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Surface modification and characterization of a RS activated carbon: density, yield, XRD, ash, and moisture content

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ABSTRACT

Rice straw (RS) is considered to be agricultural waste in developed countries. Since the collection and disposal of this residue is becoming more difficult and expensive, it is left unused as waste material or simply burned in the fields, thereby, creating significant environmental problems like poor air quality that has serious consequences for the population. Converting this renewable biomass to produce activated carbons would have both economic and health benefits for rural communities. Communities could gain income from the sale of the RS to companies or institutions processing them to carbon. In addition, wastewater treatment from organic and inorganic pollutants using the RS based carbon could be developed. Otherwise, this reduces the pollution resulting from RS burning. In this concern, single-step steam activation was performed at temperature ranges 550–750°C. The activated RS was subjected to liquid-phase oxidation by different modifying agents include KOH, HNO_3, H_2SO_4, H_2O_2, and $KMnO_4$ to obtain carbon with various surface characters. We studied the physicochemical properties of the 18-activated carbons derived from RS and prepared by one-step steam pyrolysis. Most of adsorbents exhibited bulk density greater than 0.25 g/ml (American Water Works Association lower limit of AC for practical use). Treatment of carbons with oxidizing agents leads to an increase of ash content except KOH; this may be due to silica which is dissolved easily in KOH. X-ray analysis of activated RS results *showed* a reduction of the amorphous structure.

Keywords: Activated carbon; Rice straw; Density; Yield; XRD; Ash and moisture content

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1. Introduction

The need for purification of potable and wastewaters from various pollutants requires the development of new methods for the production of highly efficient adsorbents. In this respect, carbon adsorbents occupy a prominent position among current adsorbents as versatile and universal materials, and they attracted special attention on the account of their well-developed porous structure, large active surface area, and good mechanical properties [1].

Although coal was more widely used as activated carbon source, agricultural wastes may be a better choice in some places because of its availability and cheapness. The conversion of agricultural by-products to activated carbon, represents a possible outlet. This

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would result in the creation of a versatile value-added product.

The annual harvest and processing of various agriculture crops grown in Egypt yields considerable quantities of agricultural by-products, about 25 million tons per year of agro-residues are estimated, of which 12 million tons are used in energy production or for industrial purposes. Another, 3 million tons are used to produce biogas. The remaining is burned directly. The waste products, which are the main contributors to biomass burning, are wheat residue and rice straw (RS). RS is a lignocellulosic agricultural by-product containing cellulose (37.4%), hemi-cellulose (44.9%), lignin (4.9%), and silicon ash (13.1%) [2]. The disposal of RS by open-field burning frequently causes serious air pollution, hence, new economical technologies for RS disposal and utilization must be developed.

The conversion of RS to activated carbon give good outlet of this agro residue, but unfortunately, this straw-type cellulosic precursor characterizes by much softer and less dense than wood- or fruit stone-type cellulosic precursor. In fact, although Patrick and Streat [3] have recently prepared some active carbons with no high surface area from wheat straw, it was generally considered difficult to make high-performance porous carbons from straw-type precursors due to the softness and relatively high content of the inorganic components. To produce activated carbon from such straw-type precursor, it must be mixed with suitable binder and compressed into briquettes or pellets to increase the density of the final product [4].

Limited knowledge is available for production of activated carbon from RS especially without using binder. Marshall and Champagne [5] evaluated by-products of soybean and cottonseed hulls, RS, and sugarcane bagasse as metal ion adsorbents in aqueous solutions. Ahmedna et al. (6) and Johns et al. [4] have evaluated RS-based GACs for their ability to decolorize sugar or remove metals and low molecular weight organic compounds from solution. Pendyal et al. [6] studied the effect of different binders on physical and chemical properties of activated carbon prepared from low-density precursors such as RS, rice hulls, and sugarcane bagasse and evaluated their ability to remove sugar colorants. In recent studies by Yun et al. [7], investigated pre-carbonization effect on the porosity of RS based carbons prepared by one-stage and two-stage process. Also, Oh and Park [8] prepared a high adsorption capacity active carbon from RS using KOH as activating agent.

