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Ultrasound assisted chemical activation of peanut husk for copper removal

https://doi.org/10.1515/gps-2017-0162 Received November 22, 2017; accepted February 15, 2018; previously published online June 11, 2018

Abstract: The ultrasound assisted chemical activation of peanut husk using phosphoric acid was studied at a frequency of 20 kHz. Experiments were carried out for the activation of peanut husk in presence of ultrasound followed by the study of consequent effect on the adsorption behavior of copper. Effect of sonication during chemical activation on copper adsorption was studied with respect to various parameters such phosphoric acid concentration, acid impregnation ratio, temperature, duty cycle, sonication time, power and the probe height dipped in the adsorbent-acid slurry. Results showed that after the application of ultrasound during the activation process, copper uptake capacity of the adsorbent is improved with increasing ultrasound power and the activation process is more feasible at 30°C. The copper uptake after activation treatment of 5 min in the presence of ultrasound was found to be 19.6 mg/g as against 17.8 mg/g by conventional chemical activation method performed for 20 min. Thus, after acid treatment in the presence of ultrasound, the adsorbent shows a good adsorption capacity at lower time of chemical activation.

Keywords: adsorption capacity; chemical activation; copper adsorption; peanut husk; ultrasound.

1 Introduction

Water is extremely important for the survival of living beings on the earth, even the living cell of human body is made of 80% of water. About 0.02% of the total available water on earth is immediately available for direct use in the form of water from river, lakes etc. Release of heavy metals in the environment has increased tremendously due to human activities like urbanization and industrialization

in past few decades [1]. The toxic nature of heavy metal is globally known and ill effects of heavy metals to human beings and environment are tremendous. Moreover, heavy metals are not eco-friendly as they are not bio-degradable and tend to amass in living organisms resulting in several diseases and disorders as well as it leads to severe ecological hazards [2, 3]. The abundant use of heavy metals like antimony (Sb), chromium (Cr), copper (Cu), lead (Pb), manganese (Mn), mercury (Hg), cadmium (Cd) have significantly damaged ecological environment. Copper is widely used in the industries like metallurgy industry, metal processing industries, electroplating industry and tannery industries etc. resulting in water pollution and affecting the fresh water bodies. Copper is a micronutrient but can be fatal if taken in high dosage. Continuous inhalation of copper containing sprays is linked with lung cancer [4]. Numerous techniques are available for water purification and metal recovery from waste water streams. Conventional methods are employed for the removal copper from waste water which include precipitation, coagulation, ion exchange, membrane separation processes (such as ultra-filtration, electro dialysis, reverse osmosis etc.) and adsorption [5]. However, chemical precipitation and electroplating treatment are uncommon as they produce large quantity of sludge, which has to be treated with great effort. Moreover, when copper ion concentration in aqueous solution is around 1-100 mg/l these methods are ineffective. Ion exchange, membrane processes are extremely expensive and cannot be used for large scale [6]. As an alternative technique, adsorption is a cost effective and has capability to overcome the disadvantages of conventional method. In the queue of these techniques, use of biological materials including living and non-living microorganisms as an adsorbent is more popular and the process is called biosorption. This is due to better performance microorganisms to remove or recover toxic and precious metals from industrial waste at ease of availability and low cost. This effective technique depends on the parameters like capacity, affinity, specificity of biosorbent and the conditions in effluents. Bio sorption can be used for the treatment of wastewater with squat heavy metal concentration as a cost effective, simple and valuable alternative to conventional methods. The ability of the biomaterial to bind and accumulate heavy metals is due to the active sites available on their surface and this

