Application of Zeolite for Post Combustion Carbon Capture

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Abstract— **Stabilization of anthropogenic Carbon dioxide on massive scale calls for development and deployment of cleanup mechanisms as Carbon Capture and Storage. Adsorption by physical regenerable sorbents is a promising method of separation of flue gases. This paper focuses on evaluation of carbon capture potential of Zeolite 4A. Removal efficiency, adsorption capacity of selected adsorbent is analyzed volumetrically. Six Adsorption cycles are carried out in a fixed bed adsorption column using Pressure Swing Adsorption. The adsorption capacity ranges from 0.17- 2.5 mol/kg and removal efficiencies vary between 6.7 – 33.3 %. Temperature increase limits the adsorption potential of the sorbent.**

Keywords—Carbon capture; adsorption, regenerable sorbents, zeolte.

I. INTRODUCTION

Despite of number of mitigation policies global green house emissions have reached 49 Gt CO_2-eq in 2014 [1]. It is estimated that the CO_2 emissions will increase by 3,210 mt by 2030. Coal driven thermal power plants contribute 37% of total CO_2 emissions [2]. India has submitted the emission inventory in the Second national Communication (NATCOM) in 2012. In 2000 the total GHG emissions were reported to be 1523 Mt-CO_2-eq. in which energy sector contributed 92.7% [3]. Being party to UNFCCC, India is abided to stabilize anthropogenic CO2 emissions by 33-35% of the base year 2005 by 2030. However, in the present situation we are far behind than that of the emission reduction targets. Massive reduction in CO_2 with continued use of fossil fuels calls for deployment of Carbon Capture and Storage (CCS) technologies.

A. Approaches and mechanisms of carbon capture

CCS is an "end-of-pipe" treatment which refers to separation of CO2 from the flue gases emitted from a point source and its subsequent long term storage in biological or geological sinks. Three basic approaches are implemented for carbon capture (Fig.1) namely pre-combustion capture, post combustion capture and oxy-fuel combustion capture [4]. Pre-combustion processes include gasification of fuel to form mixture of CO_2 and H_2. Hydrogen is used for energy generation and CO_2 is captured. In oxy-fuel combustion oxygen is used instead of air for fuel combustion. This results in reduced volume and concentrated stream of flue gases containing oxides of sulfur (SO_2), carbon dioxide and water.

Post combustion process employs physical and chemical means to separate CO_2 from flue gases. The CO concentration in flue gas varies from 5-15 % by volume. Post combustion capture involves CO2 removal at higher temperature and lower concentration. More over higher volumetric loads need to be handled [5]. Despite of these limitations post combustion processes have highest near term potential of development and commercialization.

Absorption, adsorption, membrane separation and cryogenics are four mechanisms for CO_2 capture. Adsorption, absorption and membrane processes are suitable for post combustion capture whereas cryogenics is appropriate for oxy-fuel and pre combustion carbon capture [5, 6].
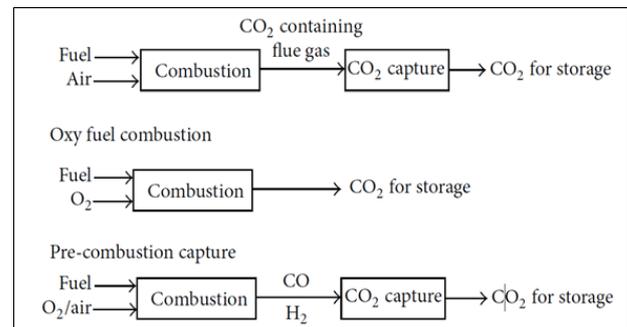

Fig.1 Carbon capture approaches

Present study focuses on removal of CO2 by adsorption using solid regenerable sorbents. Brunetti et al. have critically compared this mechanism in view of energy requirement, operational parameters, recovery, stages of development and future commercialization opportunities [7]. According to International Energy Agency (IEA) CCS technologies will contribute 19-20% curtailment in CO2 emissions by 2050 [8].

B. Carbon Capture by adsorption

Carbon capture by adsorption is gaining growing concern over the most established wet scrubbing process. It is one of the most studied mechanisms having near term application potential as emission cleanup technology [9]. This process employs solid sorbents to remove CO2 from the flue gases by physical or chemical adsorption. A pure stream of CO2 is obtained during desorption cycle and the adsorbent can be regenerated [10].