In the present study, we prepare active carbon from RS by one-stage steam pyroysis, then surface modification using different oxidizing agents. We characterized their physical properties using density, yield, XRD (X-ray diffraction), ash, and moisture Content.

2. Experimental

2.1. Preparation of active carbon

A laboratory scale fluidized bed reactor was designed and fabricated for the preparation of active carbon. The fluidized bed reactor consists of two staleness steel cylindrical tubes. The inner tube is of 20 cm inner diameter, 3 mm wall thickness, and 50 cm length. The tube is fitted with 1 mm-perforated stainless steel gas distributer. A fine mesh of 0.1 mm is fixed over the distributor to arrest the flow of fine particles through the perforations. Two electric heaters in the top and bottom of the tube used to heat it and the bed temperature is measured by a thermocouple. The bed can be operated between ambient temperature and 900˚C and the temperature is controlled by means of temperature controller. The fluidized gas is admitted into the reactor through flow meter depending on the range employed. The outer cylinder tube is cone shaped at the bottom with a screw holder that makes it easier to clean from tar. The samples were fed to the reactor via a screw feeder to the top of the fluidized bed. The water is pumped to the steam generator by a peristaltic pump. The steam temperature is around 105˚C.

The fluidized bed reactor was heated until the bed temperature reach 150˚C. One half kilogram dried RS was fed into the reactor and subject to heating rate of 50˚C/10 min under inert environment of nitrogen. The flow rate of nitrogen used was sufficient to provide good mixing of the RS within the reactor (300 ml/min). When the temperature of the furnace reach 350˚C, steam produced by a steam generator, entered the reactor at a rate of approximately 5 cm^3/min and furnace heating was continue up to final temperature of 550, 650, or 750˚C, holds at this temperature for one hour. Then, the furnace is switched off and the samples are left to cool down and the steam flow is interrupted at temperature 250–300˚C, then the sample was withdrawn from the reactor and weighted. The samples were taken the abbreviations, RS_1, RS_2, and RS_3 for carbons activated at 550, 650, and 750˚C, respectively.

2.2. Oxidative modification of activated carbon (RS_1, RS_2, and RS_3)

The obtained one-step steam activated RS was subjected to liquid-phase oxidation using different oxidizing agents in order to obtain materials with various surface characters.

2.3. Potassium hydroxide treatment (RS/KOH)

The activated RS (RS_1, RS_2, and RS_3) were treated with 1 M KOH. The oxidation was carried out at the boiling temperature for 2 h. The oxidized activated carbons were washed and then dried over night at 50°C [9]. The carbon treated in this way is denoted as RS_i/KOH where $i = 1$ or 2 or 3 according to the activation temperature 550, 650, and 750°C, respectively.

2.4. Nitric acid treatment (RS/HNO_3)

Analar-grade 65% nitric acid was used to oxidize the carbons under severe conditions. The oxidation process was carried out by adding 50 cm^3 of the acid to 5 g of the carbon placed in a conical flask. The mixture was heated at 60°C on a hot plate with constant stirring until brown gas stopped to evolve (about 3 h), then washed with deionized water, decanted, and the samples were dried overnight in an oven at 110°C and stored in a desiccator for latter use [10]. The carbon treated in this way is denoted as RS_i/HNO_3.

2.5. Sulfuric acid treatment (RS/H_2SO_4)

The samples were treated with 2% H_2SO_4 (v/v) at 150°C for 24 h. The heated material was washed with deionized water until the solution pH was stable. Afterward, the material soaked in 1% sodium bicarbonate solution overnight to remove residual acid. Finally, the samples were dried overnight in an oven at 110°C, cooled at room temperature, and stored in desiccators [11]. The carbon treated by sulfuric acid is labeled as RS_i/H_2SO_4.

2.6. Hydrogen peroxide treatment (RS/H_2O_2)

RS was immersed in 30% H_2O_2 with a ratio of H_2O_2 to RS of 10 ml/g at room temperature until complete degradation of the H_2O_2 (when there was no further gas evolution). Following oxidation, the samples were then washed by hot deionized water, separated by filtration, and dried at 110°C overnight [12]. The carbon treated in this way is refereed as RS_i/H_2O_2.

