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Rice straw charcoal: Characterization and adsorption of Pb²⁺ from aqueous solution

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ABSTRACT

Carbonaceous sorbent from rice-staw that can be used as adsorbents for the purification of water were prepared and evaluated. Effects of charring temperature and activation of charring rice straw by acid and alkali on textural and adsorption were investigated. The adsorptive capacities of charring rice straws were measured by Pb ion adsorption. The charring was characterized by electron microscope (SEM) and IR spectrum; round-shaped and porous structures were formed by activation.

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KEYWORDS

Rice straw;
Charring rice straw;
Activation;
Pb adsorption.

INTRODUCTION

Activated carbon has the unique ability to adsorb a wide range of compounds from both the liquid and gas phase. This adsorptive feature is responsible for removing contaminants from water and other substances where the adsorbent material is added.

Commercially activated carbon is produced from bituminous or lignite coal. The long-term availability of coal, environmental impacts and potentially increasing cost has encouraged researchers to find other alternatives, which may be cast effective and equally potential. Activated carbon is a form of carbon that has been processed to make it extremely porous and thus to have a very large surface area available for adsorption or chemical reactions. It can be produced from carbonaceous source materials like nutshells, peat, wood, coir, lignite, coal and petroleum pitch by one of the following processes; (a) Physical reactivation by using one or a combination of Carbonization and *Activation/Oxida-*

tion. (b) Chemical activation; the raw material is impregnated with certain chemicals, and then carbonized at lower temperatures. Activated carbon can be manufactured from any material that has reasonable elemental carbon content. Any lignocellulosic material can be converted to an activated carbon. The literature mentions many precursors for activated carbon such as bagasse^[1-2] scrap tires, saw dust^[3], almond, pecan, English walnut, black walnut, macadamia nut^[4], pistachio^[5] hazelnut shells^[6] (Koby, 2004), rice husk^[7], rice bran^[8] and corn cobs^[9] etc.

In recent years, the use of low-cost waste, especially agricultural by-products as precursor to prepare activated carbons has attracted much attention. In the past 20 years, several researchers began to focus on developing porous activated carbons from rice straw for the removal of pollutants from wastewater^[10].

Ken-Lin et al found that, the availability and suitability for production of carbonaceous adsorbents from rice straw biomass makes it one of the materials that

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can be used for removal of carbofuran from aqueous medium^[11]. Oh and Park adopted a 2-steps activation in which the raw rice straw was first carbonized and subsequently activated with KOH. Porous carbons with high surface area and high adsorption capacity for both methylene blue and iodine were obtained^[12].

In Egypt rice cultivation in the River Nile Delta produces large amounts of rice straw as residue. Assuming that about 20% were used for other purposes about 80% were left on the fields for burning within a period of 30 days, where there is a short duration to prepare the field for the next crop, to get quickly rid of leftover debris. It has been observed that open field burning of crop residues is a process of uncontrolled combustion during which air pollutants are emitted into the atmosphere. And these air pollutants have significant toxicological properties and are notably potential carcinogens. The resulting emissions give a significant contribution to the air pollution called the "Black Cloud"^[13].

Besides activation time, temperature, affects the characteristic of the activated carbon produced. Generally, for commercial activated carbon usually conducted at temperature above 800°C in a mixture of steam and CO₂^[14]. Organic matter may be converted by controlled thermal decomposition into carbon. The mechanisms involved in the conversion of organic matter to carbon are: (1) desorption of adsorbed water up to 150°C, (2) splitting of matter structure and water between 150 and 260°C, (3) chain scissions, or depolymerization, and breaking of CO and C-C bonds within ring units evolving water, CO and CO₂ between 260 and 400°C, (4) aromatization forming graphitic layers above 400°C, and (5) above 800°C, the thermal induced decomposition and the rearrangement reaction are almost terminated leaving a carbon template structure. The major components of organic matter break down in a stepwise manner at 200-260°C (hemicellulose), 260-350°C (cellulose) and 280-500°C (lignin). Between 260°C and 400°C almost 80% of the total weight loss occurs which may vary between 40% (lignin) to about 80% (cellulose) due to evolution of H₂O, CO₂, and volatile hydrocarbon species from fragmentation reactions of the polyaromatic constituents^[15].

