

# Preparation of Copper Impregnated Rice Husk Activated Carbon Promoted by Zinc for Enhancing Carbon Dioxide Adsorption Capacity

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**ABSTRACT:** CO<sub>2</sub> adsorption on raw and impregnated rice husk activated carbon (AC) at 298 K and pressures at 100 kPa has been investigated experimentally, using a vacuum adsorption apparatus, in order to get more information about the applicability of this material for flue gas cleaning. The adsorbents were prepared by impregnation with two metals Cu and Zn on the surface of the acid modified AC using a two-stage modification. At the first stage, the samples of AC were pretreated by oxidizing agent (nitric acid) to increase the amount of oxygen surface groups and at the second stage, the acid modified AC were impregnated by two metal salts Cu and Zn on the surface to produce a superior CO<sub>2</sub> adsorbent. Metal-loaded acid modified AC was prepared by using different ratios of Cu/Zn ranging from 4 to 20%. Synthesized samples were characterized by FTIR

Impregnation by metal shows up to 60% increasing in CO<sub>2</sub> adsorption capacity at 120 kPa. The adsorption data were fitted to the popular and applicable isotherm models, Freundlich and Sips, which are applied to heterogeneous adsorption systems. The Sips model was found to give the best fit for the adsorption of carbon dioxide on raw AC and Freundlich for impregnated AC.

**KEYWORDS:** Rice husk, Adsorption isotherm, Carbon dioxide, Activated carbon, Metal Impregnation (Cu/Zn).

## I. INTRODUCTION

Greenhouse gases have become a serious world problem and carbon dioxide (CO<sub>2</sub>) is the most prevalent greenhouse gas which can potentially cause severe changes in the global climate. Therefore reduction of CO<sub>2</sub> emissions seems to be an important task. On the other hand adsorption phenomenon is an efficient process which can be used to decrease CO<sub>2</sub> emissions [1, 2]. Activated carbons, with microcrystalline structure, are versatile adsorbents which are widely used as adsorbents of gases and vapors. Their adsorptive properties are due to their microporous structure, high surface area and high surface reactivity [3]. Although structural properties including surface area and pore size distribution are effective on the adsorptive capacity of activated carbon but when specific interactions of adsorbate/adsorbent play role in the adsorption process, other features such as surface chemistry should also be taken into consideration [4]. The surface chemistry of the activated carbons can be efficiently modified by chemical impregnation which together with the intrinsic nature of the activated carbon can highly increase the adsorption capacity. According to the nature of the adsorbate, the surface chemistry of activated carbon can be modified by creating acidic or basic groups. As CO<sub>2</sub> is an

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acidic gas, the presence of basic species can cause a priority basic character of the carbon surface which promotes the CO<sub>2</sub> capture capacity [3, 5].

Activated carbons (ACs) are versatile adsorbents which are mostly utilized as adsorbents of liquid, gases and vapors. Their adsorptive properties are due to high surface area, microporous structure and high surface reactivity [6]. Although structural properties of activated carbons are effective on their adsorptive capacity but specific interactions of adsorbate/adsorbent play an important role in the adsorption process [7]. Improvements of adsorptive properties of activated carbons can be achieved by using chemical and physical methods [8]. Metals loading such as salts of chromium, copper, iron and zinc on activated carbon has been recently reported [9–12].

The effect of impregnation of activated carbon with two metal oxides Cr<sub>2</sub>O and Fe<sub>2</sub>O<sub>3</sub> on its adsorptive properties of carbon dioxide studied [9]. The results indicated that impregnation of Cr<sub>2</sub>O increased the CO<sub>2</sub> adsorbed by about 20% in compared to unmodified activated carbon, while Fe<sub>2</sub>O<sub>3</sub> was not an effective impregnating species. Co-impregnation of Cr<sub>2</sub>O with zinc carbonate hydroxide led to another 5% increase in CO<sub>2</sub> capture capacity. According to literatures, the presence of copper oxide on carbon materials can increase the adsorption capacity of the acidic adsorbate [13,14].

