%, fixed carbon 60% and ash 2.6%.

Chemical activation of the powdered precursor was done with $ZnCl_2$. 10 g of dried precursor was well mixed with 100 ml solution that contained 10 g of $ZnCl_2$. The chemical ratio (activating agent /precursor) was 100% in this case. The mixing was performed at 50°C for 1 h. After mixing, the slurry was subjected to vacuum drying at 100°C for 24 h.

The resulting chemical loaded samples were placed in a stainless steel tubular reactor and heated (5°C min^{-1}) to the final carbonization temperature under a nitrogen flow rate of 150 $\text{cm}^3\text{min}^{-1}$ STP. Samples were held at the final temperature (carbonization temperature) for different carbonization times of 1, 2, 3 h before cooling down under nitrogen. Nitrogen entering in the reactor was first preheated to 250–300°C in a pre-heater. The products were washed sequentially with 0.5 N HCl, hot water and finally cold distilled water to remove residual organic and mineral matters, then dried at 110°C. In all experiments, heating rate and nitrogen flow were kept constant. The experiments were carried out for different chemical ratio (100%–500%) and carbonization temperature (300–600°C).

Weight loss of the carbon samples was calculated on a chemical free basis and chemical recovery (CR) was estimated according to:

$$CR = \frac{W_{Pi} - W_{Pf}}{W_C} \times 100 \tag{1}$$

where W_{Pi} and W_{Pf} are the weight of product before and after washing and W_c is weight of chemical used. Chemical recovery and weight loss are regarded as indicators of the process efficiency in the chemical activation process.

Characterizations of the activated carbons were determined by nitrogen adsorption at –196°C using a Micromeritics Flowsorb-2300 and Quantachrome Autoscan Mercury Porosimeter. The BET surface area was calculated from N_2 adsorption isotherms by using the Brunauer-Emmett-Teller (BET) equation (Gregg et al., 1982). The cross sectional area for the nitrogen molecule was assumed to be 0.162 nm^2. The Dubinin-Radushkevich (DR) equation was used to calculate the micropore volume from the N_2 adsorption data. The micropore surface area were then determined from the values obtained from the micropore volumes (Iley et al., 1973). The amount of N_2 adsorbed at a pressure near unity corresponds to the total amount adsorbed at both micropores and mesopores and consequently, the substraction of the micropore volume (from the DR equation) from the total amount (determined at p/p_o = 0.98 in this case) will provide the volume of the mesopore. The average pore diameter can be determined according to the surface area and the total pore volume (the sum of the micropore andthe mesopore volumes), if the pores are assumed to be parallel and cylindrical. Activated carbon was characterized by determining the methylene blue number, phenol number, iodine number etc. by adopting the standard procedures (APHA, 1989; ISI, 1989; Vogel, 1969). The various physico-chemical characteristics of the activated carbon prepared from *Sterculia alata* nutshell at the optimum conditions is given in Table 1.

Table 1 Physico-chemical characteristics of *Sterculia alata* nutshell activated carbon produced at the optimum conditions

S. No.	Control tests	Sterculia alata nutshell
1	Carbon yield (%)	46
2	Ash content (%)	7.6
3	Moisture content (%)	9.62
4	Bulk density (g/ml)	0.84
5	BET Surface area (m^2/g)	712
6	Total pore volume (cm^3/g)	0.542
7	Iodine number (mg/g)	1122
8	Methylene blue number (mg/g)	388
9	Phenol number (mg)	16.56
10	Matter soluble in water (%)	1.6
11	Matter soluble in acid (%)	2.1

2.2 Phenol adsorption procedure

Adsorption kinetics and equilibrium studies were conducted using the bottle-point isotherm technique by placing a known quantity of the adsorbent in glass bottles containing 100 ml of an aqueous solution of phenol with a predetermined concentration (Mohanty et al., 2005). The final adsorbent concentration thereby achieved was 5 g/l, unless otherwise stated, as from our initial experiments we found that this is the optimum adsorbent dose. After such solution preparation, each bottled was shaken vigorously in a temperature controlled incubator shaker for specific time intervals till equilibrium is reached. At the end of the adsorption process, the adsorbent particles were filtered out using 0.45 micron membranes and the phenol concentration in the supernatant was then analyzed.

The concentrations of phenol were determined following the method (Gregg et al., 1982) based on the spectophotometric analysis of the color developed as a result of the reaction of phenol with 4-aminoantipyrine. The phenol concentration retained in the adsorbent phase was calculated according to:

$$q_e = \frac{(C_o - C_e)\,V}{W} \tag{2}$$

where C_o and C_e are the initial and equilibrium concentrations (mg/l), respectively, of phenol in solution; V is the volume (L); and W is the weight (g) of the adsorbent. Two replicates per sample were done and the average results are presented.

Adsorption equilibrium isotherms on the selected carbon were determined using sample dosages of 0.4 g/200 ml aqueous solutions of initial concentrations in the range of 10–100 mg/l, at natural solution pH 6.5. For these experiments, the bottles were shaken, keeping constant the temperature (25°C) and agitation speed (120 rpm) for the minimum contact time (300 min) required to attain equilibrium, as determined from the kinetic measurements detailed above.

The effect of pH on the equilibrium adsorption of phenol on prepared activated carbon was further investigated over a pH range of 2–8. The pH values were adjusted with dilute sulphuric acid and sodium hydroxide solutions.

3 Results and discussions

The results for the characterization of the prepared activated carbon are discussed in this paper. The tests for characterization include weight loss, chemical recovery, BET surface area, micropore volume and adsorption capacities of the activated carbon towards phenol removal.