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sorption is popularly termed as biosorption [7]. Biomass is composed mostly of proteins, polysaccharides and fats, and has many functional groups able to bind heavy metal ions. Biomass includes low cost agricultural waste, algae, fungi, bacteria, microalgae, marine algae etc. In spite of availability of numerous techniques for the treatment of effluents containing heavy metals, sorption by agricultural waste is highly effective. Recent studies showed that common agricultural waste products can be used as potential biosorbent for the removal of heavy metals. Considering the environmental aspects, waste coming as agricultural material particularly which contains cellulose has witnessed adsorption for various pollutants. The basic composition of agricultural waste is hemicelluloses, lignin, lipids, proteins, simple sugar, water, hydrocarbons and starch containing various functional groups. These waste are abundantly available, has low cost and moreover renewable in nature which make them economical as well as eco-friendly. Moreover, these precursors have high carbon content and low ash content [8, 9]. Among the agricultural waste reported in the literature peanut husk is considered as one of the best precursor for copper removal as it is cheaper and abundantly available. Activation of biosorbent consists of multiplying amount of pores of certain carbonaceous material to produce extremely porous structure. Activation can be done by physical or/ and chemical means. In physical activation carbonization of carbonaceous materials is carried out at elevated temperatures (500-900°C) in an inert atmosphere followed by activation of the resulting in activated carbon in the presence of activating agents such as CO₂ or steam. Whereas in chemical activation, the precursor is impregnated with a chemical activator such as ZnCl₂, H₂PO₄, KOH, etc. and the impregnated precursor is heated in an inert atmosphere. Chemical activation is favored over physical activation as it is carried at lower temperature with less activation time. Moreover, it develops better porous structure with high yield [10]. The chemical activation process has been extensively used for the preparation of activated carbons from different agricultural wastes by various activators such as pine fruit shell by H₂SO₄ [11], walnut shell by ZnCl₂ [12], jute fiber by KOH [13], pistachio shell by H₂PO₄ [14], citrus fruit peel by H₃PO₄ [15], piassava fibers by ZnCl₃ [16], leather shaving waste by H₃PO₄ [17], vetiver root by H₂PO₄ [18]. There are very few reports available on the activation of carbonaceous materials of the adsorbent by ultrasound. When ultrasound waves are passed through a liquid medium, an alternating adiabatic compression and rarefaction cycle are observed [19, 20]. This phenomenon is termed as cavitation which is the formation, growth, and sudden collapse of the micro-bubbles in liquids which

are formed in the rarefaction cycle of the ultrasonic wave when a large negative pressure is applied to a liquid. The nature of the micro-bubbles formed is dependent on the frequency used and the fluid under study [19, 20]. Ultrasonic vibrations decrease the surface tension at the solid liquid interface, leading to diffusion of the liquid into the solid pores and enhancing the mass transfer. Therefore, the diffusion of dissolved species in liquids through porous media will be enhanced by ultrasound [21, 22]. In the past decade, literature reveals that the ultrasound plays a vital role in the adsorption and/or desorption processes. For example, for the sorption in a batch system, Schueller and Yang [23] found that ultrasound acts like a mixer, improving the mass transfer coefficients through cavitation and acoustic streaming.

The aim of present study is to carry out chemical activation of peanut husk in presence of ultrasound using H₃PO₄ as an activating agent and to optimize the various parameters affecting the chemical activation based on maximum copper (II) adsorption.

2 Materials and methods

2.1 Materials

Peanut husk (APMC Market Vashi Navi Mumbai) was washed extensively using tap water for 1-2 h to remove mud and soil particles. It was further washed with deionized water several times and dried in oven at 80°C for 48 h. The dried husk was grounded in a fine power, sieved by ASTM standard sieve of 320 µm and stored in an airtight container for further use. A copper (II) solution of 1000 mg/l concentration was prepared by dissolving appropriate quantity of CuSO, 5H,O (Mol. Wt. 249.68.), S. D. Fine Analytical grade in deionized water. Other concentrations were prepared from stock solution by dilution varied between 10 and 100 mg/l and the pH of the working solutions was adjusted to desired values with 0.1 m HNO3 or 0.1 m NaOH using a pH meter (Labtronic digital pH meter Model No. LT-10). Fresh dilutions were used for each experiment. 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 stedim arium Water Purifier Unit.

2.2 Ultrasound assisted chemical activation

The peanut husk adsorbent is treated with H,PO, (85% by weight) for chemical activation in the presence of ultrasound. The ultrasound assisted chemical activation was carried out using ultrasound probe sonicator (Dakshin India Ltd., Mumbai) consisting of ultrasound homogenizer operating at 20 kHz with a power rating of 120 W, equipped with a probe with a tip of 18 mm. A 100 ml of glass beaker was used as a reactor and top of the reactor received the ultrasonic transducer and is covered with aluminum foil. The probe tip was dipped in the solution at a length of 0.5 cm. The temperature is maintained at 30°C throughout the experiment using a thermostatic water bath.