II. LITERATURE REVIEW

Various physical and chemical adsorbents are evaluated to assess their CO2 capture capacity by number of researchers. Novel amine enriched HSC sorbent [11], Potassium carbonate coated polymers [12], amine modified activated carbon [13], amine coated fly ash [14], and chemically modified activated carbons [15] are evaluated using Thermo Gravimetric Analysis. TGA results indicated that carbon capture is favored at low temperature and regeneration preferentially takes place at higher temperature. Temperature Swing Adsorption is suitable for chemisorbents.

Physical adsorbents mainly include carbonaceous adsorbents, and molecular sieves. Activated carbon is most studied carbonaceous adsorbent. Experimental investigations on activated carbon are carried out using Pressure Swing Adsorption [16, 17, 18, and 19]. Modified activated carbons are reported to have reduced capture potential than raw activated carbon [5]. According to Hedin et al. activated carbons can withstand high pressure 110 kPa and have capture potential of 3-4 mol/kg [20].

Krutka et al. have investigated commercial activated carbon, amine modified activated carbon and carbonate modified activated carbon and observed to have working capacity of 0.7%, 0.4% and 1.2 % respectively by weight [21]. Saxena et al. have presented adsorption kinetics study on activated carbon is presented by [22]. Adsorption isotherms indicate saturation capacity of 0.4 m^3/g. Hauchhum et al. have obtained breakthrough curves for activated carbon at variable temperature (25-60 ^{0}C) and pressure (0-1 bar). The highest capture potential of 2.5 mol/kg was obtained at 1.0 bar and 25^{0}C. The results are comparable with Langmuir isotherm model predictions [18].

Zeolite is another group of sorbents having higher surface area and pore volume. They are porous alumina-silicate materials with uniform pore structure and minimum channel diameter ranging from 0.3 to 1.0 nm [23]. The molecular sized pores remove CO_2 both through mechanical sieving and surface adsorption [24].

Siriwardane et al. have compared CO_2 removal by Zeolite 13X and natural zeolites under pressure swing adsorption. The maximum CO_2 capture of 7 mol/kg is reported at 250 psi for zeolite 13X where as for natural zeolite capture potential is observed as 1.32 mol/kg. Grande et al. have evaluated performance of zeolite 13X using principles of pressure swing adsorption. The amount of CO_2 adsorbed was measured at different operating pressures. The observed results are compared with Langmuir model. Similar study was conducted by Hauchhum et al. [18]. Breakthrough curves and adsorption isotherms were obtained at variable pressures. Freundlich isotherm is used to compare the observed results and estimated values.

Songolzadeh et al. have explored wide range of synthetic adsorbents that are being generated and assessed for their carbon capture potential. Carbon nanotubes (CNT) are Carbon nanotubes (CNTs) are the most famous among nano-hollow structured materials. Graphite Nano Platelets (GNP) are new generation carbonaceous adsorbents and reported to process highest adsorption capacity, 13.52 mol/kg and 3.8 mol/kg [8,18]. In the past two decades a new class of crystalline porous material Metal Organic Framework (MOF) has emerged. Li et al., and Liu et al. have investigated variety of MOFs along with their operational parameters and adsorption capacity[25, 26]. They are crystalline three dimensional structures with interconnected pores. They are suitably used both under temperature and pressure swing adsorption [27].

III. OBJECTIVES

The project work focuses on experimental evaluation of carbon capture capacities of selected solid physical regenerable sorbents using a lab scale model. The objectives within the scope of the project work are enlisted below.

- To evaluate carbon capture capacity of selected adsorbent on volumetric basis.
- To assess effect of pressure and flow rate on performance parameters of selected adsorbent.
- To measure removal efficiency of selected adsorbents at constant flow rate.
- To monitor effect of temperature on adsorption capacity.
- To assess recovery of CO2 during desorption process.

IV. METHODOLOGY

A fixed bed adsorption column is designed to conduct the experimental work. The adsorbent material is selected based on physical properties, surface area, porosity, availability, cost, and operational parameters. Zeolite 4A procured from Zinolab (Sinnar, Maharshtra) is chosen as adsorbent for present study.

The material characterization involves determination of physical, chemical and surface properties of the materials. The surface chemistry of the zeolite is determined by Infrared Spectroscopy where as chemical composition was determined through elemental analysis. The porosity data and chemical composition data was obtained from the manufacturer. Simulation of flue gas is an essential step in experimental work.

Mixture of CO_2 and inert gas in desired proportion is provided by Nataraj Enterprises (Satpur, Nashik). The adsorption cycles are run at variable pressure and constant flow rate for predecided cyclic period. The outlet gas is sampled in gas sampling bladder and CO_2 concentration is analyzed using ORSAT apparatus volumetrically. Adsorption potential is evaluated by varying operational pressure and flow rate.

