

# Synthesis of Activated Carbon from Rice Husk for the Enhancement of Microporosity and Carbon Dioxide Adsorption

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**ABSTRACT:** Microporous carbons synthesized from Rice husk activated carbon (RHAC) by KOH and/or ZnCl<sub>2</sub> treatment were examined as adsorbents for CO<sub>2</sub> capture. The specific surface area and micropore volume of the resulting carbons varied from 1187 m<sup>2</sup>/g (0.52 cm<sup>3</sup>/g) to 1802 m<sup>2</sup>/g (0.72 cm<sup>3</sup>/g), respectively. The acquired microporous carbon materials showed high CO<sub>2</sub> adsorption capacities at 40 bar pressure reaching 16.4 mmol/g. The objectives of this work were to develop a modified method to produce activated carbon from rice husk and to compare its physical and morphological characteristics with the activated carbon obtained at different temperature. The internal structure of the carbon was analysed with scanning electronic microscopy (SEM). The results of FTIR (Fourier-Transform Infrared Spectroscopy) strongly support significant chemical and spectral changes occurring with the activation of the rice husk with potassium hydroxide and ZnCl<sub>2</sub> treatment.

**KEYWORDS:** Activated carbon, RHAC, Microporous materials, CO<sub>2</sub> adsorption.

## I. INTRODUCTION

Carbon dioxide is one of the greenhouse gas produced by power plants, refineries, chemical, petrochemical, iron, steel and cement industries [1, 2]. The elimination of carbon dioxide from flue gases would reduce of air pollution and global warming [3, 4]. Removal of CO<sub>2</sub> is commonly accomplished by passing the gases through aqueous amine solution. This method has been widely applied in industry for about 60 years and is thought to be the most applicable technology for CO<sub>2</sub> capture before 2030 [5]. The alkanolamine solutions have some drawbacks such as high equipment corrosion rate, high energy consumption in regeneration, and a large absorber volume [6]. This method is costly and inefficient [7]. Among technologies proposed for reduction of CO<sub>2</sub> emissions such as physical absorption [8], adsorption [9, 10] and membrane separation [11], adsorption is considered a promising technology for CO<sub>2</sub> capture [12]. Different types of solid sorbents were investigated as potential adsorbents for CO<sub>2</sub> capture: zeolites, activated carbons, calcium oxides, hydrotalcites, organic-inorganic hybrids, and metal-organic frameworks [13]. Good adsorbents present high adsorption capacity, great selectivity, good mechanical properties and they remain stable over repeated adsorption-desorption cycles [14]. The growing interest in the capture of CO<sub>2</sub> requires an intensification of research on the development of inexpensive porous materials. One of the most promising class of CO<sub>2</sub> adsorbents are highly microporous activated carbons since they have high surface area, good CO<sub>2</sub> adsorption capacity and they are less expensive than other adsorbents [15, 16].

Activated Carbons as microporous materials are of the most important adsorbents, which have been

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extensively used as adsorbents, catalyst and catalyst supports in a variety of industrial and environmental applications. Activated carbon is an extremely versatile material as an effective adsorbent with its high adsorptive capacity and high surface area the activated carbon application attractive are availability, environmentally friendly material, safe and very low cost of the starting materials coupled with its high surface area[17].

Various materials are used to produce activated carbon and some of the most commonly used are agriculture wastes such as almond shell and walnut shell [18], palm shell [19], lapsi stone[20], palm oil shell[21], snail shell[22], cashew nut shell[23], sugarcane bagasse[24], wood[25], mustard stalk, jute stalk, sesame stalk, wheat straw [26], hazelnut shell[27], bamboo[28], Sewage sludge[29],etc.

Chemical activation has been given very effective carbon with high specific surface area. The most commonly used chemical activating agents are KOH, H<sub>2</sub>SO<sub>4</sub>, ZnCl<sub>2</sub>, Na<sub>2</sub>CO<sub>3</sub>, K<sub>2</sub>CO<sub>3</sub>,NaOH, H<sub>3</sub>PO<sub>4</sub> etc. ZnCl<sub>2</sub> create waste disposal problem and that is environmental unfriendly[18].