The impregnation of activated carbons for adsorption of toxic gases has been investigated by some scientists in the literature. Improvement of adsorptive properties of activated carbons by metallic species such as salts of chromium, copper, iron and zinc has been recently reported to be effective for adsorption of acidic gases such as SO<sub>2</sub> on activated carbons [15-17].

The present work was aimed at investigating the adsorption of CO<sub>2</sub> on the activated carbon impregnated by Cu,Zn and binary mixtures of Cu/Zn with different ratios. However, no literature reported the application of Zn and the mixture (Cu/Zn) for CO<sub>2</sub> adsorption. The cubic structure of copper is more isotropic and adding hexagonal structure of zinc forms dendrite morphology (small branches). Therefore, more dendrite structures expected by a mixture of Cu/Zn [18]. The adsorption capacity of CO<sub>2</sub> on unmodified AC was compared with the modified AC that was prepared by binary mixtures of metals (Cu/Zn).

Although the introduction of metallic species as surface additives can modify the adsorptive properties of activated carbons, the used method might results negative effects on adsorption capacity. Impregnation of activated carbons by metal species can be an efficient way to improve the surface adsorption characteristics of activated carbons. However, further works are needed in order to clarify activated carbon impregnating effect on CO<sub>2</sub> adsorption. In the present work the adsorption of CO<sub>2</sub> on the activated carbons impregnated by metal has been studied experimentally. Also, best fitted isotherms for different cases were reported.

## 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) [19]. Rice husk can also be used as a low-cost CO<sub>2</sub> adsorbent [20].

- i. The major composition of rice husk is silica and several volatile organic compounds [19]. By exploring the best surface properties of silica, carbon dioxide adsorption studies are planned to carry out in depth.

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- ii. This paper mainly describes the physical characteristics, pyrolysis of rice husk by impregnation with metals Copper, Zinc and Binary mixture of Cu/Zn 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 [21].
- 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 Chemicals and materials

Activated carbons (ACs) were prepared from rice husk (obtained from a rice mill in Telaprolu, India, in the autumn of 2016) used as unmodified adsorbent. HNO<sub>3</sub> (65%) was used as oxidizing agent for pre-treatment of activated carbon and was purchased from Merck chemicals company. Most of researchers believed that the presence of carbon–oxygen surface functional group (due to oxidizing agent) on ACs surface is one of the most important factors that affect deposition of metals. HCl (36.5%), NaHCO<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub> and NaOH were used for Boehm's titration in order to study the functional groups of adsorbent surface that was prepared from Merck, India. High purity commercial powder metal nitrate, Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O (99.5%, Sigma-Aldrich) and sulfate, ZnSO<sub>4</sub>·7H<sub>2</sub>O (>99%, Sigma-Aldrich) were used as metal oxide pre- cursor to modify AC. Carbon dioxide (99.99%) and purified helium (99.99%) were purchased from Global Plus Solution Company. All other reagents and chemicals used were of analytical grade or as reported.

### 3.2 Modification of adsorbent

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[7].

The initial stage of preparation method for the modification of adsorbent is pre-treatment of AC by using HNO<sub>3</sub> solution. 50 g of AC was mixed with a proper amount of HNO<sub>3</sub> solution (1 g carbon/3 mL HNO<sub>3</sub>). The mixture was continuously stirred and heated for 2 h at temperature range 90–105 °C in a reflux system to prevent loss of HNO<sub>3</sub>. After oxidation, the AC was washed several times with distilled water to remove the residual HNO<sub>3</sub> and the sample was then filtered. The sample was then dried in an oven at 110 °C for 12 h and ready for loading of metals. The samples were impregnated with Cu(NO<sub>3</sub>)<sub>2</sub> and ZnSO<sub>4</sub> by an aqueous ion-exchange process. The amounts of metal loading ranged from 2 to 10% of AC for each pure metal. The AC was first impregnated by using an aqueous solution of the precursor (Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O). During the impregnation, the solution was heated to 70 °C and stirred until the liquid was totally evaporated. The samples were then dried at 110 °C for 24 h and following calcination under N<sub>2</sub> flow at 600 °C for 4 h. The procedure was repeated for impregnation using ZnSO<sub>4</sub>. The percentage of metal loading was used to index samples. The prepared samples are (Cu/Zn (4%), Cu/Zn (8%), Cu/Zn (12%), Cu/Zn (16%) and Cu/Zn (20%)) and the total metal loading is reported as 4, 8, 12, 16 and 20%. The amount of metals was equally considered for impregnation. The properties are summarized in Table 1.