3.1 Effect of different variables on the preparation of activated carbons

3.1.1 Carbonization time

The effects of carbonization time on weight loss, chemical recovery, BET surface area and micropore volume of $ZnCl_2$ activated carbons are shown in Fig. 1. For all these samples the carbonization temperature is 500°C and the chemical to nutshell ratio is 100% (mass basis). As seen from the figure, carbonization time does not have much effect on the weight loss, whereas BET surface area and pore volume first increases with carbonization time and reaches its maximum at 1 h and thereafter it decreases. The decrease (after 1 h) was possibly due to some of the pores being sealed off as a result of sintering for a prolonged time. Generally, a longer carbonization time is needed to enhance porosity as well as to clear blocked pore entrances before detrimental effects set in at prolonged times. From an initial high surface area and pore volume, it deteriorated with increasing carbonization time. It can be seen from Fig. 1 that the chemical recovery values decrease with the carbonization time. This might be due to the evaporation of $ZnCl_2$ from the precursor at longer carbonization time. Therefore, it can be concluded that in chemical activation with $ZnCl_2$, the impregnation method with a shorter carbonization (1 h in this case) produces activated carbons with a well developed pore structure.

3.1.2 Carbonization temperature

The effects of carbonization temperature on weight loss, chemical recovery, BET surface area and micropore volume of $ZnCl_2$ activated carbons are shown in Fig. 2 for a carbonization time of 60 min and a chemical ratio of 100% (mass basis). Our preliminary studies revealed that this time and chemical ratio were optimum. As can be seen from the figure, that the carbonization temperature does not have much effect on the weight loss. Overall weight loss was found to increase with increasing temperature, resulting in decreased yield with the rise in temperature. This weight loss was essentially due the volatilization of the *Sterculia alata* fibres upon heating and as expected, the quantity of volatiles evolved increased with increasing temperature. The final yields of the char resulting from carbonization at different temperatures were ranging between 43% and 48% of the original weight of the pre-dried extracted fibre. The chemical recovery values decreases with the carbonization temperature which may be due to the evaporation of $ZnCl_2$ from the precursor at higher carbonization temperature.

The effect of carbonization temperature on BET surface area and micropore volume is shown in Fig. 2. When the carbonization temperature was 300°C, pyrolysis reactions had just commenced, thereby creating very small BET surface area as well as pore volume. This phenomenon was due to the inadequate heat energy produced at this low carbonization temperature and the release of volatiles was insignificant for the pore development. Asthe temperature was further increased

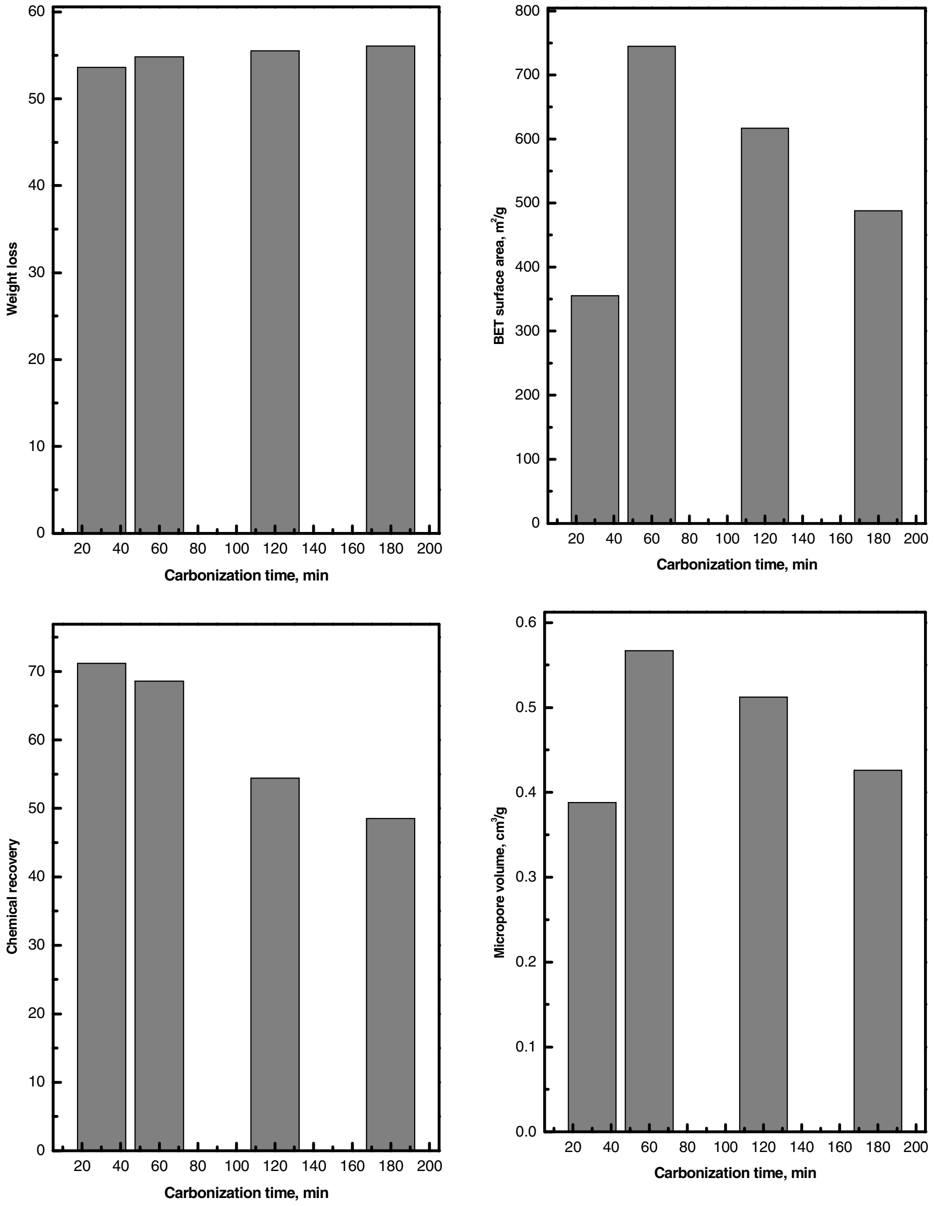


Fig. 1 Effect of carbonization time on weight loss, chemical recovery, BET surface area and pore volume of activated carbons obtained by $ZnCl_2$