In this work,the influence of the texture of rice husk activated carbons on the adsorption of CO<sub>2</sub> was studied.RHAC was modified by KOH and/or ZnCl<sub>2</sub> treatment.Our motivation was to increase the porosity and adsorption capacityof obtained materials for CO<sub>2</sub>.

## II. RELATED WORK

Rice husk is an important biomass resource in South Asia including India and China. When compared to other agricultural Bio-wastes rice husk is known for low utilization value as husk and highly utilized as ash which is mostly in the form of silica. Organic matter of rice husk can be used as energy source and the inorganic composition has high value added utilization potential. Rice husk ash contains more than 80% of SiO<sub>2</sub> (w/w) [30]. Rice husk can also be used as a low-cost CO<sub>2</sub>adsorbent [31].

- i. The major composition of rice husk is silica and several volatile organic compounds (30). By exploring the best surface properties of silica, carbon dioxide adsorption studies are planned to carry out in depth.
- ii. This paper mainly describes the physical characteristics, pyrolysis of rice husk by impregnation with KOH and ZnCl<sub>2</sub> and their CO<sub>2</sub> adsorption capabilities.
- iii. Surface area and pore volume plays important role in any adsorption studies. Guest molecule CO<sub>2</sub> should fit in to the matrix of the host surface rice husk ash, so CO<sub>2</sub> molecules will hook on to the surface (32).
- iv. Calcination of rice husk at different temperatures in different atmospheres will produce a suitable matrix which removes volatile organic matter. This results in surface exposure due to increase in porosity. Higher the surface area greater will be activity.

## III.MATERIALS AND METHODS

### 3.1 Preparation of Activated Carbon

Activated carbons (ACs) were prepared from rice husk (obtained from a rice mill in Telaprolu, India, in the autumn of 2016) as following procedures: firstly, the rice husks were washed with water to remove dirt and other contaminants, oven-dried at 110 °C for 12 h then grounded and sieved to fractions with average particle size of 1.0 mm. Secondly, the prepared husks were carbonized at 400 °C under nitrogen flow (40 mL min<sup>-1</sup>) for 2 hours[33]. Theresulting rice husk activated carbons was treated by KOH and/or ZnCl<sub>2</sub>.Mass ratio of RHAC:KOH was equal to1:1.Saturated solution of KOH was used.

The concentration of ZnCl<sub>2</sub> in water was equal to 20%.The volume of 2cm<sup>3</sup>of solution was used per one gram of RHAC.The soaking time was 3h.The mixtures were dried for 19h at 200<sup>0</sup>C and pyrolyzed in a horizontal electric furnace at temperature of 800<sup>0</sup>C, during 1 h in flow of N<sub>2</sub>. The resulting materials were repeatedly washed with water, 5 M solution of HCl and water again until filtrate was neutral and free of chloride ions. Finally these samples were dried at 200<sup>0</sup>C for 12 h. The materials were denoted as RHAC/KOH, and RHAC/ZnCl<sub>2</sub>.

Material named as RHAC/KOH/ZnCl<sub>2</sub> was prepared in a multistep activation process with KOH and ZnCl<sub>2</sub>. In

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the first step, the activation process was done with KOH as described above. In the second activation step, the resulting RHAC/KOH was mixed with 20% solution of  $ZnCl_2$ . The other steps of RHAC/KOH/ $ZnCl_2$  preparation were the same as described above for RHAC/ $ZnCl_2$ .