The influence of parameters affecting modification of peanut husk and further adsorption of copper (II) such as solute to solvent ratio, concentration of $\rm H_3PO_4$, temperature, duty cycle, sonication time and power was studied. A known amount of adsorbent and phosphoric acid was treated in 100 ml glass beaker covered with aluminum foil. The ultrasound power of 70 W and duty cycle 83% was used for the initial run. The activated samples were washed, filtered and the pH is adjusted to 7 with 0.1 m NaOH and 0.1 m HCl. The adsorbent was dried at 100°C in the oven. The dried adsorbent was kept in a desiccator till it reached to room temperature (30 \pm 2°C). This adsorbent is further used to check copper adsorption.

Initially, the effect of concentration of phosphoric acid was studied by varying the acid concentration from 10% to 80%, and the effect of solute to solvent ratio was studied for different ratios in the range of 1:4 to 1:16. Further, the effect of temperature was carried out at various temperatures ranging from 10°C to 40°C using temperature controlled water bath. The ultrasound parameters like effect of duty cycle, sonication time, duty cycle, probe height dipped in adsorbent-acid slurry and power were also studied.

2.3 Copper adsorption experiment

Effect of activation in the presence of ultrasound on peanut husk was further studied by determination of copper adsorption using method reported earlier [24]. As per the method, 100 ml metal ion solution of 100 mg/l concentration was equilibrated with 0.5 g of the chemically activated adsorbent prepared in presence of ultrasound. Sorption process was carried out at pH 4.5 in a conical flask placed in a thermostatic water bath at fixed temperature till 1 h. One milliliter of the 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 and ultrasound activation on adsorption capacity of the adsorbent. The metal uptake (mg/g) at equilibrium were evaluated from the given equation as

Metal uptake
$$(q_e) = \frac{(C_i - C_e)}{M} * V$$
 (1)

 C_i is the initial metal ion concentration before sorption process in mg/l,

 \mathbf{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 biosorbent taken in g.

3 Results and discussion

3.1 Effect of H_3PO_4 concentration

The concentration of acid plays an important role in altering the surface characteristics of the adsorbent. Hence,

the effect of concentration of phosphoric acid was studied by varying the concentration of acid from 10% to 80% by weight at a constant temperature ($30^{\circ}C \pm 2^{\circ}C$) with 5 g of peanut husk and 20 g of phosphoric acid (impregnation ratio 1:4). The power and frequency of ultrasound were kept constant at 70 W and 20 kHz respectively for 5 min. The acoustic cavitation helps in proper infusion of the acid into the pores of the adsorbent material. Figure 1 depicts the outcome of acid concentration during activation on copper adsorption. It clearly shows that with an increase in acid concentration from 10% to 20% by weight there is an increase in the adsorption capacity of copper. At the acid concentration of 20% during activation, the resulting adsorbent gave significant copper uptake of 10.9 mg/g, but beyond 20% acid concentration, the copper uptake reduced gradually. This indicates that at lower concentration of acid, the surface modification is lower and at higher concentration of acid, leaching occurs and the adsorptive capacity drops. Similar results were observed by Tao and Xiaoquin [25] where lower concentrations of nitric acid had enhanced the surface characteristics of peanut husk for removal of lead. However, this study was reported for conventional chemical activation.

3.2 Effect of acid impregnation ratio

It is an important parameter which decides the amount of acid required for adsorbent modification and thus economy of the process. Acid impregnation ratio is defined as weight of adsorbent to the weight of acid. The effect of acid impregnation on adsorbent modification is

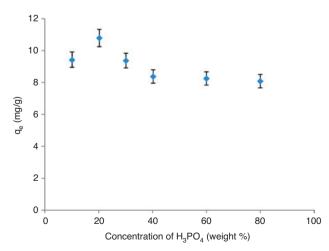


Figure 1: Effect of concentration of phosphoric acid under conditions of 70 W, 20 kHz power impregnation ratio 1:4 and 85% duty cycle at 30°C.

studied at optimum acid concentration of 20% by weight. Figure 2 shows that with an increase in acid impregnation ratio from 1:4 to 1:6, the copper uptake after activation increases and the maximum uptake of 11.5 mg/g was obtained at 1:6 impregnation ratios. The enhanced infusion of the acid into the adsorbent as a result of shockwaves and thermal effect of the ultrasound, leads to increase in porosity. This might lead to development of meso pores and micro pores on the surface of the adsorbent which is predominant at lower value of acid impregnation ratio than at higher values till 1:6. With the further increase in acid impregnation ratio i.e. from 1:8 to 1:12 there is decrease in copper uptake due to the leaching of the surface during activation.