to 400°C and subsequently to 500°C, more volatile matters were progressively released during carbonization, thereby resulting in the development of some new pores, and hence the BET surface area increased progressively. The decrease in surface area with further increase in temperature to 600°C might be due to the sintering effect at high temperature, followed by shrinkage of the char, and realignment of the carbon structure which resulted in reduced pore areas as well as volume.

3.1.3 Chemical ratio

The effects of chemical ratio ($ZnCl_2$ to nutshell ratio) on weight loss, chemical recovery, BET surface area and micropore volume of activated carbons are shown in Fig. 3 for a carbonization temperature of 500°C and carbonization time of 60 min. Our previous experiments revealed that this temperature and time were optimum. It can be seen in the figure that the addition of chemical

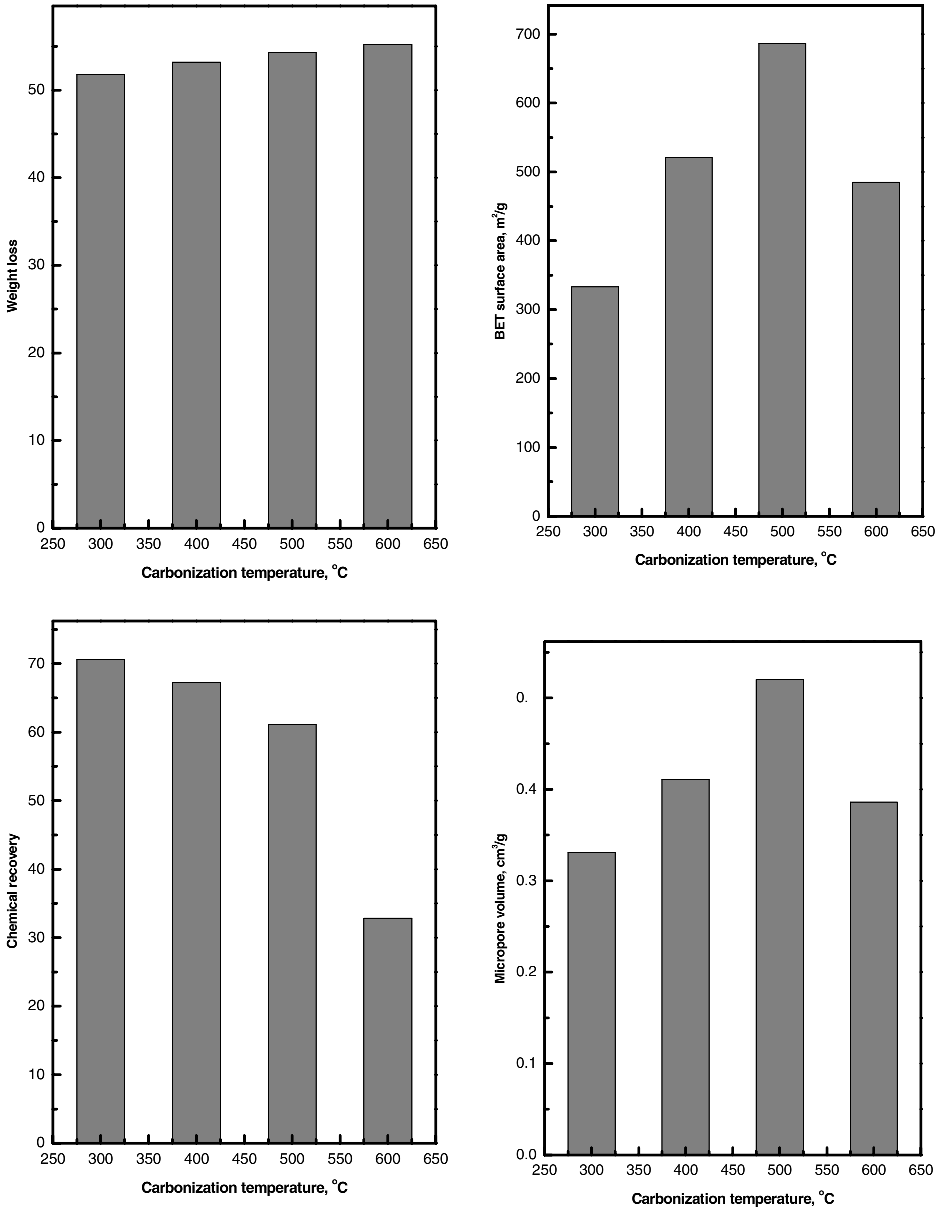


Fig. 2 Effect of carbonization temperature on weight loss, chemical recovery, BET surface area and pore volume of activated carbons obtained by $ZnCl_2$

agents to the precursor decreases the weight loss of the carbon products. The reduction of weight loss is most likely due to the effect of the chemical agent in which it promotes the Scholl condensation (polymerization) reactions. These reactions, which occur among the aromatic hydrocarbons and tar-forming compounds, result in the formation of large molecules (viz. polycyclic aromatics) in the structure of the activated carbon and increase the carbon yield (Ahmadpour et al., 1997). The effect of chemical ratio on chemical recovery is shown in Fig. 3. A portion of chemicals added to the carbon precursor can be recovered during the washing stage of the activated products after carbonization. It is obvious that with increasing the chemical ratio, the recovery should be increased as well.