## 3.2 Characterization

Textural characterization of carbonaceous materials was carried out by  $N_2$  adsorption–desorption isotherms measured at 77 K using Quantachrome Autosorb Instrument. Before analyses the samples were degassed at  $260^\circ C$  under vacuum for 16 h. The specific surface area was calculated using the Brunauer–Emmett–Teller equation. The total pore volumes were estimated on the basis of the volume adsorbed at a relative pressure of about 0.95. The pore size distribution and micropore volume was obtained after application of the QSDFT model to the nitrogen adsorption data and assuming a slit-shape pore model. High pressure  $CO_2$  adsorption studies were performed using Sievert's apparatus–Hiden Isochema Intelligent Manometric Instrument (IMI). The samples were degassed in the IMI before the measurements at  $260^\circ C$  under vacuum for 16 h. The skeletal volume was measured by helium in the IMI before  $CO_2$  adsorption. Equilibration relaxation kinetics were monitored using a computer algorithm based on an exponential decay model. Calculations were carried out in real time with an equilibrium uptake value determined when 99.9% of the predicted value was achieved. The  $CO_2$  adsorption isotherms were measured at the temperature of  $40^\circ C$ . The temperature of sample was controlled to better than  $\pm 0.1$  deg. The equation of state was used to calculate the amounts of adsorbed  $CO_2$ . The absolute absorption capacity was calculated by software provided by Hiden Isochema.

## IV. RESULTS AND DISCUSSION

### 4.1 Nitrogen Adsorption-Desorption Isotherms

The nitrogen adsorption-desorption isotherms presented at the Fig. 1 cannot be rigorously classified into any IUPAC type [34]. Typical type I isotherm exhibit sharp uptake at low relative pressures and approaches a limiting value as  $p/p_0$  tends to 1. A little adsorption at higher pressures was observed but we can classified them as type I isotherm that is encountered with microporous powders [35]. All the isotherms exhibit hysteresis loops. The two branches are nearly horizontal and parallel over a wide range of  $P/P^0$ . Type H4 loop is associated with narrow slit-like pores. The isotherm type I reveals a type H4 hysteresis loop indicating the presence of micropores rather than mesopores. The hysteresis about  $0.45 P/P^0$  suggests the presence of mesopores. The size of the hysteresis loop is related to the mesopore volume of the pores. The size of the hysteresis loop is similar for all the materials. This finding suggests that volume of mesopores was not affected by modification procedure. The amount of adsorbed nitrogen below  $0.01 P/P^0$  increases more quickly over RHAC/ $ZnCl_2$  than over RHAC. The amount of adsorbed nitrogen below  $0.01 P/P^0$  increases the most quickly over RHAC/KOH and RHAC/KOH/ $ZnCl_2$ . This finding suggests that  $ZnCl_2$  treatment develop micropore volume. The micropore volume of RHAC/KOH and RHAC/KOH/ $ZnCl_2$  is similar and the highest. The treatment of RHAC/KOH by  $ZnCl_2$  does not effect on the micropore and mezopore volume.