Recently, the researchers have been working out on optimizing the final activation temperature to econo-

mize the cost of production. As reported by several authors, activation temperature significantly affects the production yield of activated carbon and also the surface area of activated carbon. The temperature used as low as 200°C and up high to 1100°C^[16].

The optimum temperatures have been reported to be between 400°C to 500°C by most the earlier researchers irrespective of the time of activation and impregnation ratio for different raw material^[17].

The importance of this work lies in the urgent need to utilize rice straw which creates a common problem known as *Black Cloud* by producing new carbonaceous sorbent from rice-straw. In addition, sorbent adsorption capacities and surface properties as an adsorbent for the removal of heavy metals from water will be evaluated.

EXPERIMENTAL

Raw material

The straw from the Nile valley, harvested in October was used for this study. The straw (leaves and stems) were collected after and the seeds were separated. They were washed and oven dried at 110°C overnight before using.

Methods

Charring

A weighed amount of rice straw charged into the furnace at a temperature of 300, 400, 500 and 600°C for 0.5, 1.0, 1.5 and 2 h. The resulting charred material was collected and cooled at room temperature. The domain of variation of these factors is defined according to Bornemann *et al.*,^[18].

A known amount of rice straw charcoal (10 g) was transferred in a beaker (250 mL) and added 100 mL distilled water to it and continued adding water to it up to 200 mL to completely soak the charring rice straw in beaker. When the charring rice straw completely settled down then decanted the distilled water and repeated this process for several times until the decanted water become cleared. Rice straw charcoal was then filtered through ordinary filter paper and washed again with distilled water. Cleaned rice straw charcoal was dried in an oven to get a constant weight and stored in an air

tight bottle for further use.

Charring yield

The resulting cooled charred materials obtained from the above process were weighed to determine the yield of each material at different charring temperatures^[19]. The percentage yield for each charred material was determined using the following equation (1):

$$\text{Percentage Yield (\%)} = \left[\frac{W_F}{W_I} \right] \times 100 \quad (1)$$

Where W_I = Initial weight before charring and W_F = Final weight after charring

Chemical treatment of rice straw charcoal

A known amount (10g) of uniform particle sized rice straw charcoal was transferred in a two separate beakers and added to each beaker 200 mL of 1.0 M NaOH, or HCl. The mixtures were kept overnight. The superficial solutions were then decanted and added to it 200 mL of distilled water. The distilled water was again decanted and the process was repeated until the decanted water becomes cleared. Rice straw charcoal was then filtered through ordinary filter paper and washed again with distilled water. Cleaned and rice straw charcoal was dried in an oven till constant weight and stored in an air tight bottle for further use.

The samples were later soaked in 2% (w/v) NaHCO_3 or 10% acetic acid to remove any residual acid and alkali left respectively. The resulting mixture were further washed with distilled water to bring the pH to 7.0 and finally drained and dried overnight in an oven at 110°C ^[20]. They were later cooled at room temperature and weighed to determine the amount of the material washed-off in the process. The percentage washed-off for each material was determined from the relation (2).

$$\text{Percentage washed-off (\%)} = \left[\frac{\omega_I - \omega_F}{\omega_I} \right] \times 100 \quad (2)$$

Where ω_I = Initial weight before activation and ω_F = Final weight after activation

Characterization of sorbents

Rice straw charcoal was characterized before and

after chemical treatments. Various techniques like FTIR, elemental analysis, and SEM were used to ascertain the presence of various functional groups and morphology studies.

Elemental analysis

Determination of C, H, N, and O (by difference) was performed with a GmBH Elemental Analyzer System of from ELEMENTAR Analyses system, Germany.

Characterization using FTIR

FTIR spectra of the sorbents were obtained by using NICOLET 380-FTIR, Thermoscientific, UK, Fourier Transform Infrared Spectrophotometer along with OMNIC XP software and computer control disc (ZnSe) unit. The FTIR spectra of the samples were recorded in the region of $400\text{-}4000\text{ cm}^{-1}$.

Characterization by scanning electron microscopy (SEM)

The morphologies of the sorbent particles were analyzed using scanning electron microscope FEI INSPECTS Company, Philips, Holland, environmental scanning without coating.