The effects of chemical ratio on BET surface area and micropore volume is shown in Fig. 3. It shows that surface area as well as the pore volume increases

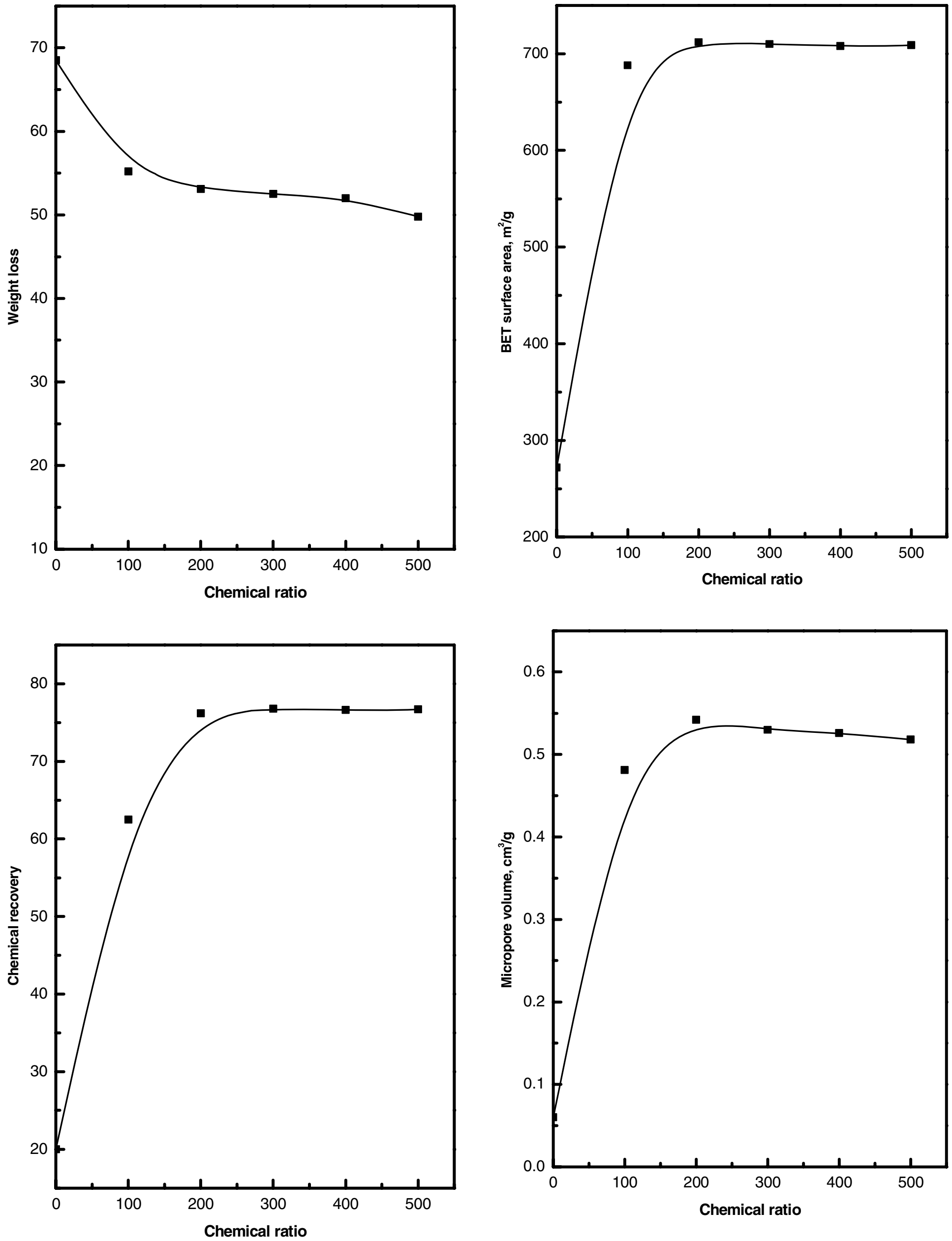
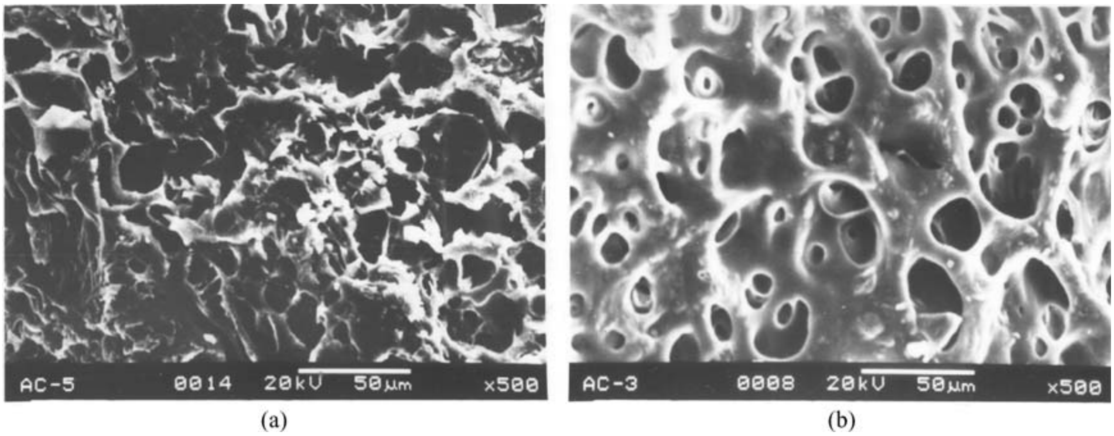