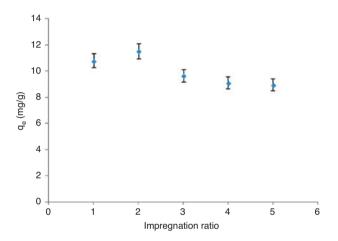


Figure 2: Effect of solute to solvent ratio under conditions of H₂PO₄ concentration 20% 70 W, 20 kHz, impregnation ratio 1:4, 85% duty cycle, 30°C.

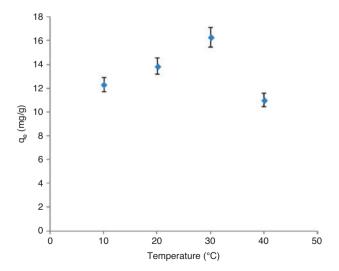


Figure 3: Effect of temperature under conditions of 70 W, 20 kHz power impregnation ratio 1:6 and 80% duty cycle.

3.3 Effect of temperature

During sonication the temperature increases rapidly with time. This function is utilized to investigate the effect of temperature on the degree of modification of the adsorbent by H₂PO₄ using ultrasound. The effect of temperature is studied by varying the temperature from 10°C to 40°C. In the Figure 3 it is seen that as the activation temperature of the adsorbent-acid slurry is increased from 10°C to 30°C, the adsorbent showed an enhancement in copper uptake from 12.33 mg/g to 16.29 mg/g. This was observed due to the synergetic effect of the temperature rise and ultrasound waves which leads to enhanced infusion of the phosphoric acid into the pores of the adsorbent. This results in an increase in the extent of phosphoric acid functionalization on the overall surface of the adsorbent increases. The adsorbent prepared at 40°C showed a reduction in the adsorption capacity which can be again accounted for the synergetic effect of temperature rise and ultrasound shockwaves leading to the degradation of the surface functional groups and thereby reducing the functionalization capacity. The use of ultrasonic irradiations lowers the strong dependency on the operating temperatures allowing possible use of lower operating temperatures which can give safe and economically favorable operation as stated by Chavan et al. [26].

3.4 Effect of duty cycle

The study of duty cycle is important to determine the appropriate ON-OFF timing during the ultra-sonication, as unnecessary increase in the ON timing would lead to excessive heating and unnecessary power consumption [27]. The ultrasonic parameter, duty cycle was varied from 20% to 100% keeping the power at 70 W, time 5 min, frequency at 20 kHz and temperature 30°C. Duty cycle of 80% was found to be more efficient with highest adsorptive capacity of 16.2 mg/g. As shown in Figure 4, an increase in duty cycle increases the acoustic cavitation, thereby favoring the formation of mesopores. However, at 100% duty cycle due to excess cavitation i.e. formation of more bubbles, the heat is entrapped and development of surface is hindered. This is supportive to the fact that longer ultrasound irradiations leads to intense cavitation intensity, due to which the pore structure gets damage thereby reducing the adsorption capacity. Similar results were obtained by Chavan and Gogate [27] for ultrasound assisted epoxidation of sunflower oil where the conversion of oxirane oxygen was increased at low duty cycle and subsequently conversion decreased with increased in duty cycle.

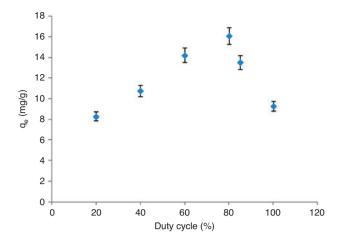


Figure 4: Effect of duty cycle under conditions of 70 W, 20 kHz power impregnation ratio 1:6 at 30°C.

3.5 Effect of sonication time

The sonication time is one of the ultrasound parameters which decides power consumption and thereby the viability of the process. This effect is studied by varying the time from 1 min to 15 min at non uniform intervals i.e. 1 min, 3 min, 5 min, 10 min and 15 min. It was found that the maximum removal of copper i.e. 16.3 mg/g was obtained at 5 min of ultrasound treatment of the adsorbent as shown in Figure 5. Adsorption is higher for lower period of ultrasound application due to the implosions caused at the surface of the material which leads to enhanced surface area resulting in increase in copper uptake. If the activation is performed for less ultrasound time, lower copper intake was obtained as time is not sufficient for the development of the surface area. However, at higher value of time, the micro pore structure gets damaged and thus it

is must to perform chemical activation using ultrasound at optimum time i.e. 5 min.