Desorption of captured CO_2 is carried out by reversing the direction of gas flow and operating the reactor in down sweep mode. Desorbed gases are passed through absorbing liquid and quantity of CO2 desorbed is measured gravimetrically. The CO2 recovery is calculated from the amount of CO2 adsorbed and amount of CO2 desorbed.

V. EXPERIMENTATION.

A. Description of experimental setup

The adsorption column is prepared using UPVC pipe of 1.5 inch (35.1 mm). The assembly is provided with inlet and

outlet arrangement using 0.5 inch pipes. (Fig 2). The inlet is provided with diffuser plate to ensure equal distribution of gas through the adsorbent bed. Outlet is provided with porous plate to retain the adsorbent material in fixed bed mode. The adsorption column is operated in up-flow fixed bed mode.

A gaseous mixture simulating flue gas is mixture of Argon (Ar, 85%) and Carbon dioxide (CO_2, 15 %) is used to simulate flue gas during adsorption cycle whereas pure Nitrogen (N_2) is used as carrier gas during desorption cycle. The cylinder is equipped with regulator to control and measure pressure (0-16 kg/cm^2). A rotameter (0-15 LPM) monitors the feed flow rate and the thermometer (0-150^{0}C) monitors temperature of the adsorbent throughout the adsorption cycle. Table I summarizes operational parameters during experimentation.

B. Experimental procedure

Six adsorption cycles are carried out at operational pressures 0.2, 0.4, 0.6, 0.8 , 1.0 and 1.2 kg/cm^2. The duration of adsorption cycle is 15 minutes. At every five minutes temperature is recorded. Outlet gas is sampled at 5 minute interval in sampling bladders and concentration of CO_2 id measured analyzed using ORSAT apparatus.

After every adsorption cycle desorption of the gases is carried out. The carrier gas is passed through the column from top at 0.1 kg/cm^2. The desorbed gases are allowed o absorb in saturated Barium Hydroxide (Ba (OH)$_2$) solution to form a precipitate of Barium Carbonate. (BaCO$_3$). The precipitate is then filtered, oven dried and weighed. The amount of CO_2 precipitated is calculated from proportion of molecular weights of BaCO$_3$ and CO_2 and further convertd into number of moles.

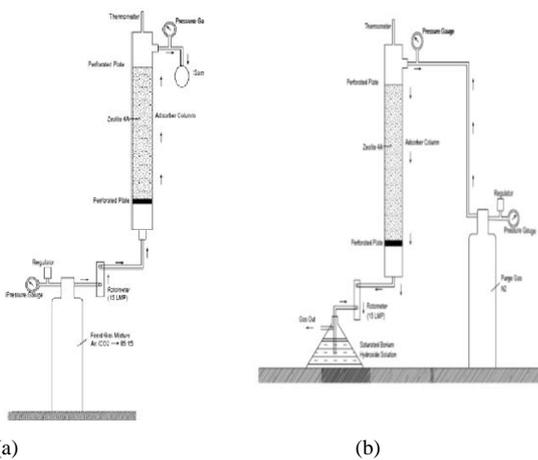(a)   (b)

Fig 2 Experimental setup for (a) Adsorption design (b) Desorption

TABLE I. OPRATIONAL RAMETERS FOR EXPERIMENTATION

Sr.No	Parameter	Value with unit
1	Inlet CO2 concentration	15%
2	Gas flow rate	1.-5 lpm
3	Type of operation	PSA
4	Diameter of column	38 mm (1.5")
5	Depth/ height of column	600 mm
6	Volume of the bed	680 cm^3
7	Operational pressure	0.0-1.2 kg/cm^3
8	Cyclic period	10 minutes
9	Weight of adsorbent	300 g

The reactor column is refilled with Zeolite 4A after every adsorption cycle. Response of adsorbent to variable flow rates is evaluated at optimum pressure and adsorption period.

