COPPER REMOVAL USING ACID ACTIVATED PEANUT HUSK FROM AQUEOUS SOLUTION

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Abstract. Peanut husk, an agricultural waste is a precursor biomass veteran for chemical activation using phosphoric acid. The work deals with activation of peanut husk using phosphoric acid followed by its application. The effect of various parameters such as impregnation ratio, temperature, particle size and agitation speed on copper adsorption were also studied. Physical characterization of the activated adsorbent was done using SEM, BET and FTIR to understand the modifications. The optimum parameter values of chemical activation influencing adsorption were 20% phosphoric acid concentration, 1:8 acid impregnation ratio, 60 °C activation temperature and 350 rpm stirring speed. The copper uptake was 14.3 mg/g for peanut husk whereas by chemical activation it was increased to 17.88 mg/g. Thus adsorption capacity of peanut husk can be successfully improved using phosphoric acid activation.

Keywords: peanut husk, phosphoric acid, chemical activation, bio adsorbent, copper adsorption, adsorption capacity.

Introduction

Due to rapid industrialization and urbanization heavy metals have entered into the ecological system in the form of water pollution (Sari, Tuzen 2008; Uluozlu *et al.* 2008; Donghee *et al.* 2010). These heavy metals get accumulated in living organisms, consequently leading to environmental hazards (Loutseti *et al.* 2009; Batley *et al.* 2004). Copper, one of the metals finds varied application in diverse fields such as electroplating, etching, rinsing, chemical and mechanical polishing, etc. which lead to accumulation of gallons of wastewater contributing to heavy metal pollution (Ingle *et al.* 2014). Moreover, copper being a micronutrient if consumed in the form of inhalation in higher dosage leads to be fatal, hence there is a need to remove copper before it is discharged (Ali *et al.* 2013). The heavy metals can be removed from the waste water by ion exchange (Gaikwad *et al.* 2010), sedimentation (Song *et al.* 2000), biological operations (Wang 2002), membrane processes (Valenzuela *et al.* 2005), chemical precipitation (Barakat 2011). But at the same time these conventional methods have drawbacks of high capital and operational cost (Sharma, Forster 1994). Moreover, these techniques fail to remove heavy metals when present at low concentration. From past few years researchers are interested in substituting costly methods of water treatment by low cost

and agricultural by-product adsorbent (Namasivayam, Ranganathan 1995). Adsorption process has been proven one of the best water treatment technologies around the world and activated adsorbent is undoubtedly considered as a better adsorbent for the removal of diverse types of pollutants from water (Bhatnagar, Sillanpaa 2010). Adsorption using biological waste is an effective technique for the removal of heavy metals and the process is termed as bio sorption. This effective technique depends on the parameters like capacity, affinity, specificity of bio sorbent and the conditions in effluents. Bio sorption can be used for the treatment of wastewater containing low heavy metal content and found to be an inexpensive, simple and effective alternative to conventional methods. Bio sorption is the capability of active sites on the surface of biomaterials to bind and concentrate heavy metals from even the most dilute aqueous solutions (Wang, Chen 2006). Minimization of chemical sludge, recovery of the adsorbents and possibility of metal recovery are added advantages of these potential adsorbents. Among the agricultural waste reported, peanut husk is considered as one of the best precursors for copper removal as it is a cheaper, abundantly available. Although the bio sorbents are cheaper in cost, the low adsorption capacity is the major problem which can be improved by chemical activation. It has been reported that there is an increase in the adsorption of metal

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ions after chemical activation of peanut hull (Wafwayo *et al.* 1999). Similar kind of behavior of higher adsorption of copper was obtained by other researchers (Charmarthy *et al.* 2001)

Activation of biosorbent consists of multiplying amount of pores of certain carbonaceous material to produce extremely porous structure. Activation can be performed by physical or/and chemical means. Physical activation involves carbonization of carbonaceous materials at high temperatures (500–900 °C) in an inert atmosphere followed by activation in the presence of activating agents such as CO_2 or steam resulting in activated carbon. In chemical activation, the precursor is impregnated with a chemical activator such as $ZnCl_2$, H_3PO_4, KOH, H_2SO_4 etc. This activated material can be used directly or the impregnated precursor is further heated in an inert atmosphere to convert it in activated carbon. Chemical activation is preferred over physical activation as it is carried at lower temperature with less activation time. Moreover, it develops better porous structure and also gives high adsorption capacity (Guo, Rockstraw 2007). It is revealed from the work of other researchers that phosphoric acid has the capability of creating physical and chemical modifications on the botanical structure by penetration, particle swelling, and partial dissolution of the biomass, bond cleavage and reformation of new polymeric structures resistant to thermal decomposition (Girgis *et al.* 2002).