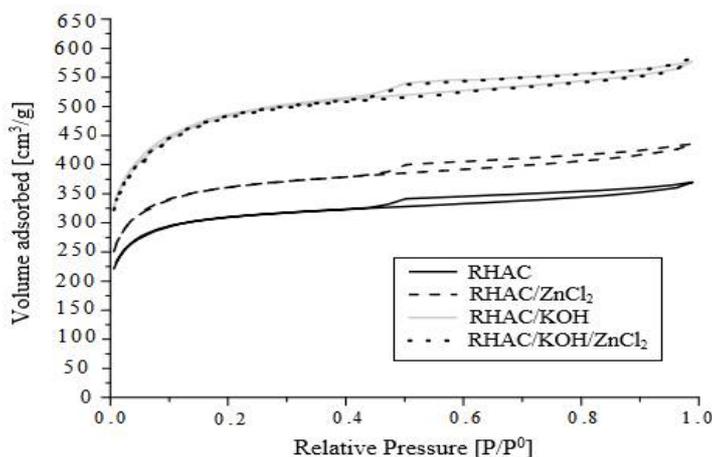


Fig. 1. Nitrogen adsorption-desorption isotherms at temperature of 77 K

#### 4.2 Pore Size Distribution

Fig. 2 shows the variation of pore size distribution evaluated by density function theory. Two dominant peaks are observed for the all materials. One locates located in the range 0.6 – 2 nm (max.0.8 nm) and the second in the range 2.7 – 4 nm. The micropores (pores up to 2 nm diameter) are dominant. The volume of mezopores (pores from 2 to 50 nm diameter) is very low. No pores with a diameter of more than 4 nm are found. Changes in the height of peak located in the range 0.6 – 2nm indicate changes in the porous structure of the activated carbon,in particular the formation of new micropores . This peak for RHAC/ZnCl<sub>2</sub> is higher than for the pristine sample.The highest peak in the micropore region is observed for RHAC/KOH and RHAC/KOH/ZnCl<sub>2</sub>. The high of the peak in the range 2.7 – 4 nm remain the same regardless of the treatment. This finding are consistent with the result shown in Fig.1.Table-1 shows BET surface area,Pore volume and micropore volume for the rice husk activated carbon synthesized samples.

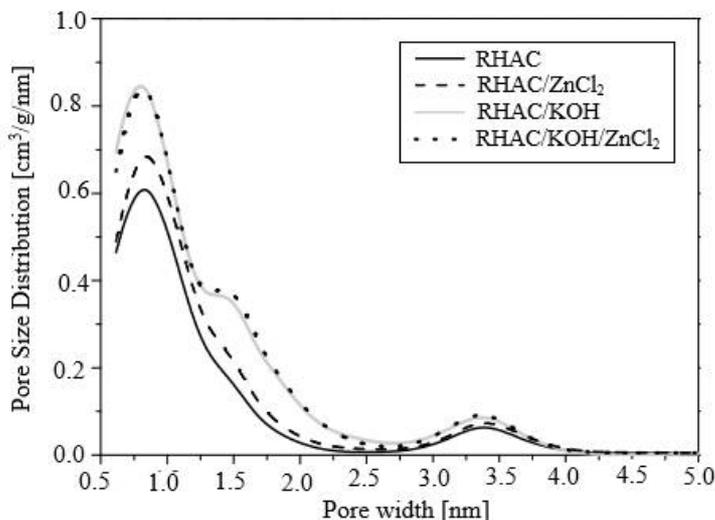


Fig. 2. Pore size distribution of Microporous carbons by density function theory

The carbons obtained with KOH exhibit the highest specific surface area and micropore volume (about 1802

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m<sup>2</sup>/g and 0.72 cm<sup>3</sup>/g respectively). The specific surface area of rice husk sample is 1187 m<sup>2</sup>/g and micropore volume 0.42 cm<sup>3</sup>/g.

**Table-1 Rice Husk activated carbon BET surface area, Pore volume & Micropore volume**

Name of the Sample	BET surface area (m <sup>2</sup> /g)	Micropore volume (cm <sup>3</sup> /g)	Pore Volume (cm <sup>3</sup> /g)
RHAC	1187	0.42	0.63
RHAC/ZnCl <sub>2</sub>	1368	0.63	0.72
RHAC/KOH	1802	0.72	0.89
RHAC/KOH/ZnCl <sub>2</sub>	1790	0.67	0.91

### 4.3 FTIR Analysis

The qualitative characterization of surface functional groups of AC samples was performed by the FTIR technique. The results illustrated in Fig. 1 show that the band at 3425 cm<sup>-1</sup> can be assigned to O–H stretching of hydroxyl groups [37]. The bands at 2924 cm<sup>-1</sup> and 1393 cm<sup>-1</sup> are attributed to C–H stretching of aliphatic carbon. The band at 2858 cm<sup>-1</sup> indicates the vibration of CH<sub>3</sub>–O group. The band appearing at 1627 cm<sup>-1</sup> corresponds to the C=O stretching of aromatic carboxyl groups [38]. The bands around 1545 cm<sup>-1</sup> indicates that C=C stretching of alkenes and aromatics. The band at 1096 cm<sup>-1</sup> are assigned Si-O-Si bond generally found in carbonaceous material, such as activated carbon [39].