2.7. Potassium permanganate treatment ($RS/KMnO_4$)

In this concern, 1 g of RS activated carbon (RS_1, RS_2, and RS_3) was subjected to oxidation treatment by 50 ml 0.1 N $KMnO_4$ solution at 50°C for 48 h. The treated carbons were thoroughly washed with distilled water, dried at 120°C, and stored in stoppered bottles [13]. This carbon is denoted as $RS_i/KMnO_4$.

2.8. Characterization of the prepared carbon

2.8.1. Apparent density

The apparent density is equal to the mass (weight) of a quantity of carbon divided by the volume it occupies (including pore volume and interparticle voids). This can be measured simply by measuring the volume occupied by a given weight with the aid of a graduated cylinder.

2.8.2. Bulk density (packed density)

Bulk density was determined by a tamping procedure described by Ahmedna et al. [14]. A weighed amount of 12–40 mesh granular carbon was added to a 10 ml graduated cylinder. The cylinder was tapped constantly until there was no volume change and the bulk density was calculated as [14]:

$$\text{Bulk density (g/ml)} = \left[\frac{\text{wt. of dry material (g)}}{\text{Vol. of packed dry material (ml)}}\right] \tag{1}$$

2.8.3. Ash content

The gravimetric determination of ash contents is performed according to British Standard BS 1016 and is summarized in the following section.

A weighed quantity of carbon (1.0 g) was placed in a porcelain crucible and set in a circulating air oven at 115°C for 4 h. The samples were then weighed and placed in a muffle furnace, and heated at 800°C for 1.5 h. The crucibles were removed from the muffle furnace and allowed to cool in desiccators and reweighed. Percent ash was calculated by [15]:

$$\%\,\text{Ash} = \left[\frac{\text{remaining solid wt (g)}}{\text{orginal carbon wt (g)}} \times 100\right] \tag{2}$$

2.8.4. Moisture content

The moisture content of activated carbon is often required to define and express its properties in relation to the net weight of carbon. The dry weight of an adsorbent is normally measured at a standard temperature of 105°C under an inert atmosphere, for example nitrogen. This method avoids oxidation of carbonaceous adsorbents. The sample should be dried in this manner for 24 h and stored in desiccators [15]. The moisture content was calculated by

$$(\%)\,\text{Moisture} = \left[\frac{\text{weight after heating (g)}}{\text{orginal carbon weight (g)}} \times 100\right] \tag{3}$$

2.9. Burn-off and carbon yield

Burn-off of the carbon material is taken as a measure of the degree of activation. Both burn-off and activated carbon yield were estimated which give a measure of process efficiency. The burn-off is the weight loss of material, determined on a dry weight basis that occurs during the activation process. Yield is the amount of original precursor remaining after activation [16].

$$\text{Yield} = \left[\frac{\text{Original wt. (g)}}{\text{Final wt. (g)}}\right] \times 100 \tag{4}$$

$$\text{Burn} - \text{off} = \left[\frac{\text{Original wt. (g)} - \text{Final wt. (g)}}{\text{Original wt. (g)}}\right] \times 100 \tag{5}$$

2.9.1. X-ray analysis

The X-ray powder diffraction patterns of the carbon samples were recorded on film at room temperature in a Philips XRG3100 X-ray diffractometer using Cu-Kα X-ray operated at 30 kV and 30 mA with a fixed slit. XRD spectra was used to analyze the (002) and (100) peaks of various carbon samples. Activated carbons in general show a 002 diffraction maxima at $2\theta \sim 25^\circ$ and overlapped 100 and 101 maxima (usually identified as the 10 peak) at $2\theta \sim 42^\circ$ [17]. The peak centered around 25° corresponding to the 002 set of planes, was used to measure the interlayer spacing [18].

3. Results and discussion

The physical properties of the various 18-prepared carbon samples under investigation were studied in term of density, ash, and moisture content. In addition, their structure and yield were also characterized.

3.1. Apparent and bulk density

Density is a prosaic property of all matter. It reveals much about the composition of the matter [19]. There are two types of density associated with activated carbon called apparent and bulk (packed) density.

The apparent density is equal to the mass (weight) of a quantity of carbon divided by the volume it occupies (including pore volume and interparticle voids) while, bulk density exclude these pore volume and interparticle voids.