The present work is focused on the preparation of activated adsorbent using phosphoric acid as an activating agent. The effect of various parameters affecting activation such as concentration of acid, impregnation ratio, temperature, particle size and stirring speed is studied. In order to understand the changes induced by the activation as well as to characterize the adsorbent, the acid activated materials before and after adsorption are analyzed and characterized using Scanning Electron Microscope (SEM, Jeol) and Fourier Trans-form Infrared Spectroscopy (FTIR, Bruker) and Specific surface area and porosity analyzer (BET, PMI Incorp. Ltd.).

1. Materials and methods

1.1. Materials

Peanut husk (APMC Market Vashi, Navi Mumbai) were washed extensively using tap water for 1–2 h to remove any mud and soil particles adhered to it. Further it was washed several times with deionized water and then dried in an oven at 80 °C for 48 h. The dried husk was grounded in a fine powder, sieved by ASTM standard sieve and stored in an airtight container for further use. Phosphoric acid was obtained from S.D. Fine Chem. Ltd., Mumbai, India. All the chemicals used were analytical grade and were used as received from the suppliers. The deionized

water for the experiments was obtained from Sartorious stedimarium Water Purifier Unit.

1.2. Chemical activation of peanut husk

The chemical activation was performed in a 100 ml glass reactor provided with stirrer. Experiments were carried out in a reactor using 15 g of peanut husk and correspondingly phosphoric acid. The acid-adsorbent mixture with various impregnation ratios (1:4–1:12) was agitated at different stirring speeds (100–650 rpm) using mechanical stirrer within the temperature range 20–80 °C. The temperature is maintained throughout the experiment using a water bath. After predefined time, the activated samples were filtered and the pH was adjusted to 7 with 0.1 M NaOH and 0.1 M HCl using pH meter (Labtronic digital pH meter Model No. LT-10). Then the adsorbent was dried at 100 °C in the oven.

1.3. Copper adsorption experiment

In order to study the effect of activation on copper adsorption, 100 mL metal ion solution prepared in the laboratory of 100 mg/L copper concentration with initial pH 4.8 is equilibrated with 0.5 g of chemically activated adsorbent. Sorption process is carried out in a conical flask placed in Remi Orbital incubator shaker at room temperature (30±2 °C) for 12 h till equilibrium was attained. After equilibrium, 1 mL of sample was withdrawn to determine the metal ion concentration. The metal ion concentration was measured by using standard method for copper ion determination with PAN indicator (1-(2-Pyridylazo)-2-naphthol) using a UV-VIS Spectrophotometer. This method was used for all the experiments to determine the effect of chemical activation on adsorption capacity of the adsorbent. The metal uptake at equilibrium was evaluated from the given Eq. (1):

$$Metal\,uptake\left(q\right)=\frac{\left(C_i - C_e\right)}{M}\cdot V \,, \qquad (1)$$

where C_i is the initial metal ion concentration before sorption process in mg/L; C_e is the equilibrium metal ion concentration after sorption process in mg/L; V is the volume of metal ion solution in mL; M is the mass of bio sorbent taken in g.