Fig. 3 Effect of chemical ratio on weight loss, chemical recovery, BET surface area and pore volume of activated carbons obtained by $ZnCl_2$

with increase in chemical ratio. The two distinct regions of pore evolution can be observed. In the first region (chemical ratio $< 150\%$), the surface area and the micropore volume increase at a high rate, while in the second region (chemical ratio $> 150\%$), in addition to pore opening, some portion of the chemicals are responsible for the widening of the micropores, resulting in slower rate of increase in porosity. The first one is the micropore formation which starts with the addition of chemicals to the raw materials and the second one is the pore widening which is the result of chemical effect inside the opened pores. Pore widening normally begins when there are a number of opened pore in the structure, therefore, it starts acting when the chemical



(a) (b)

Fig. 4 SEM micrographs of the *Sterculia alata* nutshell (a) before and (b) after carbonization (at 500°C, 60 min, 200% chemical ratio)

ratio is reasonably high. There is a range in the chemical ratio where both mechanisms are competing with each other. The destructive effect of high $ZnCl_2$ ratio on the micropore structure of active carbon can be observed in the second region (Ahmadpour et al., 1997).

3.1.4 SEM analysis of the activated carbons

Scanning electron microscopy (SEM) technique was employed to observe the surface physical morphology of the nutshell derived activated carbon. Figure 4 shows the SEM photographs of the *Sterculia alata* nutshell before and after the carbonization at the optimum operating condition with five hundred times magnification. Pores of different size and shape could be observed. It can be seen from the micrographs that the external surface of the chemically activated carbon is full of cavities. The reason for the formation of the cavities on the $ZnCl_2$-activated carbon is not clear. According to the micrograph, it seems that the cavities resulted from the evaporation of $ZnCl_2$ during carbonization, leaving the space previously occupied by the $ZnCl_2$. The carbonization temperature for chemical activation was too low to cause the agglomeration of the char structure. Since the carbonization temperature for physical activation is high (900°C), caking and agglomeration occurred on the char structure and thus resulted in the formation of chars with an intact external surface (Ahmadpour et al., 1997). It can also be seen that some salt particles are scattered on the surface of the activated carbon, probably due to the presence of remaining zinc chloride or other metal compounds on the activated carbon. Some particles were even trapped into the pores and could possibly block the entry of pores to some extent. The above findings were verified with the results obtained by Hg porosimetry of activated carbon. Clearly, it demonstrates that the adsorptive capacity of the products could be further increased on improving the washing procedure.

3.2 Phenol removal

3.2.1 Kinetics of phenol adsorption process

The relationship between contact time and phenol removal by activated carbon obtained by $ZnCl_2$ activation of *Sterculia alata* nutshell for four different initial concentrations is presented in Fig. 5 for an adsorbent dosage of 5 g/l and at natural pH of the solution. It can be seen from the Figure that an increase in initial phenol concentration results in decrease in the phenol removal. It can be concluded that the rate of phenol binding with activated carbon is more at initial stages, which gradually decreases and becomes almost constant after a period of 300 min. Similar types of observations have already been reported by several authors (Banat et al., 2004; Goud et al., 2005).

Numerous kinetic models have been proposed to elucidate the mechanism by which pollutants may be adsorbed. The mechanism of adsorption depends on the physical and/or chemical characteristics of the adsorbent as well as on the mass transport process. In order to investigate the mechanism of phenol adsorption, three kinetic models were considered as follows:

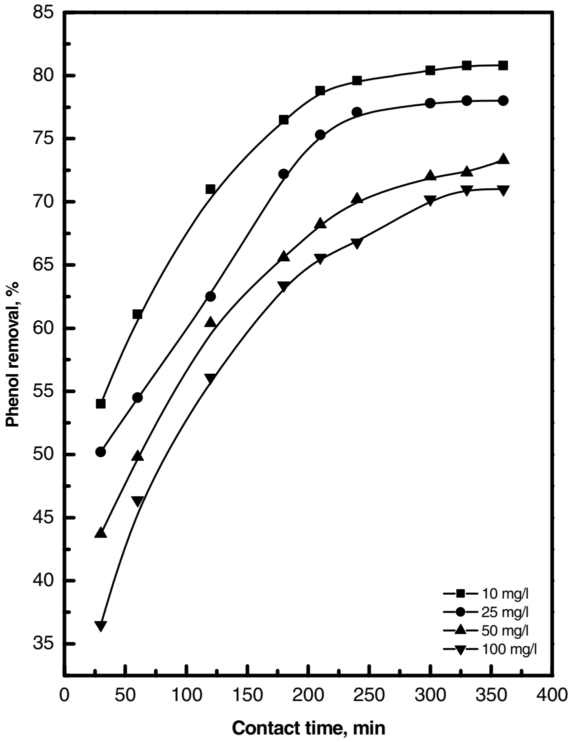


Fig. 5 Effect of contact time on percent removal of phenol (adsorbent dose = 5 gm/l)

Lagergren model

Lagergren (1898) proposed a pseudo-first-order kinetic model. The integral form of the model is

$$\log(q_e - q) = \log q_e - \frac{K_{ad}}{2.303}t \tag{3}$$

where q is the amount of phenol sorbed (mg g^{-1}) at time t (min); q_e is the amount of phenol sorbed at equilibrium (mg g^{-1}) and K_{ad} is the equilibrium rate constant of pseudo-first-order adsorption (min^{-1}). This model was successfully applied to describe the kinetics of many adsorption systems.