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Table 1 :Properties of original AC, HNO<sub>3</sub>-treated and modified AC samples.

Sample	SBET (m <sup>2</sup> /g)	Micropore volume (cm <sup>3</sup> /g)	Mesopore volume (cm <sup>3</sup> /g)
AC	1326	0.701	0.188
HNO <sub>3</sub> -treated AC	1192	0.697	0.132
Cu/Zn-4% AC	937	0.598	0.124
Cu/Zn-8% AC	860	0.552	0.117
Cu/Zn-12% AC	790	0.502	0.109
Cu/Zn-16% AC	740	0.486	0.104
Cu/Zn-20% AC	598	0.399	0.089
Cu-20% AC	645	0.423	0.096
Zn-20% AC	622	0.408	0.094

### 3.3 CO<sub>2</sub> adsorption procedure

The CO<sub>2</sub> adsorption was carried out in a fixed-bed column. The experimental apparatus used to measure CO<sub>2</sub> adsorption is shown in Fig. 1. The stainless steel fixed-bed reactor was designed with dimension 33 cm height, 2.5 cm diameter and equipped with a porous plate that is located 3 cm from the base of the column. The gas manifold system consisted of two lines fitted with mass flow controllers from Bronkhorst High-Tech with flows ranging between 1 and 1000 mL/min. The controllers demonstrated an accuracy of 1% full scale and a repeatability of 0.1% full scale. Two feeds lines contain CO<sub>2</sub> and He was installed in the system. Different gas mixtures can be prepared by adjusting the concentrations of both gases. Three temperature sensors (type K thermocouple) are placed at the inlet, the middle and the outlet of the adsorption column for temperature measurement. The thermocouples provided continuously monitoring of the column temperature with an accuracy of  $\pm 1.5$  °C. The temperature was controlled by an electrical resistor coiled around the reactor inside the thermal insulation jacket. A back-pressure regulator was installed to control the column pressure with a repeatability of 0.5% full scale. The system was also equipped with a CO<sub>2</sub> analyzer (GFG,50%) at outlet of the column. The CO<sub>2</sub> concentration of effluent gas was continuously monitored as a function of time to obtain the breakthrough curves. Before each run, the adsorbent is dried the following He(50 mL/min for 15 min at 100 °C at atmosphere pressure. After the drying step, the bed temperature and pressure were adjusted and gaseous mixture CO<sub>2</sub>/He was fed through the column. The inlet flow rate was kept constant (250mL/min). Different operational conditions such as pressures (100-200 kPa), temperatures (30, 40 and 50 °C) and CO<sub>2</sub> concentrations (5-50%) were tested. CO<sub>2</sub> adsorption tests were carried out volumetrically in a stainless steel vacuum system. Fig. 1 shows a schematic diagram of apparatus which consists of a gas injection chamber of known volume connected to an adsorption chamber. Cell volumes were estimated using water displacement method [22]. Adsorption cell volume in the presence of sample was determined by the well-known helium expansion method [23]. The gas chamber and adsorption chamber volumes were  $77.1 \pm 0.1$  and  $31.2 \pm 0.1$  cm<sup>3</sup>, respectively. Using the volumetric method with P-V-T measurements, the total amount of gas introduced into the system and the remained in the system after reaching adsorption equilibrium was determined. The adsorbed amount was calculated using ideal gas law at low pressure prevailing, by measuring the pressure drop caused by CO<sub>2</sub> adsorption. The system was operated at room temperature and the CO<sub>2</sub> pressure which was provided in adsorption tests in the range of  $30-110 \pm 0.1$  kPa. Prior to the adsorption measurements, the samples were regenerated at ambient 313 K under high vacuum 0.02 kPa for 1 h.