1. 4. Characterization of acid treated peanut husk

In order to analyze the surface area and porosity change after phosphoric acid treatment the standard method of BET analysis (BET, PMI Incorporated Ltd.) was carried out. The surface area is evaluated by nitrogen multi-layer adsorption which is carried out at 77 K, measured as a function of relative pressure. Scanning Electron Microscope (SEM, Jeol) and Fourier Transform Infrared Spectroscopy (FTIR, Bruker) were done to examine the

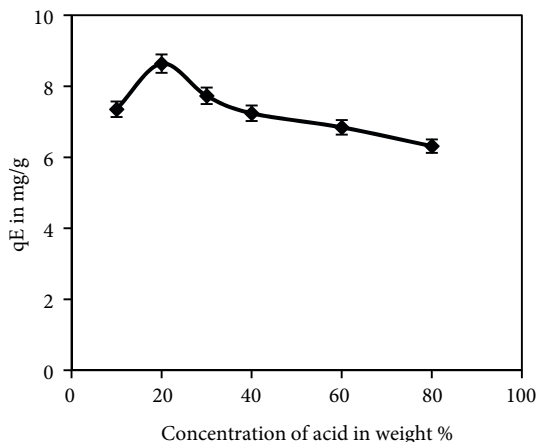changes in the structure of acid activated peanut husk before and after adsorption.

Fig. 1. Effect of acid concentration in weight % at 30 °C, impregnation ration 1:12 and speed of stirring 150 rpm
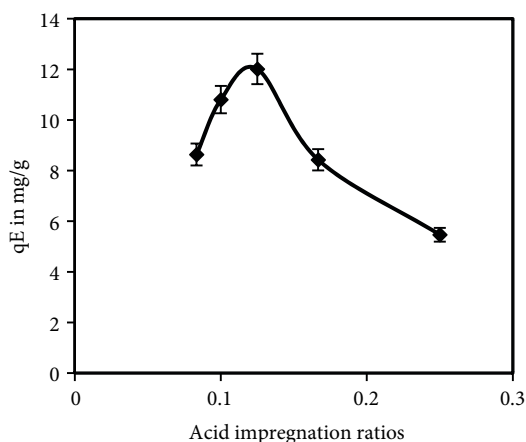

Fig. 2. Effect of acid impregnation ratios at 20% by weight acid concentration at 30 °C and speed of stirring 150 rpm
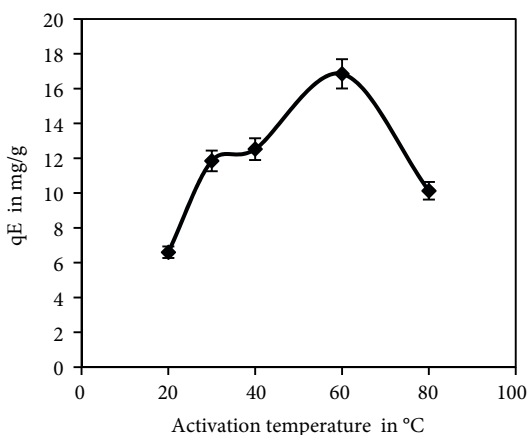

Fig. 3. Effect of activation temperature at 20% by weight acid concentration, impregnation ratio 1:8 and speed of stirring 150 rpm

2. Results and discussions

2.1. Effect of phosphoric acid concentration

The surface characteristics and porosity of the bio sorbent are mainly decided by the concentration of acid used for activation. The effect of acid concentration was studied by varying the concentration from 10 to 80%. The experiments were carried out at 30 °C with 1:12 impregnation ratio and stirring speed 150 rpm. It is known that phosphoric acid swells the botanical material and modifies the surface characteristics. The adsorption of copper after activation has increased at lower concentration of acid till 20% and then with further increase in concentration of acid the copper uptake has decreased considerably (Fig. 1). This may be due to the structural abrasions on the surface of the peanut husk. The copper ion uptake was 8.68 mg/g at 20% (weight) of phosphoric acid concentration. Similar trend is observed by (Rahman *et al.* 2005), where 20% (weight) of phosphoric acid concentration was used for activation of rice husk for the removal of malachite green dye.

2.2. Effect of acid impregnation ratios

Acid impregnation ratios are defined as weight of adsorbent to the weight of acid. This parameter decides the quantity of phosphoric acid required for activation. The acid impregnation ratios was studied using different ratio in the range of 1:4–1:12 by keeping other parameters constant at acid concentration 20%, temperature 30 °C and speed of stirring 150 rpm. With an increase in impregnation ratios the copper uptake increases till 1:8 impregnation ratios (Fig. 2).