3.6 Effect of power

The effect of power on adsorption of copper has been investigated at different operating powers such as 25, 50, 75, 90, 100 and 120 W (the range of operating powers is based on the equipment operating limitations). Figure 6 shows that the adsorption of copper increases with an increase in the operating power during activation. This trend is observed till 90 W power input in activation process where the resulting activated adsorbent showed adsorptive capacity of 18.43 mg/g. Further increase in the power input (both the power levels as 100 W and 120 W) during activation ensued reduction in copper uptake. With an increase in power, higher cavitational activity is observed which leads to enhanced modification in the structure of peanut husk by phosphoric acid during the activation process; thereafter increase in adsorption capacity of copper. Above a certain power level there is decrease in the adsorption activity due to cavity cloud formation leading to reduction in infusion of phosphoric acid in the adsorbent and so the adsorptive capacity reduced to 12.3 mg/g at 120 W power [28]. Similar trends of increased activity at optimum value and then further decrease was observed in the literature [29, 30]. It reveals that at higher power conditions, cushioning effect is witnessed which results in decreased transfer of energy into the system, that results in lowering of cavitational activity. The copper uptake obtained was 18.9 mg/g at the optimum power of 90 W. The actual power dissipated in the solution was estimated by calorimetric study, which is based on the measurement of the rate of temperature

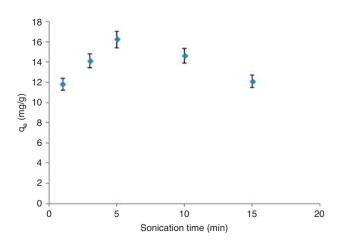


Figure 5: Effect of sonication time under conditions of 70 W, 20 kHz, power impregnation ratio 1:6 at 30°C with duty cycle 80%.

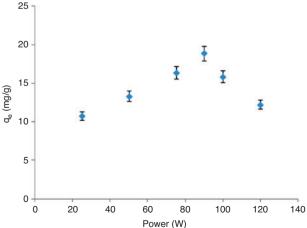


Figure 6: Effect of power under conditions of 20 kHz, power impregnation ratio 1:6 at 30°C with duty cycle 80%.

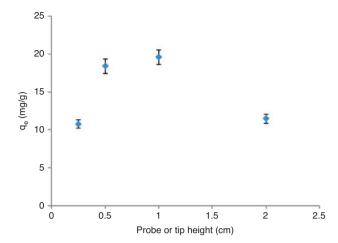


Figure 7: Effect of probe height in adsorbent- acid mix under conditions of 20 kHz and 90 W power impregnation ratio 1:6 at 30°C with duty cycle 80%.

increase in a system. The power dissipated and ultrasonic intensity in ultrasonic horn operated at 20 kHz was found to be 14.93 W and 6.09 W/cm², respectively.

3.7 Effect of probe height dipped in the solution

To study the effect of probe dipped in the solution on activation of peanut husk the tip of probe was dipped in the adsorbent-acid mixture at varied heights i.e. 0.25, 0.5, 1.0 and 2 cm. It is clear from Figure 7 that as the probe length dipped is increased from 0.25 cm to 1 cm the copper uptake on the adsorbent has increased. The ultrasound shockwaves and the thermal effects may not reach till the bottom of the material, due to which the adsorbent surface is not properly modified by phosphoric acid, which ultimately results in a low adsorption capacity at probe length 0.25 cm and 0.5 cm. Further at an optimum probe length there is an enhancement in the adsorption capacity, this may be due to proper distribution of cavitation bubbles and phosphoric acid infusion inside the pores of the adsorbent. The optimum tip length is observed to be 1 cm where copper uptake is maximum i.e. 19.64 mg/g. At the optimum height the bubbles generated by cavitation are properly scattered in the mixture and moreover, due to shock wave the energy distributed by the imploding bubbles are properly dispersed in the mixture. Similar results were observed by Mohod and Gogate [31]. The extent of mixing in the reactor is also dependent on the immersion depth of the horn tip as demonstrated by Nishida [32].