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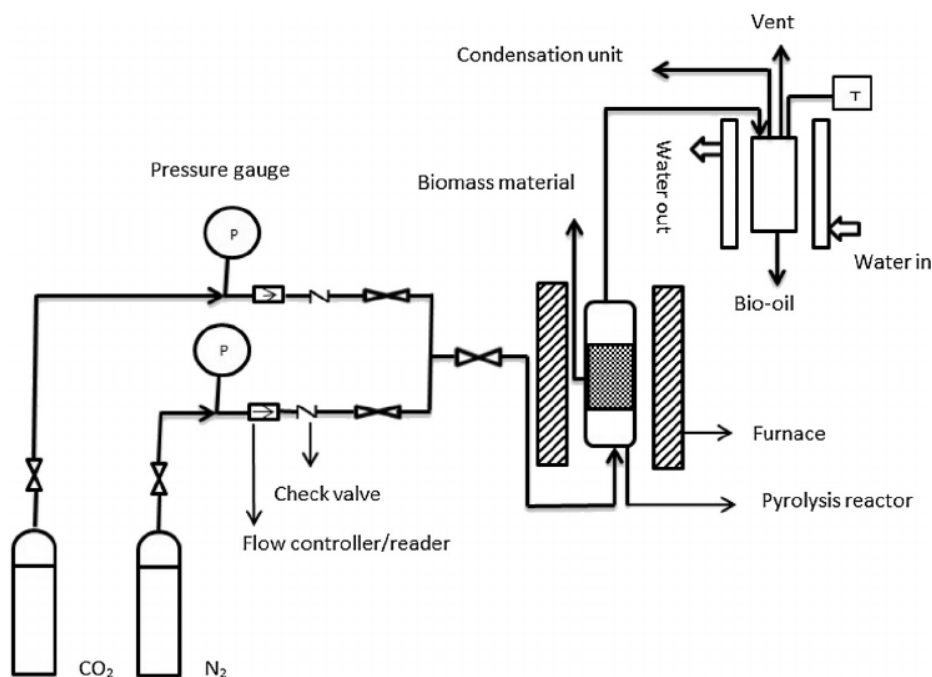


Fig. 1. Schematic diagram of volumetric adsorption system.

The CO<sub>2</sub> adsorption data on the impregnated activated carbons at room temperature and pressures range from 20 to 120 kPa were fitted to standard isotherm models such as Freundlich, Langmuir and Sips equations [24]. These equations are presented in Table 2.

Table 2. Different isotherm equations [24].

Freundlich	Langmuir	Sips
$q = kp^{1/n}$	$q = q_{max} \frac{bp}{1 + bp}$	$q = q_{max} \frac{(bp)^{1/n}}{1 + (bp)^{1/n}}$

## IV. RESULTS AND DISCUSSION

### 4.1 Characterization of adsorbent

Nitrogen adsorption/desorption is a standard technique for the characterization of porous materials. Figs. 2 showed N<sub>2</sub> adsorption/desorption for unmodified AC and modified samples (metal loaded AC). The isotherms can be classified as type IV isotherm which is typical of adsorption in micropores and mesopores [25]. The metal loading was caused a decrease in the surface area of AC. This decrease may be due to some of pores blocked by the presence of oxide functional groups introduced during the HNO<sub>3</sub> treatment and loading metals during the modification [26] so that N<sub>2</sub> molecules cannot transport into the adsorptive sites. Also commercial ACs may contain inorganic species which can be removed by HNO<sub>3</sub> oxidation. As seen in Table 1, after treatment, the surface area decreased slightly from 1326 to 1192 m<sup>2</sup>/g and more diminish from 937 to 598 m<sup>2</sup>/g was appeared when the

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metals were loaded from 4 to 20%. However, no change was occurred in the micropore volume of the activated carbon, suggesting that the  $\text{HNO}_3$  treatment does not affect significantly in the micropores [27,28]. The reduction in surface area, total pore volume, micro and mesopore volumes were dependent on the amount of loaded metals.