This may be due to increase in size of active site of peanut husk with the interaction with phosphoric acid. The impregnation of acid might work through alteration in the surface functional group chemically rather than physical interaction (Kim *et al.* 2006). The copper uptake has increased up to the maximum value of 12.01 mg/g at optimum impregnation ratio 1:8 due to pore enlargement and surface modification. The pores may get ruined by the presence of excess acid as the impregnation ratios is decreased beyond the optimum value. So for the further studies the acid impregnation ratios is kept constant at 1:8.

2.3. Effect of temperature on chemical activation

In order to study the effect of temperature, the activation of peanut husk was carried out within the temperature range 20 to 80 °C. The other parameters like impregnation ratio 1:8, acid concentration 20% and the stirring speed of 150 rpm was kept constant throughout the study. It is observed that the copper uptake after activation of peanut husk was increased with elevation in temperature from 20 to 60 °C (Fig. 3). This is because an increase in

temperature initiates the rate of diffusion of phosphoric acid into the pores of peanut husk. The diffusion results in the conversion of micro pores to mesopores and macro pores. The copper uptake after activation increased from 6.6 mg/g at 20 °C to 16.8 mg/g at 60 °C. The reduction in the adsorption capacity of copper beyond 60 °C may be consequence of increase in temperature, resulting into the pore destruction. The adsorption of copper using peanut husk as a precursor was also carried out and the copper uptake was found to be 9.6 mg/g whereas the phosphoric acid modified peanut husk has proved better as the copper uptake was 16.8 mg/g which is nearly doubled.

2.4. Effect of stirring speed on chemical activation

The stirring speed is an important parameter in the activation process, as it dictates the extent of mass transfer. The experiments were carried out at varied speed from 100--850 rpm. The experimental parameters of acid concentration, impregnation ratio, activation temperature were maintained at optimum values. It is seen that the copper uptake increased from 10.8 mg/g to 17.88 mg/g as the stirring speed is increased from 100 rpm to 350 rpm (Fig. 4). This is in relation with the fact that an increase in stirring speed reduces the film resistance created by the boundary layer surrounding the adsorbent particles (Aman *et al.* 2008). Due to low film resistance the phosphoric acid diffuses within the peanut husk, thereby modifying the pore structure. Thus increase in stirring speed increases the external film mass transfer coefficient leading to higher copper uptake. But beyond 350 rpm the surplus stirring may damage the pores resulting in decrease in copper uptake.

2.5. Effect of particle size on chemical activation

The particle size determines the time required by the phosphoric acid for transport within the pores which results in the formation of mesopores and micro pores (Ricordel *et al.* 2001). The particle size was varied from 150 microns to 720 microns to analyze the effect of particle size on chemical activation. The other experimental parameters were upheld at acid concentration 20%, impregnation ratio 1:8, activation temperature 60 °C and stirring speed 350 rpm. It is clear that with an increase in particle size the copper uptake decreases (Fig. 5).

The surface area of the particle increases with decrease in particle size. The phosphoric acid diffuses in a shorter period of time into the peanut husk which has smaller particle size as there is direct relation between equilibrium time and particle size. The adsorption of copper using peanut husk of 500 microns was carried out, which showed that copper uptake 9.6 mg/g whereas the phosphoric acid modified peanut husk has given copper uptake 12.3 mg/g.