Bulk density determination

Bulk density of the resulting materials were determined using the method of Ahmedna *et al.*,^[21] which consist of placing known weight of granulated charring rice straw in a 25 ml cylinder to a specified volume and tapping the cylinder for at least 1 – 2 min and measuring the volume of charring rice straw. The charred materials in this study were crushed to granules using mortar and pestle, thereafter screened with 10 BSS mesh size (over screen) and 120 BSS (under screen). The bulk density was measured as following:

$$\text{Bulk density (g/cm}^3\text{)} = \frac{W_s}{V_s} \quad (3)$$

Where W_s is the weight of the screened sample (g) and V_s is the volume of packed dry sample (cm^3).

Adsorbate

All the glassware used for this study were washed with detergent, rinsed with distilled water and allowed to dry in the oven before use. Stock solution of Pb (II) was prepared (100 ppm) by dissolving required amount

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of, PbCl₂ in acidified double distilled water. The mixture was carefully swirled together for 10 min to allow proper dissolution. Then, distilled water was added to the mixture, properly shaken for thorough dissolution of the adsorbents; then made up to 1 L mark with the distilled water^[22]. All the chemicals used were of analytical reagent grade.

Determination of adsorption capacity

A 30 mL of the Pb (II) solution was added to weighed amount (0.05g) of each of the rice straw charcoal in 250mL conical flask. The mixture in the flask was covered and placed on magnetic stirrer at 150 rpm for 2 h^[23] at an ambient temperature (28±2 °C) and pH 7.5^[24]. After which the content was allowed to stand for 1 h and the supernatant solution was filtered with Whatman filter paper into sample bottles. This procedure was repeated for all rice straw charcoal samples produced at various burning temperatures (300- 600 °C) and for different times (0.5- 2 h). The resulting filtrates were subjected to analytical analysis. Analytical method

Lead concentration was analyzed using atomic absorption spectrophotometer using ICP-AES Jobin Yvon J4 Spectrometer.

From Pb (II) concentration measured before and after adsorption (C_i and C_e , respectively), dry weight of the adsorbent (W) and the volume of aqueous solution (V in L), the amount of equilibrium adsorption of metals (Q_e) was calculated using the Eq. (4):

$$Q_e \text{ (mmol/g)} = (C_i - C_e) V / w \quad (4)$$

RESULTS AND DISCUSSION

Optimal conditions on the preparation of charring rice straw

The influence of the carbonization temperature and time was shown in Figure 1. The increasing of activation temperature reduces the yield of the charring rice straw continuously. This is expected since an increasing amount of volatiles is released at increasing temperature^[25]. These phenomena are also manifested in the decreasing volatile content and increasing fixed charring rice straw for increasing activation temperature.

Besides activation temperature, the activation time also affects the charring process and properties of char-

ring rice straw. As the time increased, the percentage of yield decreased gradually. This result is possibly due to the volatilization of organic materials from raw material, which results in formation of charring rice straw. The extent of decrease in product yield is observed when excessive activation occurs^[26].

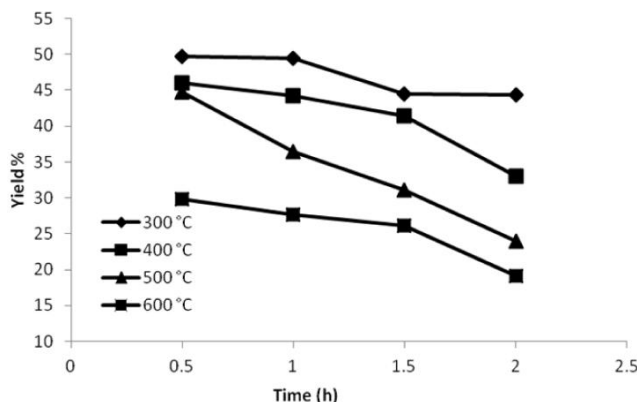


Figure 1 : Effect of carbonization temperature and time on yield%

Effect of charring temperature and time on the bulk density

Figure 2. shows the bulk density of the rice straw charcoal produced at different temperature namely 300, 400, 500, and 600 °C. The bulk density obtained after charring did not show direct relationship to the increasing charring temperature, while it decreases with increasing charring time. It was observed in this study that changes in charring temperature has no significant effect on the bulk density of charring obtained from rice straw.