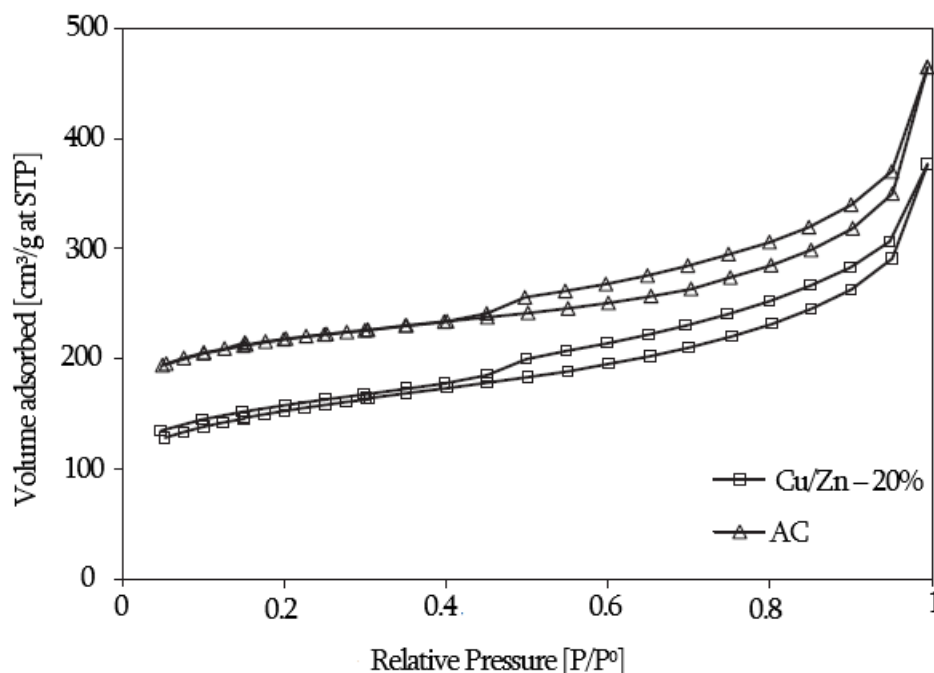


Fig.2.  $\text{N}_2$  adsorption/desorption of unmodified-AC and modified AC (Cu/Zn-20%) at 77K.

## 4.2 FTIR Analysis

Fig. 3 shows FTIR spectra of both unmodified and modified AC in the range  $600\text{--}4000\text{ cm}^{-1}$ . The wide peaks at  $3456\text{ cm}^{-1}$  and  $3452\text{ cm}^{-1}$  illustrated the expanding vibration of O–H bond involving hydrogen bonds for unmodified and modified AC, respectively. The band appearing around  $3000\text{ cm}^{-1}$  is assigned to alkenyl C–H stretch. The band at  $2852\text{ cm}^{-1}$  is revealed as alkyl C–H stretch. The most significant differences in the surface functional groups were appeared during the acidic treatment and it can be easily seen in the range of  $1600\text{--}1800\text{ cm}^{-1}$ . The appeared peak at  $1750\text{ cm}^{-1}$  in modified AC spectrum is assigned to stretching vibrations C=O bond in carboxylic acid functional group. The bands at  $1589\text{ cm}^{-1}$ ,  $1374\text{ cm}^{-1}$  and  $1232\text{ cm}^{-1}$  are appeared due to C=C aromatic, C–OH and the C–O–C stretch, respectively. Moreover, the intense peak at  $1056\text{ cm}^{-1}$  is ascribed to C–O stretching vibrations which may be caused by ether, phenol, carboxylic acid, and carboxylic anhydride. The metal bands were appeared at lower frequencies around  $750\text{ cm}^{-1}$ .