Pseudo-second-order model

The adsorption kinetics may also be described by a pseudo-second-order (Ho and Mckay, 1999) reaction. The linearized-integral form of the model is

$$\frac{t}{q} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \tag{4}$$

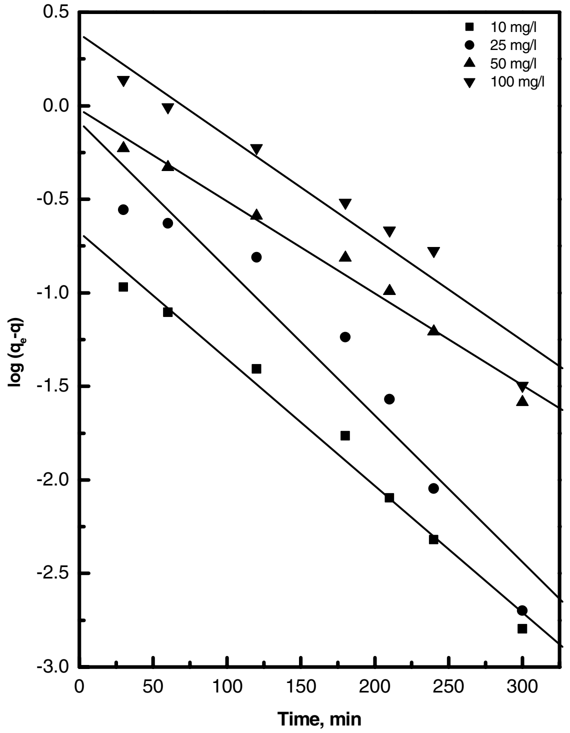


Fig. 6 Kinetics of phenol removal according to the Lagergren model

where k_2 is the pseudo-second-order rate constant of adsorption.

Diffusion model

The intraparticle diffusion model is based on the theory proposed by Weber and Morris (1963). According to this theory,

$$q = k_d\sqrt{t} \tag{5}$$

where k_d is the rate constant of intraparticle diffusion (mg g^{-1} min $^{-1/2}$).

The applicability of the above three models can be examined by each linear plot of $\log(q_e - q)$ vs t, (t/q) vs t, and q vs $t^{1/2}$ respectively and are presented in Figs. 6, 7 and 8. In order to quantify the applicability of each model, the correlation coefficient, R^2, was calculated from these plots. The linearity of these plots indicates the applicability of the three models. However, the correlation coefficients, R^2, showed that the pseudo-second-order model, an indication of a chemisorption mechanism, fits better the experimental data

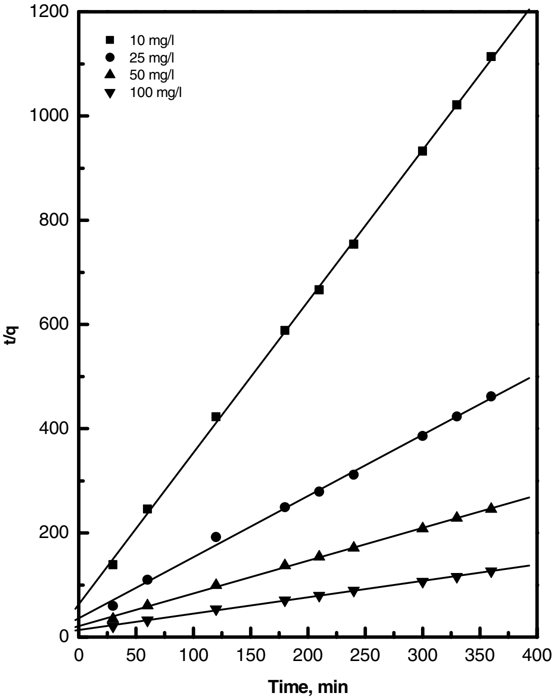


Fig. 7 Kinetics of phenol removal according to pseudo-second-order model

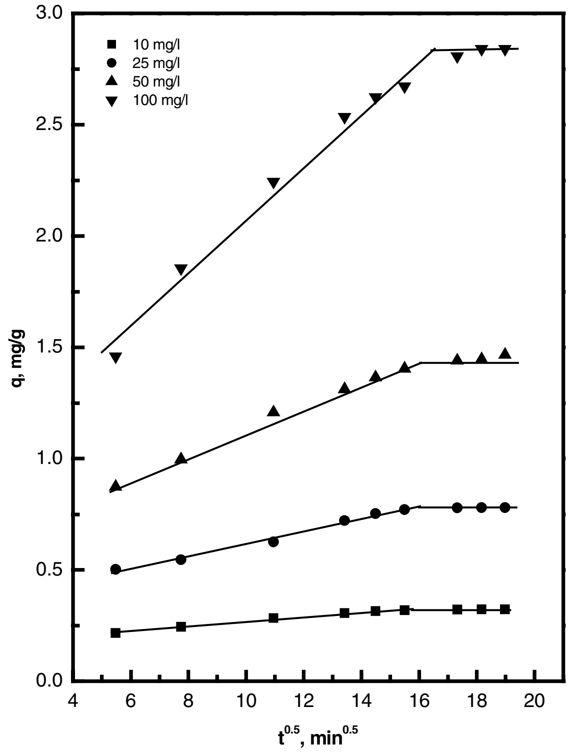


Fig. 8 Kinetics of phenol removal according to the intraparticle diffusion model

($R^2 > 0.998$) than the pseudo-first-order model (R^2 is in the range of 0.958–0.991). The intraparticle diffusion was also involved in the adsorption of phenol by activated carbons (Fig. 8). The linear portion of the plot for a wide range of contact time between adsorbent and adsorbate does not pass through the origin. This deviation from the origin or near saturation may be perhaps due to the difference in the rate of mass transfer in the initial and final stages of adsorption (Pandey et al., 1986; Mohanty et al., 2005). Further, such deviation from origin indicates that the pore diffusion is not the only rate controlling step (Mohanty et al., 2005). From the Fig. 8 it may be seen that there are two distinct regions—the initial pore diffusion due to external mass transfer effects (R^2 is in the range of 0.956–0.984) followed by the intraparticle diffusion (R^2 is in the range of 0.988–0.997). The better fit of data in the second region clearly indicates that the intraparticle diffusion plays a major role for this adsorbent-adsorbate system (Allen et al., 1989; Mohanty et al., 2005).