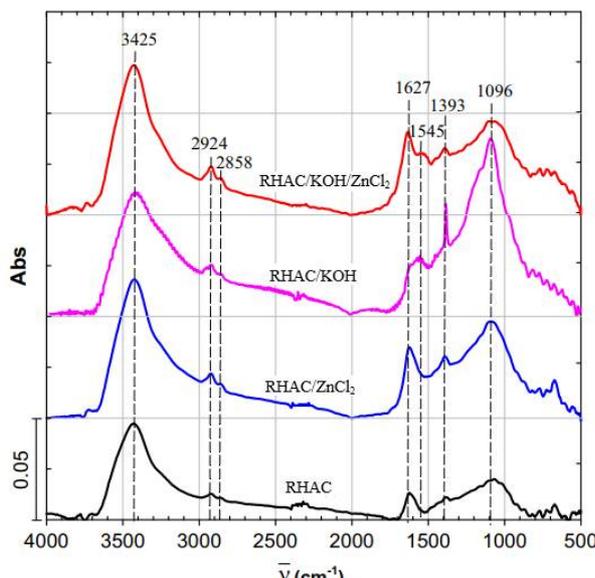


Fig.4. FTIR Spectra of RHAC and modified RHAC samples

### 4.4 Scanning Electron Microscope (SEM) Analysis

Scanning electron micrographs of AC samples were shown in Fig. 2. It can be seen from the pictures that all the AC samples exist in the form of spherical shaped particles with a size of about 10 nm that aggregated together to form pieces with different sizes. KOH impregnated Rice Husk treated with ZnCl<sub>2</sub> shows best porous structure with cracks and crevices. Which leads to best CO<sub>2</sub> adsorption when compared with other samples.

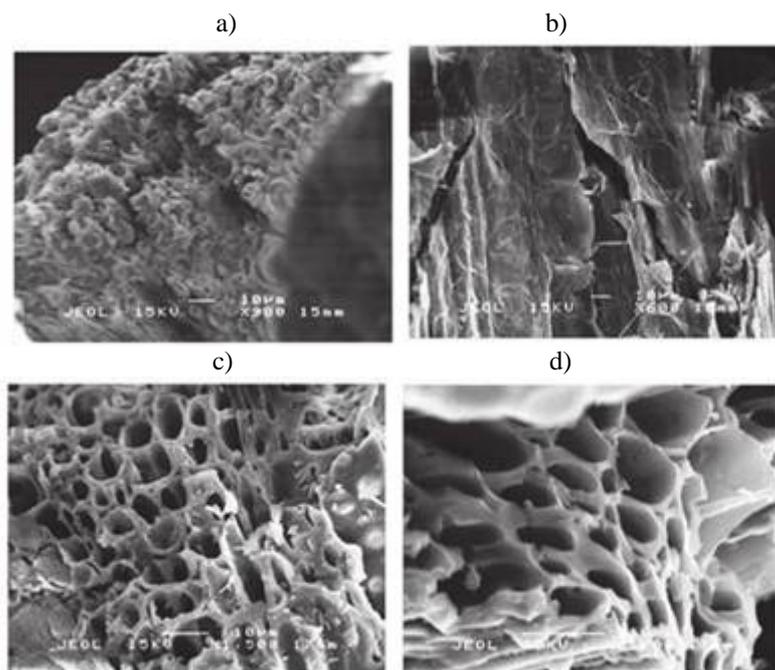


Fig.5.SEM images of rice husk samples a) RHAC, b) RHAC/ZnCl<sub>2</sub>, c) RHAC/KOH and d)RHAC/KOH/ZnCl<sub>2</sub>

#### 4.5 CO<sub>2</sub> Adsorption

The CO<sub>2</sub> adsorption was measured at 70<sup>o</sup>C up to 40 bar. The adsorption isotherms are shown in Fig. 6. As can be seen, CO<sub>2</sub> adsorption measured on the modified samples is higher than on the original sample. The values at 40 bar vary from 12.9 to 16.4 mmol/g and they are more 1.4 larger than 9.1 mmol/g CO<sub>2</sub> - uptake obtained for non-modified carbon RHAC. The values of CO<sub>2</sub> adsorption over RHAC/KOH is 1.7 times higher than over pristine. This suggests that the RHAC can be easily modified to good CO<sub>2</sub> adsorbent and even relatively small amount of KOH (KOH/RHAC ratio 1) can be used to achieve high CO<sub>2</sub> uptakes. This is an important aspect for scaling up the production [36] of modified RHAC because relatively small amount of KOH is needed to obtain good CO<sub>2</sub> adsorbent.