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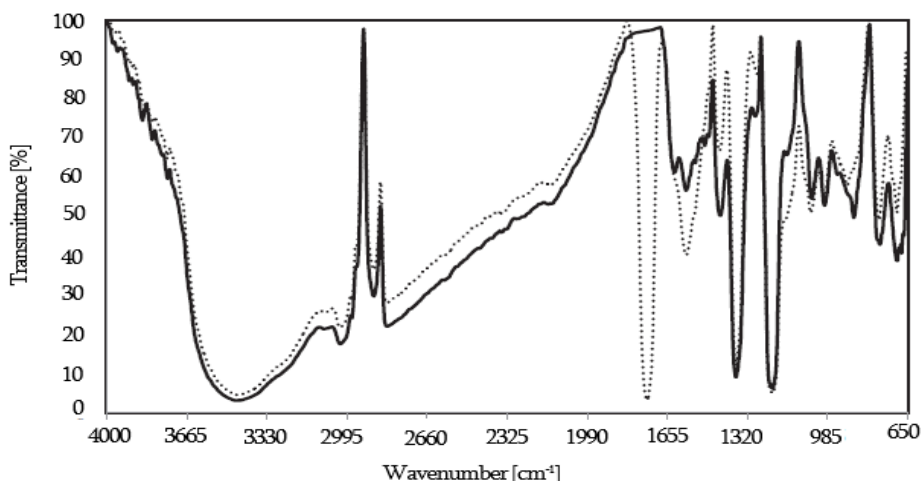


Fig.3.FTIR pattern for unmodified AC (solid line) and acid modified AC (dashed line).

### 4.3 Effect of modification

Fig.4 shows the adsorption capacity for various AC samples. The sample Cu/Zn–20% increase the CO<sub>2</sub> adsorption capacity by approximately 49% compared to unmodified AC. The modified samples with single metal (Cu or Zn) indicated higher adsorption capacity than unmodified AC. The Cu loaded AC revealed higher capacity than Zn loaded AC. The comparison indicated the modification process is effective for increasing adsorption capacity. All the experiments were carried out in the same operating condition (120 kPa, 30 °C and 15% CO<sub>2</sub> concentration). The CO<sub>2</sub> adsorption capacity is greatly enhanced because of the reaction of CO<sub>2</sub> with the supported metals occurred when they were physically adsorbed onto the micropores of the carbon [29]. Many researchers reported similar conclusion for activated carbon modified with different metals [10–12,30].

The values of the rate constants for different modified AC samples are summarized in Table 5. As given in Table 5, the CO<sub>2</sub> adsorption capacity of co-impregnation Cu/Zn–20% was around 11% higher compared to single metal impregnation Cu-20%. This clearly indicates that surface modifications by metal impregnation increase the affinity of carbon towards CO<sub>2</sub>.

In Fig. 4 adsorption capacity at different pressures (from 20 to 120 kpa) and constant temperature of 25°C is presented in Table.3. The sample Cu/Zn–20% increase the CO<sub>2</sub> adsorption capacity when compare with the modified samples with single metal (Cu or Zn) and an unmodified AC.

Table-3 Adsorption capacities of unmodified and modified AC samples

Sample	Temperature(°C)	Pressure(kPa)	Adsorption capacity(mmol/g)
Unmodified AC	30	120	1.92
Zn-20% AC	30	120	2.79
Cu-20% AC	30	120	3.01
Cu/Zn-20% AC	30	120	3.32

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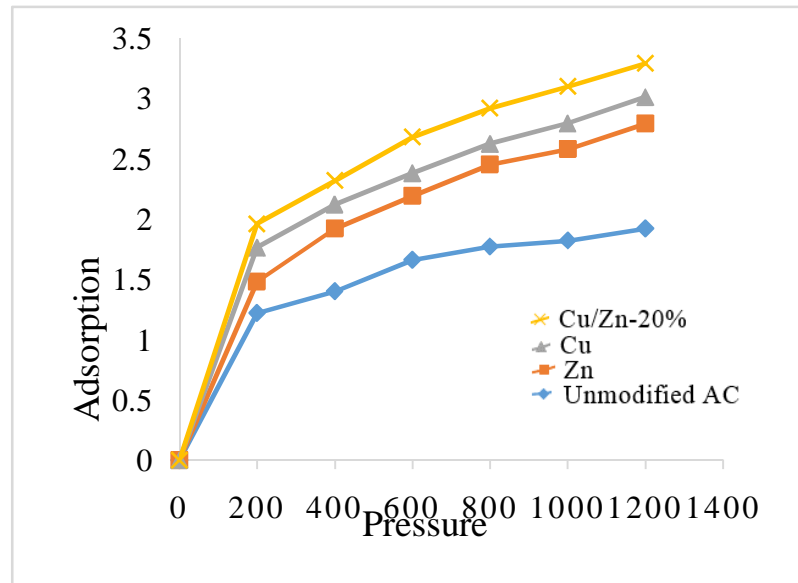


Fig.4.CO<sub>2</sub> adsorption capacity on unmodified and modified AC.