Starting material with high density is desirable for the preparation of granular activated carbons. However, the density of the final products after activation depends not only on the nature of the starting material, but also on the activation process [20]. The experimental results of the apparent and bulk density of activated and oxidized carbons based RS are presented in Table 1. It is clear that, as the temperature of activation of RS increased from 550 (RS_1), 650 (RS_2), to 750°C (RS_3) apparent density decreased. This change in densities is related to the reaction rate between carbon and steam. This essentially depends on the reaction temperature. Increasing temperature of activation process, causes a successive release of volatiles, gases, and oils or tars. Therefore, the steam penetrates easily into the surface of RS and subsequently causes development of its porosity [21].

In addition, the measurements of apparent and bulk density of activated carbon can provide information regarding to porosity and pore volume. As we see from Table 1, the oxidation was certainly effective in increasing the density; almost all of modified activated RS show higher density (apparent and packed) than that of activated RSs. This is an indicative of the deposition and retention of mixed oxide structure within the pores of the carbon as a result of treatment of activated carbon [22]. It can be seen that RS_1/KOH has high total porosity (50%) opposite to RS_3/H_2SO_4 that have lower total porosity (16.6%). This is due to, the former has great difference between apparent and bulk density while the later has small difference. RS_1/KOH and RS_3/H_2SO_4 have higher values of bulk density. The high value of apparent and bulk densities of RS_3/H_2SO_4 may be related to the presence of residual sulfur [23].

The calculation of pore volume through the apparent and bulk densities indicate that potassium hydroxide treatment of activated carbons prepared at 550 and 650°C had the largest pore volume with porosity of 50 and 33%, respectively. When the activation temperature increased to 750°C hydrogen peroxide treatment gives higher pore volume with porosity of 41.7%. These results are similar to that obtained from nitrogen adsorption results and cited later.

In practical terms, GACs with high bulk densities are preferred for general use. The American Water Works Association (AWWA) has set a lower limit on the bulk density of 0.25 g/ml for GACs to be of practical use [24]. Only three of the 18-prepared carbon samples would fall below this guideline. The high bulk density obtained can be explained by high lignin contents of RS.

Table 1
Density, total porosity, and pore volume of the various prepared carbons

Adsorbent (g/cm^3)	Apparent density (g/cm^3)	Bulk density (g/cm^3)	Total porosity (%)	Pore volume (cc/g)
RS_1	0.27	0.36	25	0.91
RS_1/KOH	0.38	0.75	50	1.32
RS_1/HNO3	0.39	0.45	12.5	0.31
RS_1/H_2SO_4	0.29	0.36	20	0.69
RS_1/H_2O_2	0.19	0.23	16.6	0.84
$RS_1/KMnO_4$	0.39	0.56	30	0.76
RS_2	0.23	0.32	28.5	1.24
RS_2/KOH	0.13	0.19	33.3	2.59
RS_2/HNO_3	0.29	0.34	16.6	0.57
RS_2/H_2SO_4	0.24	0.35	33.3	1.41
RS_2/H_2O_2	0.27	0.36	25	0.91
$RS_2/KMnO_4$	0.36	0.45	20	0.55
RS_3	0.21	0.28	27.7	1.35
RS_3/KOH	0.17	0.2	14.2	0.83
RS_3/HNO_3	0.42	0.51	16.6	0.39
RS_3/H_2SO_4	0.92	1.53	16.6	0.43
RS_3/H_2O_2	0.25	0.42	41.6	1.69
$RS_3/KMnO_4$	0.25	0.36	30	1.17

3.2. *Ash content*

The ash content of a carbon is the residue that remains when the carbonaceous material is burned off. The ash content of the solid products depends on the chemical composition of the raw materials (mainly inorganic compounds) and the degree of carbonization. The removal of volatile organic compounds in the thermal treatment results in enrichment of the inorganic material in the solid residues, which undergoes deep carbonization [25].

Natural RS has ash content of 15% [7]. The high ash contents of activated carbon made from RS can be explained by their high-specific mineral content, especially their richness in silica [5,26]. The values of measured ash content are given in Table 2.