3.8 Comparison of ultrasound assisted chemical activated adsorbent

The adsorption of copper using peanut husk as well as chemically activated peanut husk was also carried out at optimum conditions (data not shown). It is observed that the copper adsorption obtained with native peanut husk and chemically activated peanut husk was found to be 14.3 mg/g and 17.8 mg/g, respectively. The chemical activation was performed in a stirred vessel using H₂PO, at acid Impregnation ratio of 1:8. The ultrasound assisted activation of peanut husk not only gives highest copper adsorptive capacity of 19.64 mg/g but also reduces the time of activation to 5 min from 20 min and acid impregnation ratio of 1:6 from 1:8 of chemical activation. This indicates that the ultrasound has a very significant effect in inducing the changes in the morphology of the adsorbent which leads to functionalization of the inner pores by phosphoric acid.

3.9 Surface morphology of ultrasound assisted peanut husk

A scanning electron microscope was used to look at the effect of ultrasound on the morphology of peanut husk. Figure 8 shows the SEM photographs of the biosorbent

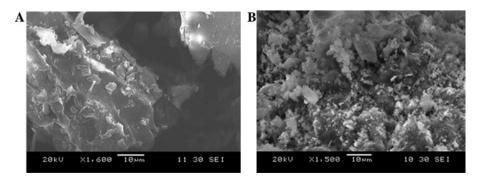
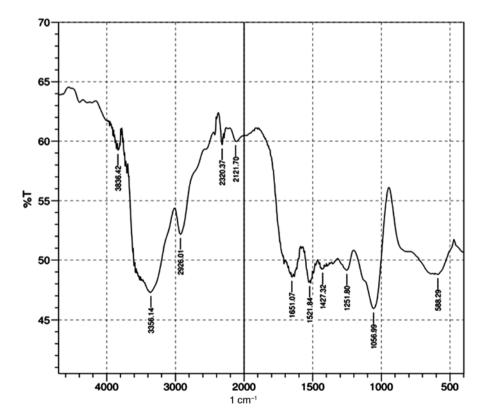


Figure 8: (A, B) SEM micrographs of untreated peanut husk and ultrasound assisted peanut husk.

Figure 9: FTIR of untreated peanut husk.

4000



2000

1 cm⁻¹

3000

1500

1000

500

Figure 10: FTIR of ultrasound assisted chemically treated peanut husk.

before and after ultrasonication. Without ultrasonication, the surface is microscopically smooth. Due to the ultrasound irradiation the surface becomes rough and pitted as it can be seen from the images. These pits might be the result of the shocks and micro jets due to ultrasound waves. In addition, shock waves impinge on the particles, generating local surface pitting and microscopic erosion. On the basis of the surface morphology changes observed, an increase of surface area would be intuitively expected. Increased copper removal would be a consequence of newly formed sorption sites.

3.10 Fourier transform infrared spectroscopy spectral analysis

The FTIR spectrum of Figures 9 and 10 shows a wide and strong -OH stretching band observed between 3300 and 3400 cm⁻¹ which is ascribed to the presence of polyphenols, luteolin, cellulose, and hemicellulose. Also, this broad band related with hydrogen bonding verifies that water is still remaining in the samples. The absorption band at 1710 cm⁻¹ is ascribed to the stretching vibrations of carboxyl groups on the edges of layer planes or to conjugated carbonyl groups (C=0 in carbonyls, luteolin or lactones groups). Stretching absorption band at 1645 cm⁻¹ is assigned to C=C present in olefinic vibrations in aromatic region. Absorption bands at 1514.6, 1429 and 1371.0 cm⁻¹ are assigned as C-H bending vibrations. It can be observed that there are no significant changes in the main peaks of both the cases, which indicate that structure of peanut husk is still stable and not affected by ultrasound.

4 Conclusion

The effects of 20 kHz ultrasound on activation of peanut husk by phosphoric acid and its subsequent effect on the adsorption behavior of copper were studied. Adsorption of copper on peanut husk in the presence of ultrasound is higher than in the absence of ultrasound. The amount of acid requirement is also reduced as compared to conventional method of chemical activation due to proper utilization of acid. The stronger the power intensity of the ultrasonic field, the greater is the adsorption capacity of the adsorbent. However, low frequency ultrasound enhances pore infusion and initial rate of adsorption. The cavitation bubbles, and the associated micro disturbances near the surface of solid, modify the boundary layer and increase the intraparticle infusion. Further the synergetic effect of ultrasound and phosphoric acid concentration

enhanced the adsorption capacity of peanut husk by 10% and 30% as compared with conventional chemical activation and native peanut husk respectively.

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