Note that the highest specific surface area (1802 m<sup>2</sup>/g) and the highest micropore volume(0.72cm<sup>3</sup>/g) was showed by RHAC/KOH sample but its CO<sub>2</sub> adsorption capacity at 40 bar was slightly smaller than the corresponding value obtained for RHAC/KOH/ZnCl<sub>2</sub> (specific surface area 1790 m<sup>2</sup>/g, micropore volume 0.69 cm<sup>3</sup>/g). The reason can be slightly higher value of pore volume (0.91cm<sup>3</sup>/g) of RHAC/KOH/ZnCl<sub>2</sub> than of RHAC/KOH (0.89 cm<sup>3</sup>/g). The surface morphology was investigated by Scanning Electron Microscopy method some differences are observed in the modified RHAC samples.

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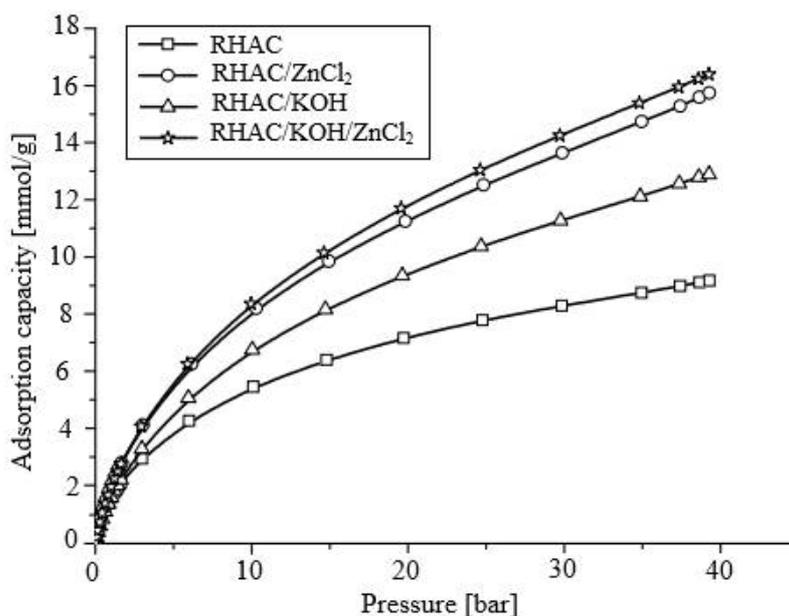


Fig. 6. Absolute CO<sub>2</sub> adsorption isotherms at 40 °C for the RHAC and modified RHAC samples

### V. CONCLUSIONS

Carbons with high volume of surface area and pores and micropores were obtained by chemical modification of RHAC. The modifications significantly improve adsorption properties of RHAC essential for enlarging the CO<sub>2</sub> uptake up to 40 bar.

This work shows that the modification of commercial activated carbon RHAC impregnated with Potassium Hydroxide followed by treatment with ZnCl<sub>2</sub> for the enhancement of Carbon Dioxide Adsorption capacity.

The highest specific surface area (1802 m<sup>2</sup>/g) and the highest micropore volume (0.72 cm<sup>3</sup>/g) was showed by RHAC/KOH sample but its CO<sub>2</sub> adsorption capacity at 40 bar was slightly smaller than the corresponding value obtained for RHAC/KOH/ZnCl<sub>2</sub> (specific surface area 1790 m<sup>2</sup>/g, micropore volume 0.67 cm<sup>3</sup>/g). The reason can be slightly higher value of pore volume (0.91 cm<sup>3</sup>/g) of RHAC/KOH/ZnCl<sub>2</sub> than of RHAC/KOH (0.89 cm<sup>3</sup>/g).

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