Activated RS carbon has high ash content compared to the starting natural RS. This indicates that during the process of activation, a substantial amount of carbon was consumed (i.e. high carbonization). The ash content dropped somewhat with increasing of activation temperature from 550, 650, to 750°C. This suggests that increasing activation temperature can affect not only the carbonaceous part, but also the inorganics (insoluble silicate) in the samples [27]. The gases evolved during activation may remove the smaller inorganic clusters, and the bigger one may undergo decomposition during the extend exposure to high temperature and steam during activation.

It is clear that, treatment of activated RS with oxidizing agents leads to changing of ash contents; KOH treatment decreases the ash content. This may be due to silica, which is the main constituents of ash of RS, easily dissolves in KOH. On the other hand, treatment using H_2SO_4 and $KMnO_4$ increases the ash content by increasing the inorganic constituent, such as sulfur and manganese compounds [23]. However, according

Table 2
Ash content of various activated carbons

Adsorbent	Ash content (%)	Adsorbent	Ash content (%)	Adsorbent	Ash content (%)
RS_1	49.0	RS_2	44.0	RS_3	40
RS_1/KOH	50.5	RS_2/KOH	19.2	RS_3/KOH	7.7
RS_1/HNO_3	54.1	RS_2/HNO_3	64.1	RS_3/HNO_3	26.8
RS_1/H_2SO_4	75.6	RS_2/H_2SO_4	51.8	RS_3/H_2SO_4	54.7
RS_1/H_2O_2	47.1	RS_2/H_2O_2	59.1	RS_3/H_2O_2	32
$RS_1/KMnO_4$	57.9	$RS_2/KMnO_4$	65.8	$RS_3/KMnO_4$	41.2

Table 3
Moisture content of various carbon adsorbents

Adsorbent	Moisture (%)	Adsorbent	Moisture (%)	Adsorbent	Moisture (%)
RS_1	16.2	RS_2	23.5	RS_3	28.5
RS_1/KOH	22.3	RS_2/KOH	26.0	RS_3/KOH	33.6
RS_1/HNO_3	15	RS_2/HNO_3	17.2	RS_3/HNO_3	23.5
RS_1/H_2SO_4	8.7	RS_2/H_2SO_4	27.8	RS_3/H_2SO_4	12
RS_1/H_2O_2	13.1	RS_2/H_2O_2	17.8	RS_3/H_2O_2	17.0
$RS_1/KMnO_4$	19.0	$RS_2/KMnO_4$	15.1	$RS_3/KMnO_4$	25.0

to the ash content data in Table 2, the amount of carbonaceous part removed and inorganic part removed or added depends not only on the type of oxidant, but also on the activation temperature of the parent activated RS. Thus, starting with an initial ash of 15%, it was possible to produce materials with varying inorganic contents [28]treatment of flue gas containing acid contaminant such SO_2 and in the treatment of wastewaters containing dissolved heavy metals [29].

3.3. Moisture content

Generally, adsorbents are priced on a moisture-free basis, although sometimes moisture content may be stipulated, e.g. 3, 5, 10% w/w unless stored in airtight packing. Under humid conditions, adsorbents may adsorb perhaps 25–30% moisture, over a few months, and still appear dry [15].

According to Macky [15], the moisture content does not adversely affect the adsorbent performance, in wastewater treatment, but merely acts as diluents. So that an additional quantity of adsorbent is required to provide the desired dry weight. In my opinion, this may occur only in adsorption of nonpolar molecules. Furthermore, the behavior of water vapor in porous carbons is very different from that of nonpolar molecules (owing to the hydrophobic or hydrophilic nature of the carbon surface) [28]. The process of water adsorption is very sensitive to the presence of surface functional groups on the carbon surface. They act as primary centers and initiate the process of adsorption via hydrogen bonding. Once the water molecule is adsorbed on the primary center, it becomes a secondary adsorption site and the clusters are formed [30]. The resulting adsorbed water molecules can prevent pollutant access to hydrophobic regions on the activated carbon surface and/or effectively block pollutant access to micropores. It is also remarked that organic contaminant adsorption from aqueous solution necessitates the displacement of water from the activated carbon surface [31].