C. Calculation of performance parameters

Evaluation of the adsorbent is carried out in terms of amount of CO_2 adsorbed ($q_{adsorbed}$, mol), CO_2 removal efficiency ($\eta\%$), and adsorption capacity (mol/kg) at the end of adsorption cycle. Knowing the concentration of CO_2 at inlet (C_i), concentration of CO_2 at outlet (C_o) and volume of simulate gas subjected to the column (V) performance parameters are calculated as follows.

$$\text{Molar } CO2\ adsorption = Ci - Co \qquad (I)$$

$$\text{Adsorption capacity (mol/kg)} = \frac{(Ci-Co)\times V}{wt.of\ adsorbent} \qquad (II)$$

$$CO2\ \text{Removal efficiency } (\eta\%) = \frac{(Ci-Co)}{Ci}\times 100 \qquad (II)$$

$$CO2\ \text{Recovery (\%)} = \frac{CO2\ desorbed}{CO2\ adsorbed}\times 100 \qquad (I)$$

VI. RESULTS AND DISCUSSIONS

This section deals with material characterization, evaluation of performance parameters of Zeolite 4A in terms of CO2 removal, removal efficiency, adsorption capacity and saturation values at variable pressure and time interval.

A. Material Characterisation data

The Zeolite belong to Na family and A group. The pore diameter of zeolite Na A is Å and hence it is commonly known as Zeolite 4A.Material characterization includes surface properties, porosity data and physical and chemical properties of selected material.(Table II) . Molecular structure of the selected zeolite is shown in Fig.3.
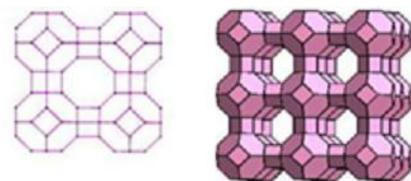

Fig 3. Molecular structure of Zeolite 4A

TABLE II. PROPERTIES OF ZEOLITE 4A

Sr.No	Parameter	Value with unit
	Physical properties	
1	Category of Zeolite	Na A
2	Particle size	4-5μ
3	Bulk density	450-500 kg/ m3
4	Pore diameter	4Å
5	Pore volume	0.2-0.4 cm3/g
	Chemical properties	
6	Sodium Oxide	18 % ± 1
7	Aluminium Oxide	29% ± 1
8	Silica	33% ± 1
9	Water	20% ± 2
10	Chemical formula	6Na2O 6Al2O3 12 SiO2 27 H2O

B. CO2 concentration at outlet(C_o)

The inlet concentration (C_i) of simulated flue gas is 0.15 mol (15% by volume). A molar fraction is adsorbed by the adsorbent. Table III summarizes the results of ORSAT analysis which depicts outlet concentrations (C_0) for 6 adsorption cycles. Fig 4 shows graphical presentation of outlet concentration of CO_2. The outlet concentration of CO_2 follows decreasing trend with time. The rapid decrease in the beginning is due to availability of large number of vacant adsorption sites. The further increase in outlet concentration indicates decrease in available adsorption sites. The point of inflection is observed after 10 minutes. The minimum outlet concentration of 10% is observed at 0.6kg/cm² (10 minutes) and 0.8 kg/ cm² (15 mins).

TABLE III. OUTLET CONCENTRATION OF CO_2 FOR 6 ADSORPTION CYCLES

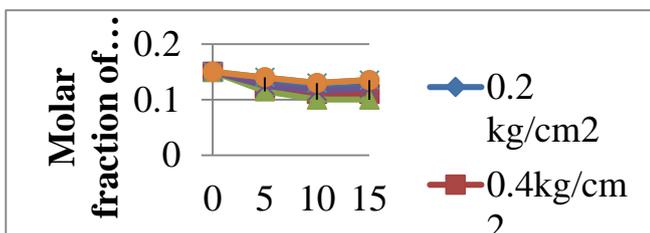| Time (min)	Molar fraction of CO2 at outlet (Co)					
	0.2 kg/cm²	0.4 kg/cm²	0.6 kg/cm²	0.8kg/ cm²	1.0 kg/cm²	1.2 kg/cm²
0	0.15	0.15	0.15	0.15	0.15	0.15
5	0.13	0.12	0.115	0.125	0.14	0.14
10	0.12	0.11	0.1	0.115	0.13	0.13
15	0.125	0.11	0.1	0.12	0.135	0.135

Fig.4 Molar outlet concentration of CO_2 at different operating pressures

data is used to calculate CO_2 removal efficiency for operational pressure range 0-1.2 kg/cm²(Fig 5). The CO_2 removal efficiency ranges from 6.67 to 33.33%. Maximum removal efficiency is obtained at 0.6 kg/cm².