2.6. Characterization of chemically activated peanut husk

To study the effect of chemical activation on the surface morphology and porosity, SEM analysis was carried out for the untreated peanut husk and the chemically activated peanut husk. In SEM micrograph of the untreated peanut husk there are some flaky structures and rudimentary pores while SEM micrograph of the chemically treated peanut husk has wide pores as a result of the treatment with phosphoric acid which is corrosive and leads to surface leaching (Fig. 6). This type of activation is dependent on the material under study and peanut husk is pectin rich, which undergoes swelling, during which the arrangement of the molecules in the direction of longitudinal axis is unaltered but the lateral bonds undergo drastic modification in terms of void formations likely due to the chemical activity of the acid (Smisek, Cerny 1970). Thus the SEM image clearly shows that the activation has played a major role in the enhancement of surface adsorptive capacity of the peanut husk. The SEM micrograph of the chemically activated peanut husk after the copper adsorption process
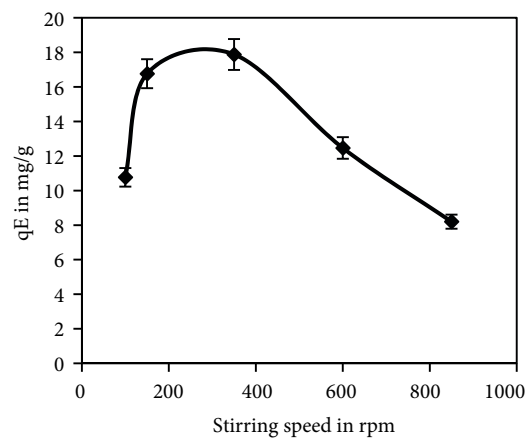

Fig. 4. Effect of stirring speed at 20% by weight acid concentration impregnation ratio 1:8 and temperature 60 °C
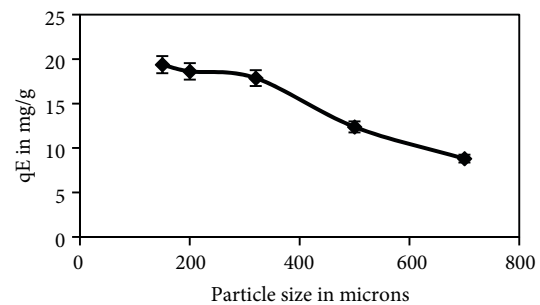

Fig. 5. Effect of particle size at 20% by weight acid concentration, impregnation ratio 1:8, temperature 60 °C and stirring speed 350 rpm

is shown in (Fig. 6c) which shows decreased void space and the surface charges as compared with SEM images (Fig. 6a and Fig. 6b) indicating the deposition of copper on the voids.
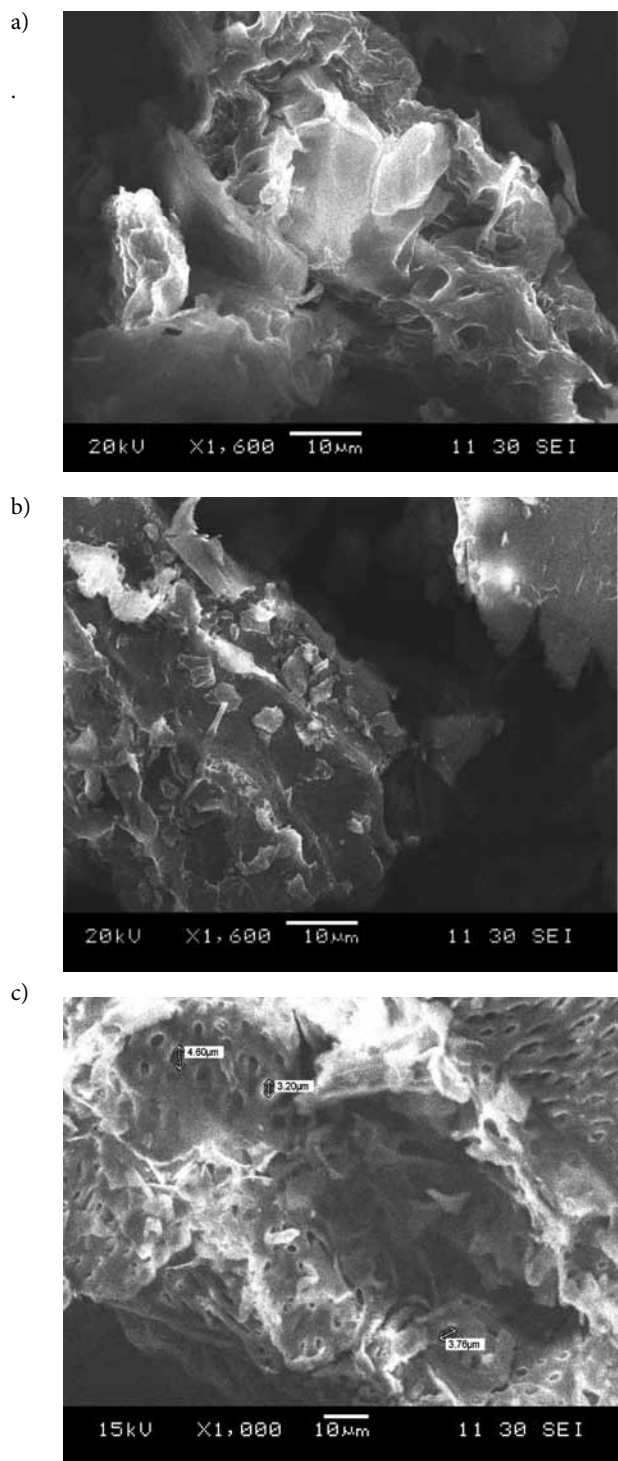
a)