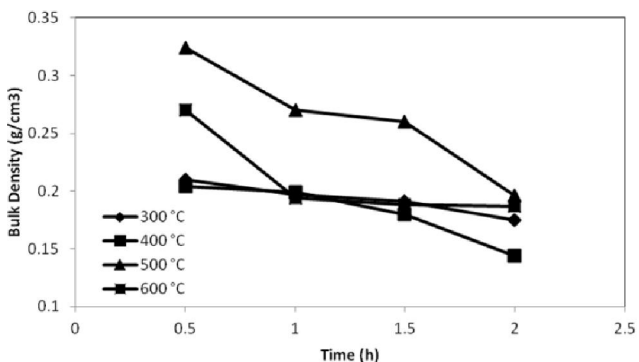


Figure 2 : Effect of carbonization temperature and time on the bulk density

Effect of activation on yield

Figure 3. shows the percentage wash-off of rice straw charcoal obtained at different temperature and

time after acid and alkali activation using HCl and NaOH. The percentage wash-off decreased as the temperature increased and increased as time increased. The overall results showed that the activation process has effect on the yield of charring obtained from rice straw. More biomass is oxidized might have been produced at higher temperature, given the reduction trend when the charcoal were activated with HCl and NaOH.

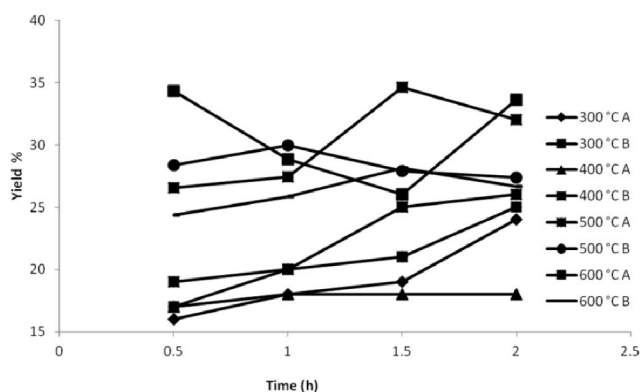


Figure 3 : Effect of activation on yield at different carbonization temperature and time (A= HCl treatment, B=NaOH treatment)

Elemental analysis

The elemental analysis of different selected charring rice straw samples which produced by charring at 500°C and acid or base activated was carried out on a Perkin-Elmer elemental analyzer. These samples were analyzed for their carbon, hydrogen, nitrogen and ash content. The amount of oxygen content in these samples as was calculated by difference. The elemental analysis of selected samples of charring rice straw has been depicted in TABLE 1. It is clear that the carbon content of charcoal was significantly higher after acidic or basic activation than untreated one and the oxygen and hydrogen were lower.

TABLE 1 : Elemental analysis of charring rice straw at 500°C for 1.5h and acid and base activated

Sample	C %	H %	N %	O %	Ash %
Untreated	42.91	2.11	1.87	53.11	41.06
Acid Treated	46.78	2.18	2.13	48.91	36.71
Base Treated	50.58	2.29	2.21	45.03	32.46

Ftir spectra of rice straw charcoal

Infrared spectroscopy in its various forms is an important and forceful technique, giving useful information about structures such as surface functional groups.

FTIR offers several advantages over conventional dispersive methods. The use of an interferometer in place of systems of grating and slits, results in a higher energy throughout the detector. Coupled with internally calibrated computer systems, to add a large number of interferograms, this produces markedly superior spectra, which can give more precise information concerning the oxidation of carbons and the formation of surface oxygen structures. FTIR can also allow measurements of lower concentrations of surface functional groups.

FTIR spectra of untreated, acid treatment and basic treatment rice straw charcoal at 500°C for 1.5h have been given in Figure 4. From the Fig., it is clear that, the intensity of -OH groups have a higher value in case of treated charcoal with HCl and NaOH than untreated one. This is due to that, the treatment with sodium hydroxide and hydrochloric acid decreases the hydrogen bonds between -OH groups. The crystallinity index (Band intensity at 1440 cm⁻¹ / band intensity at 900 cm⁻¹) are 0.883, 0.947 and 0.921 for untreated, acid treated and alkali treated rice straw charcoal, respectively. The crystallinity index of acid-treated charcoal is higher than that of alkali-treated charcoal and untreated one. This is attributed to the degradation of amorphous regions in remained cellulose chains during treatment process. The FTIR of untreated and treated charcoal shows that, the absorption at 1725 cm⁻¹, the specific peak for the carboxylic acid group is observed for the untreated and treated charcoal but the intensity of the peak is increased in acid treatment than in alkali treatment. This phenomenon indicates that acidic groups such as carboxylic groups are additionally formed by acid treatment, but decreased or eliminated by treatment. This trend supports the fact that acidic groups created from the oxidation dominate the surface charge of the charcoal^[27].