As shown in Table 3, the moisture content for the carbon sorbents is relatively high with a value ranging from 12–33%. This may be due to the swelling of the carbons during preparation and washing. It has been reported that the acidic surface oxides increase the adsorption capacity for polar molecules, such as water vapor [32].

The results showed that parent activated carbon and that treated by KOH and $KMnO_4$ are basic while that treated by HNO_3 and H_2SO_4 are acidic. Closer inspection to the data in Table 3 shows a general trend, the moisture content of basic carbon is greater than of acidic one. Oxygen functionalities on the surface of a carbon are postulated to act as primary water adsorption centers. It is assumed that the water molecules interact only with oxygen surface complexes [33]. However, recently, Stoeckli and Lavanchy [34] demonstrated that water molecules may also interact with oxygen-free basic sites of Lewis type located at regions rich in π-electrons within the basal planes. The non-localized π-electrons have the ability to form hydrogen bond with water molecules. Carboxyl groups on acidic carbon can also form hydrogen bond, but it is clear that hydrogen bond formed by basic sites are dominant in our case.

Presence of basic or acidic sites on the surface of carbon is not the only factor affecting adsorption of water vapor on activated carbon. The moisture content of different adsorbents agrees to great extent with porosity detected by gas adsorption in latter section i. e sample treated by HNO_3 and H_2SO_4 have low porosity with low moisture content and samples treated by KOH have high porosity with high moisture content. i.e. the adsorption of water from atomsphere depends on the porosity and surface chemistry (surface sites) of activated carbon and increases with both. This is in agreement with some authors [35]. The third factor affects adsorption of water vapor as stated by Tsai and Chang [36] is the ash content which increases the hydrophilicity of activated carbon. This is reasonable since RS ashes consist mainly of silica, which have very high affinities for water.

3.4. Carbon yield

Single-step steam activation of RS gasifies a considerable amount of carbon leading to an approximate 21–29% yield as shown in Table 4. According to Toles et al. [37], if product yield from steam activation can be increased to the 20–25% range, without sacrifice surface area, steam treatments would be economically advantageous due to activant cost. Steam is significantly less costly to use than other oxidizing agents such as carbon dioxide. The yield of our carbons is higher compared to that of activated carbons prepared by one-stage chemical activation of RS prepared by Oh et al. [8] which gives yield of ca. 17%. Thus, we can conclude that, the treated single-step steam activation of RS results in high yield, which is very important from the economic point of view.

Table 4 shows the carbon yield as a function of activation temperature. It can be seen that the role of steam activation process in loss of material probably via the water gas reaction. With increasing temperature these reactions are leading to decrease the yield. The ability of water vapor to penetrate into the solid material and to help desorption, distillation, and efficient removal of the volatile products from it. Also, thermal destruction of carbon can explain the lower yield of solid residue with increasing temperature. i.e. burn-off reaction between carbon and steam, which is pronounced at high temperature where severe C–H_2O reaction exist [38].

The relationship between the product yield and S_{BET} (determined from gas adsorption in latter section) is shown in Table 4. The data show a trend towards maximum S_{BET} at a product yield of about 25.6%; i.e. S_{BET} increases with decreasing product yield to about 25.6% then decreases with further decrease of the product yield. In the activation process, the steam reacts with the carbon and oxidizes it to a gas product. Thus, pores are formed in the carbon, but oxidization also occurs on the surface of the sample. The latter reaction merely erodes the surface and does not increase S_{BET}. The increase of S_{BET} with decreasing product yield, thus, results predominantly from the pore formation reaction while the decrease of S_{BET} as the product yield is further decreased occurs by surface erosion rather than pore formation.

Since activation is associated with weight loss of the host carbon, the extent of burn-off of the carbon material is taken as a measure of the degree of activation. A microporous active carbon is produced when the degree of burn-off is less than 50% and a macroporous active carbon when the degree of burn-off is greater than 75%. When the degree of burn-off is between 50 and 75%, the product is expected to have a mixed structure and contain both micro- and macropores [39]. This appears in Table 4 the fraction micropores volume decreases with decreasing yield (i.e increasing burn-off) because of extensive gasification at high temperature. The activated RS has burn off from 71 to 79% which indicates that these carbons have mixed structure of micro and mesopores and these results are in good agreement with that obtained from nitrogen gas adsorption.