b)

c)

Fig. 6. SEM micrographs of untreated peanut husk, chemically activated peanut husk; and chemically activated peanut husk after adsorption: a) peanut husk; b) chemically activated peanut husk c) chemically activated peanut husk after adsorption

2.7. Fourier transform infrared spectroscopy

Infrared spectroscopy provides information on the chemical structure of the adsorbent material. Figure 7 shows FTIR spectra of the peanut husk and the acid modified peanut husk. The most of characteristic changes are observed with the range of 1700–1730 cm^{-1}. The band centred at 1731 cm^{-1} is ascribed to the stretching vibrations of carboxyl groups on the edges of layer planes or to conjugated carbonyl groups (C-O in carboxylic acid and lactones groups). The spectrum of the acid treated adsorbent shows a significant change in this band showing the effect of phosphoric acid on the husk. The broad band between 1300 and 900 cm^{-1} in acid treated peanut husk has a maximum at 1070–1080 cm^{-1}.

The absorption in this region is the characteristic for phosphorus and phosphor carbonaceous compounds. The peak at 1057–1033 cm^{-1} is ascribed to ionized linkage P$^+$O$^-$ in acid phosphate esters and to symmetrical vibration in a chain of P-O-P (polyphosphate). The spectra therefore suggest the formation of P-containing carbonaceous structures like acid phosphates and polyphosphates in phosphoric acid-activated peanut husk.

2.8. BET surface areas and pore structure

The BET surface areas and porous structures of samples were investigated based on nitrogen adsorption-desorption.

The BET specific surface area (SBET) of untreated peanut husk and acid-activated peanut husk are 18.088 and 31.140 m^{2}g^{-1}, respectively showing that there is a large increment in the surface area due to the acid treatment.

Figure 8 shows nitrogen adsorption-desorption isotherms for the two samples. The isotherms of all samples show type III isotherms, indicating the presence of macropores throughout the material. This type of isotherm is a characteristic of weak gas-solid interactions. This weakness causes uptake at low pressure to be small which is quite evident from the figure. But once the molecule is adsorbed, the adsorbate – adsorbate interactions will further promote the adsorption process. After the acid treatment
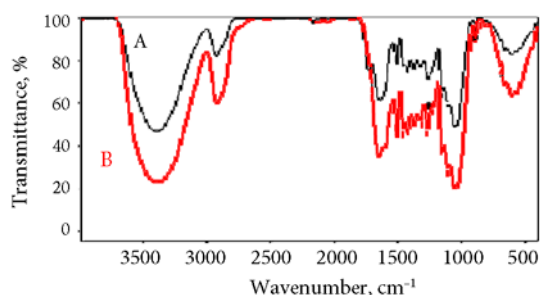

Fig. 7. FTIR of untreated peanut husk (A) and chemically treated peanut husk (B)

Table 1. Comparison of activating agents used for chemical activation of various adsorbent (Low *et al.* 1995; Acar, Eren 2006; Ngah, Hanafiah 2008; Baccara *et al.* 2009; Dubey, Gopal 2007; Li *et al.* 2007; Shukla, Pai 2005)