## 4.4 Desorption process

In order to study the regeneration process, the adsorption process was carried out by the column packed with the adsorbent and saturated with a stream of the CO<sub>2</sub>/He mixture at atmospheric pressure and the flow rate of 250 mL/min. The desorption process of two samples unmodified AC and Cu/Zn–20% modified AC was shown (Fig. 5). Fig. 5 demonstrated variation of the CO<sub>2</sub> concentration with time during desorption process. The CO<sub>2</sub> concentration was sharply decreased with time and the initial value of  $C/C^0$  is started from the value of 1. As seen that unmodified AC indicated a regeneration process time less than Cu/Zn–20% modified AC.

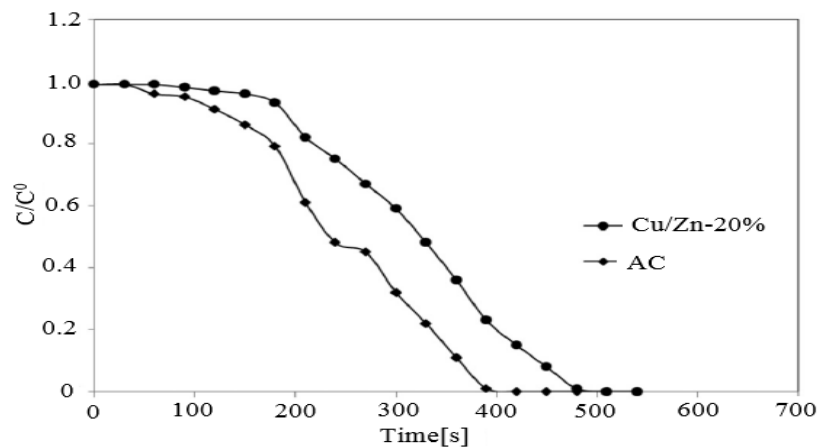


Fig.5.Regeneration curve of the saturated unmodified AC and modified 20% Cu/Zn ( $T=80^{\circ}\text{C}$ , He gas 250 cm<sup>3</sup>/min).



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## V. CONCLUSIONS

The aim of this work is the effect of binary mixture of metals on activated carbon and investigation CO<sub>2</sub> adsorption capacity of modified AC in different range of loading which can be used for flue gas treatment. Cu/Zn-modified samples adsorbed more than the unmodified sample and the adsorption was increased when the higher percentage of metal was loaded. The blend of metals improved the adsorption capacity around 42% when 20% Cu/Zn was loaded on AC. The adsorption capacity for unmodified AC in the temperature of 30 °C, pressure 120 kPa and 15% CO<sub>2</sub> concentration is 1.92 mmol/g and after modification (20% of metal loading) the capacity increased to 3.32 mmol/g. One the most important factor affecting the adsorption process is temperature. In the case of physisorption, the adsorption time increases with decreasing temperature. In the higher concentration of CO<sub>2</sub> take less time to reach the adsorption time and increasing pressure caused a longer breakthrough time. The amount of adsorbed CO<sub>2</sub> in the pretreated sample is less than unmodified sample. Since CO<sub>2</sub> is an acidic gas and the treatment by using HNO<sub>3</sub> provided more acidic functional groups and consequent a reduction on CO<sub>2</sub> adsorption due to increase acidic groups. The mechanism of CO<sub>2</sub> removal is principally due to physical adsorption and chemisorption process. The small pore sizes and large surface area of activated carbon play the major role in gas adsorption.

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