Steam activated and chemically modified carbons presented greater capacities for the adsorption of organic species of different molecular sizes from solution such as iodine, phenol, and methylene blue. Such behavior proves that the most important surface properties of these carbons affecting their solution adsorption behavior are their acidity/alkalinity and hydrophilicity. Although the carbons produced would not necessarily be appropriate for critical applications, such as potable water treatment, applications related to the treatment of industrial and municipal wastewaters, and other adsorption processes requiring less stringent quality standards, are potential beneficiaries. Assuming that production cost advantages exist by using RS carbons instead of currently available commercial activated carbons, there is potential to create new demand for the straw-derived adsorbents, thus stimulating the market for them and widening their usage further.

3.5. XRD characterization

XRD is one of the techniques commonly used for the structural characterization of activated carbon. Activated carbons in general show a 002 diffraction maxima at 2θ ~ 25° and overlapped 100 and 101 maxima (usually identified as the 10 peak) at 2θ ~ 42°.[17]

Table 4
Yield and burn-off of carbon adsorbents

Activation temp. (℃)	Yield (%)	Burn off (%)	S_{BET} (m^2/g)	Fraction micropore volume (%)
550	28.9	71	71.35	55
650	25.6	74.4	76.2	53
750	21	79	63.0	47

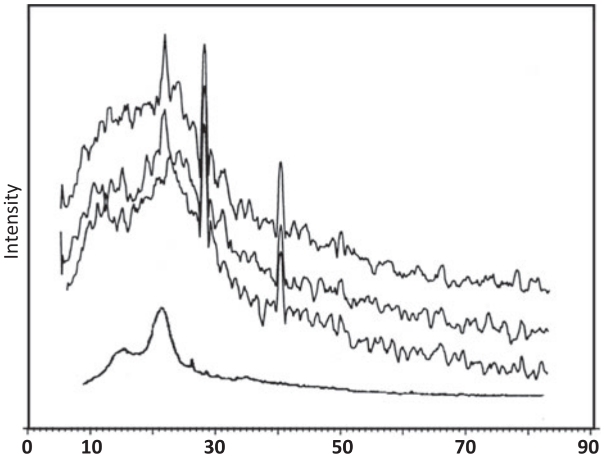

Fig. 1. X-ray diffractograms of original and activated RSs RS_1, RS_2, and RS_3 at 550, 650, and 750˚C, respectively.

The XRD patterns using Cu–Kα of the raw RS and activated carbons prepared at different temperatures are shown in Fig. 1. It can be clearly seen that, after activation of RS, the peaks at 2θ of 16 and 22E, characteristics of cellulose crystallite in RS disappear and the two newly broad peaks near 2θ of 25–30 and 38–42 exist. These new peaks that arise from carbon layer structure can be attributed to (002) and (10) reflection of the graphitic structure [40]. The sharp peak at 2θ of 21 is considered due to the crystallite of inorganic impurties, for example silicon dioxide, in RS [40].

The intensities of XRD peaks become sharper with the activation temperature. This means that the pore width increases with temperature due to collapse of small pores to larger ones. So, X-ray spectrum is sensitive to the changes during activation (this observation conformed by nitrogen gas adsorption in latter section). Typically, in a crystalline carbonaceous structure, such as graphite, the interlayer distance between two adjacent carbon sheets is 0.335 nm. In this study, the peak at 2 θ = 28. corresponds to an interlayer distance of 0.32 nm, which is quite similar to that of graphite. Based on these observations, it is postulated that activation of RS gives less ordered components, resulting in a reduction of the amorphous structure.

4. Conclusion

Activation temperature as well as modifying agent affect on the physical properties of the resultant carbon. Increasing temperature decreases the density, ash content, and carbon yield. Increasing activation temperature causes a successive release of volatiles, gases, and oils or tars. Deposition and retention of mixed oxide structure within the pores of the carbon as results of treatment of activated carbon leads to increasing density. Basic or acidic sites on the surface of carbon, porosity, and ash contents played important role water vapor. X-ray analysis indicated that activation of RS gives less ordered components, resulting in a reduction of the amorphous structure.

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