Sr. no	Agricultural waste as precursor	Chemical agent	Metal ions	Metal uptake
1	Banana pith Nitric acid	HNO_3	Cu^{2+}	13.46
2	Sawdust (Poplar tree)	H_2SO_4	Cu^{2+}	13.95
3	Sawdust (Poplar tree)	NaOH	Cu^{2+} Zn^{2+}	6.95 15.8
4	Carbon from Tunisian-olive waste	H_3PO_4	Cu^{2+}	35.5
5	Sawdust	Reactive Orange 13	Cu^{2+} Ni^{2+} Zn^{2+}	8.07 9.87 17.09
6	Groundnut husk	H_2SO_4 followed by silver impregnation	Cr^{6+}	11.4
7	Peanut husk	H_2SO_4	Cu^{2+}	10.15
8	Groundnut shells	Reactive Orange 13	Cu^{2+}	7.60
9	Jute fibers	Reactive Orange 13	Cu^{2+} Ni^{2+}	8.4 5.26
10	Peanut husk (present study)	H_3PO_4	Cu^{2+}	17.88

Fig. 8. N_2 adsorption – desorption isotherm of Acid treated peanut husk (ATPH) and Untreated peanut husk (UTPH)
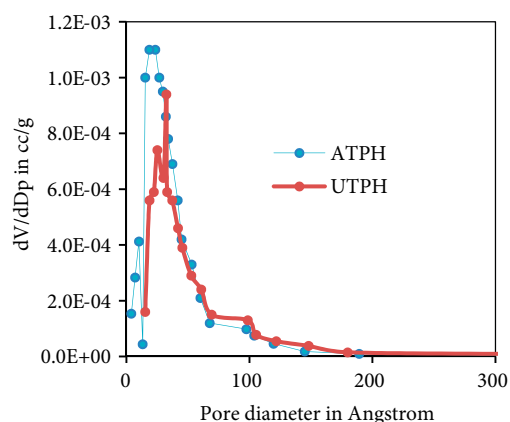
Fig. 9. Pore diameter distribution of acid treated peanut husk (ATPH) and untreated peanut husk (UTPH)

there was an increase in the surface leading to higher adsorption at higher relative pressures.

Also, from Figure 9, it can be concluded that the pore size distribution is quite broad in both the materials and the pore volume increased from 0.0214 cc/g to 0.03548 cc/g after the acid treatment. Thus, this type of results confirms the presence of mesoporous and macroporous nature of the adsorbent material.

Also, Table 1 depicts the various chemical agents used for activation and their adsorption capacities. From the table it can be concluded that the phosphoric acid is found to be the excellent chemical activating agent for enhancing the adsorption capacity.

Conclusion

1. The chemical activation of peanut husk by phosphoric acid has enhanced the copper adsorption capacity of peanut husk as compared to untreated peanut husk. Thus the activation of ligno-cellulosic material by phosphoric acid has improved the surface characteristics.
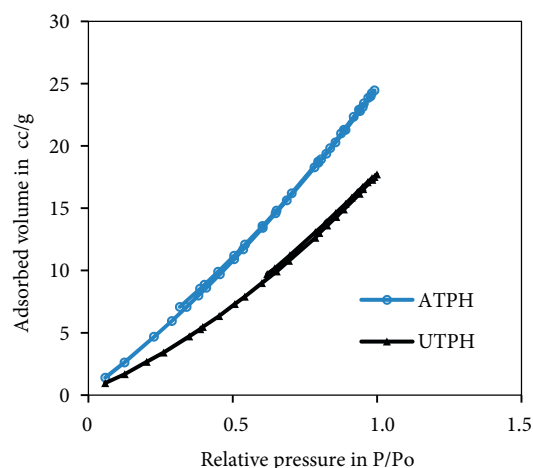2. The optimum conditions obtained from the study of chemical activation are 20% phosphoric acid concentration, 1:8 acid impregnation ratio, 60 °C activation temperature and 350 rpm stirring speed.
3. This enhancement may be due to the surface modifications and pore widening as seen in SEM micrographs and FTIR images.
4. The results indicate that the chemical activation can be used as a technique to enhance the adsorption capacity of bio adsorbents.

Acknowledgement

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