As shown from the Figure an increase in the intensity of the band at 1600 cm⁻¹ in treated charcoal than untreated one which is attributed to quinone structure. Also, the intensity of the band at 1098 cm⁻¹ which represents CO- in phenoxy structure was increased in acid treated than alkali treated than untreated rice straw charcoal. Compared with the overall spectra, it is certain that the absorption bands at 1720- 1098 cm⁻¹ are changed after chemical modification of the charcoal. In

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addition, the chemical nature of surface functionality is significantly altered by acid and base treatments.

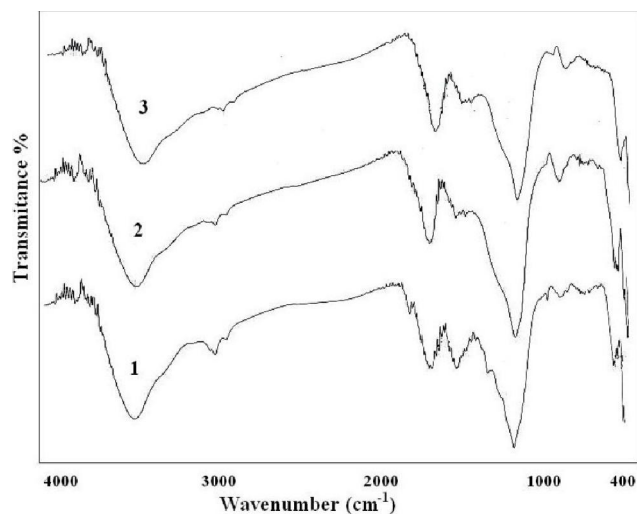


Figure 4 : FTIR spectra of (1) Untreated, (2) acid treatment, and (3) basic treatment charring rice straw at 500°C for 1.5h

Scanning electron microscopy

The SEM enables the direct observation of the changes in the surface microstructures of the carbons due to the modifications. Studies are available which have reported the utilization of the scanning electron microscopy analysis for showing the surface modification changes in the developed adsorbent. Rice straw charcoal is characterized by their strong adsorption capacity, which occurs mostly in cavities of molecular dimensions called micropores. Depending on their preparation, rice straw charcoal also contains larger pores, known as mesopores and macropores in the classification proposed by Dubinin and now adopted by IUPAC^[28]. Figure 5. shows that, there are differences could be seen between untreated and treated charcoal. In addition, there are differences to be seen between two types of activation carried out on rice straw charcoal, whereas, acid treated charcoal appears more homogeneous than alkali treated and untreated charcoal.

Lead ion adsorption

The samples of powdered selected as an outcome of this study, were evaluated for their efficiency towards removal of Pb. This metal was chosen for this study, as this are the critical and strategic metal.

Adsorption experiments were carried out for samples obtained at different burning temperature (300