3.2.2 Adsorption isotherms

Several models have been used in the literature to describe the experimental data of adsorption isotherms (Banat et al., 2004; Goud et al., 2005). The Langmuir and Freundlich models are the most frequently employed models. In the present work both models were used.

The phenol sorption isotherm followed the linearized Langmuir model as shown in Fig. 9. The Langmuir equation relates solid phase adsorbate concentration (q_e), the uptake, to the equilibrium liquid concentration (C_e) as follows:

$$q_e = \left(\frac{K_L b C_e}{1 + b C_e}\right) \tag{6}$$

where K_L and b are the Langmuir constants, representing the maximum adsorption capacity for the solid phase loading and the energy constant related to the heat of adsorption respectively. It can be seen from figure that the isotherm data fits the Langmuir equation well ($R^2 = 0.9997$). The values of K_L& b were determined from the figure and were found to be 11.16 mg/g & 0.00153 l/mg respectively. These values are well in agreement with the values reported by several others (Banat et al., 2004; Goud et al., 2005).

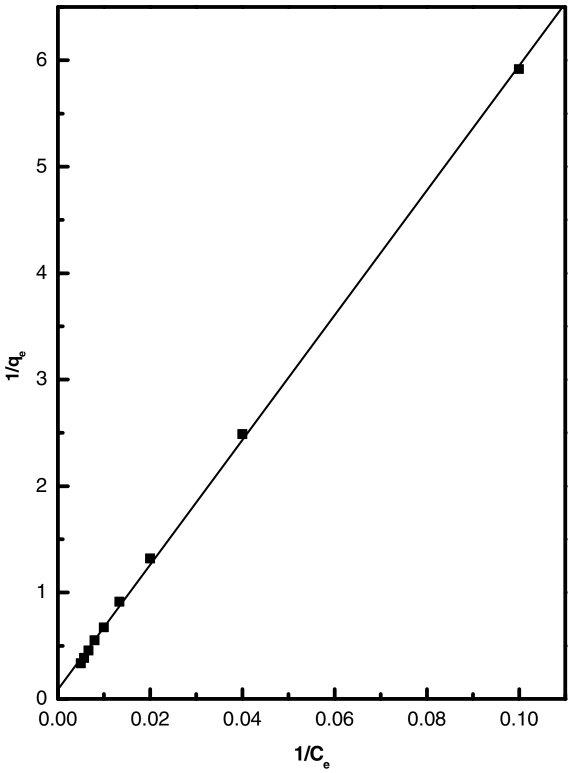


Fig. 9 Langmuir isotherm model. Adsorbent dose = 5 gm/l, temperature = 25°C

The phenol adsorption isotherm followed the linearized Freundlich model as shown in Fig. 10. The relation between the phenol uptake capacity 'q_e' (mg/g) of adsorbent and the residual phenol concentration 'C_e' (mg/l) at equilibrium is given by,

$$\ln q_e = \ln k + \frac{1}{n} \ln C_e \tag{7}$$

where the intercept ln k is a measure of adsorbent capacity, and the slope $1/n$ is the sorption intensity. The isotherm data fit the Freundlich model well ($R^2 = 0.9997$). The values of the constants k and $1/n$ were calculated to be 0.0184 and 0.953. Since the value of $1/n$ is less than 1, it indicates a favorable adsorption (Banat et al., 2004; Goud et al., 2005).

3.2.3 Effect of pH on removal of phenol

The adsorption of phenol by activated carbons obtained by $ZnCl_2$ activation was studied at various values of pH. The experiments were performed for an initial concentration of 100 ppm for different adsorbent doses (1, 5 and 10 g/l) and the results are shown in Fig. 11. It is clear from this figure that the percentage adsorption of phenol increases with increase in pH from pH 2.0 to 3.5 along with the increase in adsorbent dose and decreases thereafter. It is obvious that as the adsorbent dose is increased, the number of active sites on the surface of activated carbons available for adsorption also increases, hence there is maximum adsorption reported for the adsorbent dose of 10 g/l. It is important that the maximum adsorption at all the concentrations as well as adsorbent doses takes place at pH 3.5.

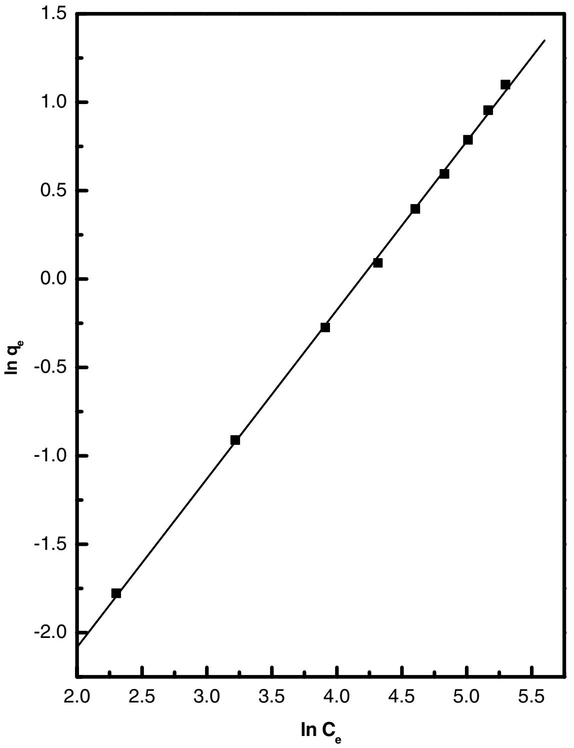


Fig. 10 Freundlich isotherm model. Adsorbent dose = 5 gm/l, temperature = 25°C

Figure 11 shows that phenol is strongly adsorbed at low pH values, and only small changes in the removal of phenol takes place up to pH 8.0. This behavior can be explained considering the nature of the adsorbent at different pH in phenol adsorption. The amount of sorbed phenol seemed to be related to the dissociation constant (*pKa*). The *pKa* value for phenol is 9.99. At pH values greater than the *pKa* value, the degree of ionization of phenol increases and thus its sorbability decreases (Banat et al., 2004). This is due to thefact that the