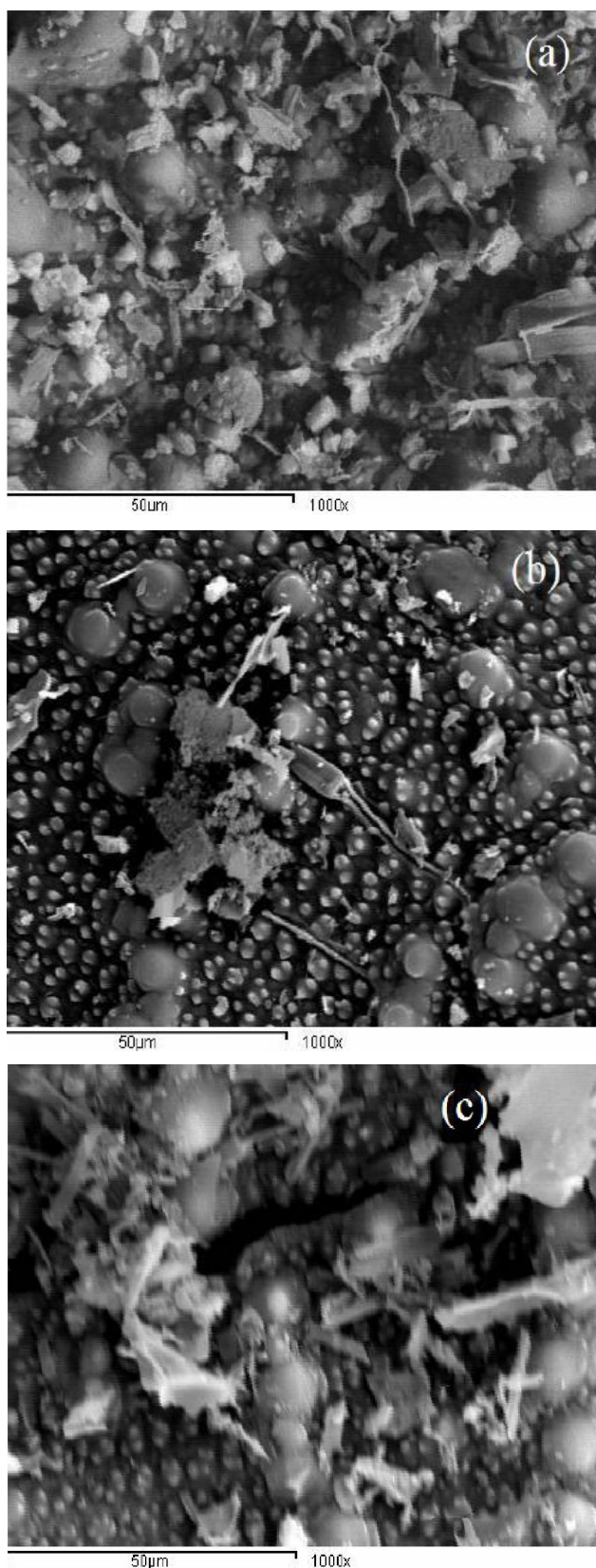


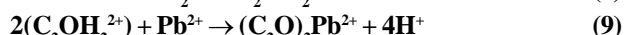
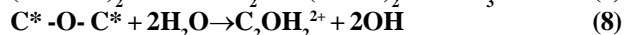
Figure 5 : Scanning electronic micrograph for charring rice straw carbonized at 500°C for 1.5h; Untreated, Acid treatment, Basic treatment

– 600 C°) and for different burning time (0.5- 2 hours) for untreated, acid and alkali treated rice straw charcoal with a fixed lead nitrate solution. As shown from Figure 6. a,b,c,d an increase in removal efficiency with increasing burning temperature from (300 – 600 C°) in untreated and treated charcoal was observed. Also, after one hour and half from burning of rice straw, maximum removal of Pb(II) ion takes place in untreated and treated rice straw charcoal. This is related to by increasing burning time and temperature leads to an increase in active sites in the samples consequently, the removal efficiency of lead ions increases.

Due to the acidic treatment of charcoal, it becomes protonated, having a positive charge on its surface, which provides effective sites for lead adsorption. Figure 6 also explains that acid treated charcoal becomes more effective in adsorption for the above reason. The decrease in sorption capacity in base treated charcoal is

attributed to the accumulation of a negative charge at the adsorbent surface, providing relatively fewer effective sites for lead adsorption.

The mechanism of adsorption of Pb(II) may be explained based on the Bronsted-Lowry acid-base model^[29] and ion exchange model^[30,31]. For acid-activated rice straw charcoal, some carbon-oxygen complexes (-COOH, C* -OH and C* -O- C*) are formed. And these surface oxygen- contained complexes hydrolytic water molecules may ion exchange with Pb(II). The reactions mechanism may be shown as follows:



The reactions proposed above present differences in adsorption capability and indicate a variety in mechanism of interaction between carbon surface and ionic