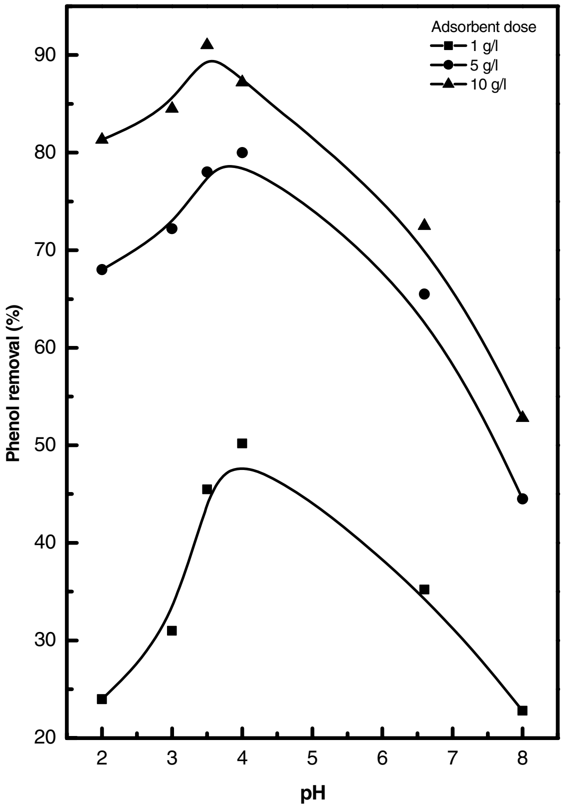


Fig. 11 Effect pf pH on phenol removal

surface of activated carbons contains a large number of surface functional groups. The pH dependence of phenol adsorption can largely be related to type and ionic state of these functional groups and also on the phenol chemistry in solution. Adsorption of phenol upto pH 3.5 suggests that the negatively charged phenolate ions bind through electrostatic attraction to positively charged functional groups on the surface of activated carbon because at this pH more functional groups carrying positive charge would be exposed. But at pH above 3.5, it seems that activated carbon possesses more functional groups carrying a net negative charge, which tends to repulse the anions. However, there is removal above pH 3.5 also, as indicated by the Fig. 11, but the rate of removal is considerably reduced. Hence, it could be said that above pH 3.5, other mechanism like physical adsorption on the surface of adsorbent couldhave taken an important role in the adsorption of phenol and exchange mechanism might have reduced (Banat et al., 2004; Goud et al., 2005).

4 Conclusions

This study has demonstrated that reasonably good surface area activated carbons can be prepared from the chemical activation of *Sterculia alata* nutshell with $ZnCl_2$ as activating agent. For the carbonization of the $ZnCl_2$ treated sample, the release of moisture and $ZnCl_2$ represents most of the evolution, indicating that $ZnCl_2$ plays an important role in retarding tar escape during carbonization. The washing process following carbonization with $ZnCl_2$ has a significant influence on the surface properties of resulting char. It was found that acid washing is a necessary step for the preparation of high-porosity carbons.

Study of various parameters during chemical activation revealed that the most important variable is the ratio of chemical agent to the nut precursor. The other important operating variables with a direct effect on the porosity development are temperature and carbonization time. Under the experimental conditions investigated, the best conditions for the production of high surface area activated carbon from *Sterculia alata* nutshell by chemical activation are: chemical ratio (activating agent/precursor) of 200%, carbonization time of 1 h and carbonization temperature of 500°C. At this optimal condition, the BET surface area and micropore volume obtained were 712 m^2/g and 0.542 cm^3/g respectively.

The batch adsorption tests indicate that the *Sterculia alata* nutshell derived activated carbon had a notable adsorption capacity for phenol from aqueous solutions. The kinetics of phenol adsorption followed nicely the pseudo-second-order rate expression. The Langmuir and Freundlich models also fit the isotherm data well. Solution pH has great effect on the uptake of phenol. The data thus obtained may be helpful for designing and establishing a continuous treatment plant for water and wastewaters enriched in phenol.

Nomenclature

$1/n$	sorption intensity, dimensionless
b	Langmuir constant, l/g
C_e	equilibrium phenol concentration, mg/l
C_o	initial phenol concentration, mg/l
Chemical ratio	activating agent ($ZnCl_2$)/precursor, g/g
CR	chemical recovery, g/g
k	measure of adsorbent capacity, l/g
k_2	pseudo-second-order rate constant, g/mg min
k_d	rate constant of intraparticle diffusion, mg/g $\text{min}^{1/2}$
K_{ad}	equilibrium rate constant of pseudo-first-order adsorption, min^{-1}
K_L	Langmuir constant, mg/g
q_e	amount of phenol adsorbed at equilibrium, mg/g
T	temperature, °C
t	time, min
V	volume of the solution, l
W	weight of the adsorbent, g
W_c	weight of chemical used, g
W_{pf}	weight of product after washing, g
W_{pi}	weight of product before washing, g

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