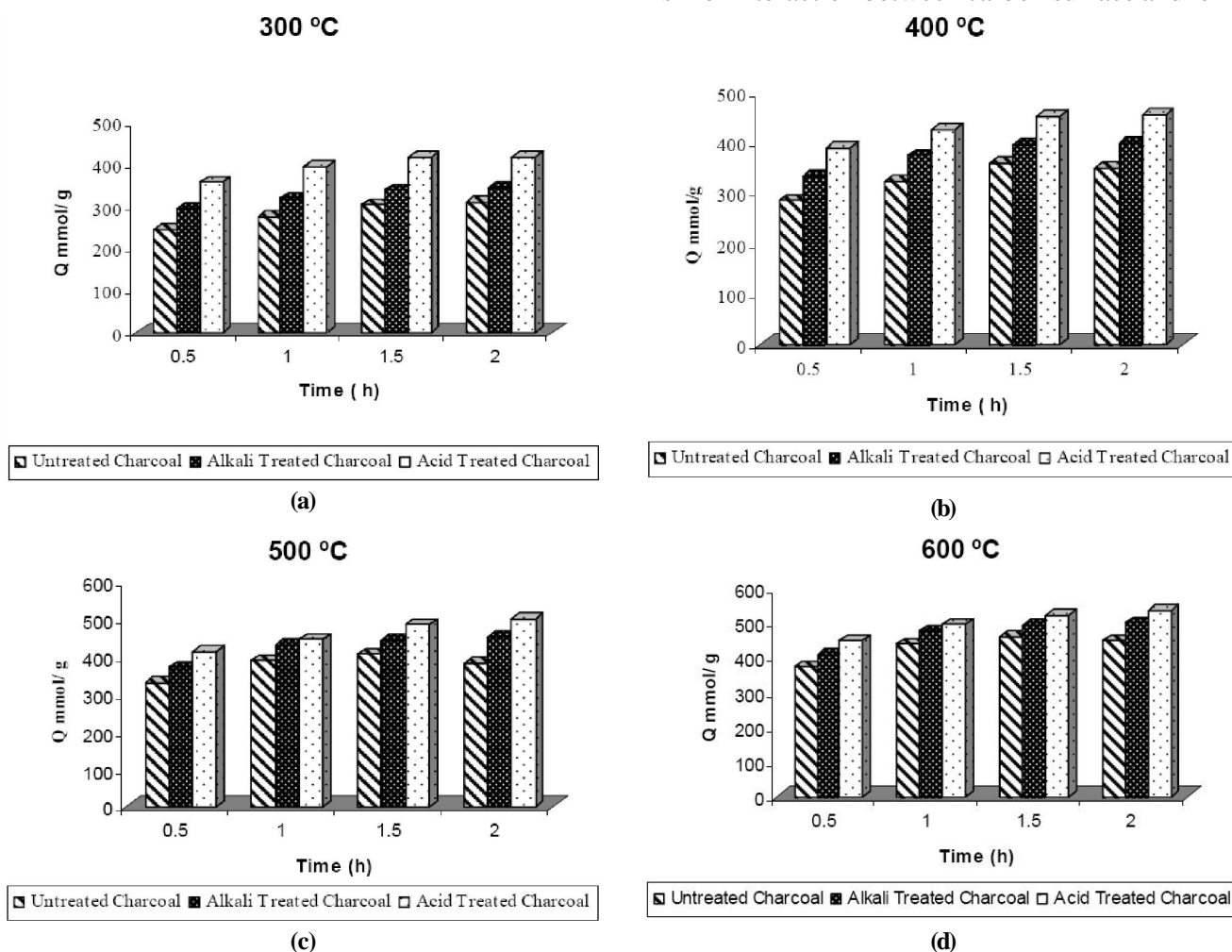


Figure 6 : Adsorption of Pb(II) unto untreated and treated rice straw charcoal at (a)300 °C(b)400 °C (c) 500 °C (d) 600 °C

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metal species present in aqueous solution.

CONCLUSION

Activated carbon is sensitive toward modification by 1 M hydrochloric acid or sodium hydroxide. Acid treatment gives rise to a large increase in the amount of total acidity resulting from the increase of surface oxide groups such as carboxyl, lactone and phenol. The increased surface oxide groups lead to an increase in the intensity of 1725–1400 cm⁻¹ absorption peaks in the FTIR spectra. In the case of rice straw charcoal modified with 1 M sodium hydroxide, the total activity was almost comparable to untreated rice straw charcoal caused by an increase of lactone groups besides of the decrease of carboxyl groups. Rice straw charcoal is an effective adsorbent for the removal of Pb(II) from aqueous solution. It could be employed for the economic treatment of wastewater containing the heavy metal studied, as this adsorbent was derived from an agricultural waste by-product and had a high adsorption capacity. Removal of Pb(II) from aqueous solution was considerably enhanced by acid treatment, which may be related to pores widening, increased cation–exchange capacity by oxygen groups, and the promoted hydrophilicity of the carbon surface.

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