TABLE IV. CO2 REMOVAL AT DIFFERENT PRESSURS

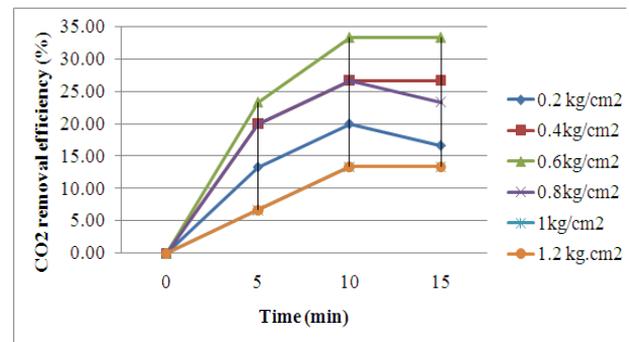| Pressure (kg/cm²)	Molar fraction of CO2 removed (Ci-Co)			
	O min	5 min	10 min	15 min
0.2	0	0.02	0.03	0.025
0.4	0	0.03	0.04	0.04
0.6	0	0.035	0.05	0.05
0.8	0	0.03	0.04	0.035
1.0	0	0.01	0.02	0.02
1.2	0	0.01	0.02	0.02

Fig.5 Removal efficiency of Zeolite 4A

D. CO2 adsorption potential

The adsorption cycle is continuous for 15 minutes the molar mass adsorption is calculated by cumulative addition of molar fractions for 5 minute interval. Fig. 6 indicates cumulative molar adsorption. The cumulative molar adsorption of CO2 indicates maximum molar adsorption at the end of each cycle. The highest molar adsorption of 0.175 mole is obtained at 0.6 kg/cm². An increase in molar CO_2 adsorption is observed with increase in operational pressure. Due to high pressure the gas strikes the adsorbent with higher velocity. This enhances the chances of the gas molecules to get adsorbed.
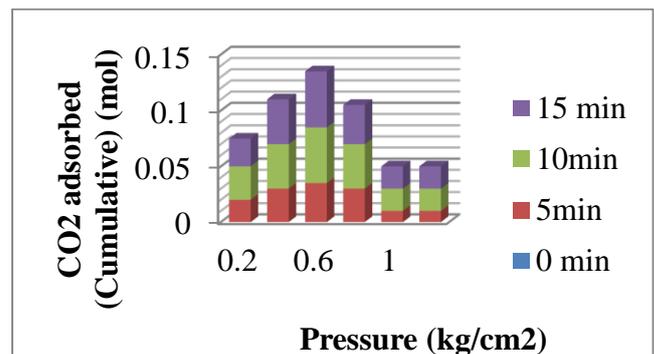

Fig. 6 Cumulative CO2 adsorption during adsorption period

However, The decrease in adsorption can be justified by increase in temperature with increase in pressure. Due to temperature increase the adsorbed gases tend to desorb.

Adsorption capacity of Zeolite 4A is obtained on volumetric basis. Table IV shows results of adsorption capacity obtained at pressure range of 0.0 to 1.2 kg/cm2. Fig 7 indicates that CO2 capture capacity ranges from 0.17 mol/kg to 2.5 mol/kg. Minimum and maximum capacities are observed at 1.2 kg/cm² and 0.6 kg/ cm² respectively.

TABLE V.   CO2 ADSORPTION CAPACITY AT

Pressure kg/ cm²	CO2 adsorption capacity (mol/kg)			
	0 min	5 min	10 min	15 min
0.2	0.00	0.33	1.00	1.25
0.4	0.00	0.50	1.33	2.00
0.6	0.00	0.58	1.67	2.50
0.8	0.00	0.50	1.33	1.75
1.0	0.00	0.17	0.67	1.00
1.2	0.00	0.17	0.67	1.00
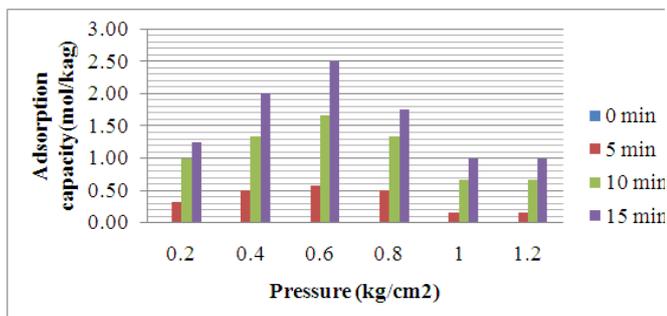
Fig 7. Variation of adsorption capacity with pressure

E. CO₂ recovery

Desorption is carried out at the end of the adsorption cycle. The cumulative CO_2 adsorbed in 15 minutes is compared with the quantity of CO_2 obtained during desorption. Fig. 8 shows results of CO2 recovered during desorption. CO2 recovery ranges from 65-76%. Maximum recovery is obtained from maximum CO2 adsorption.
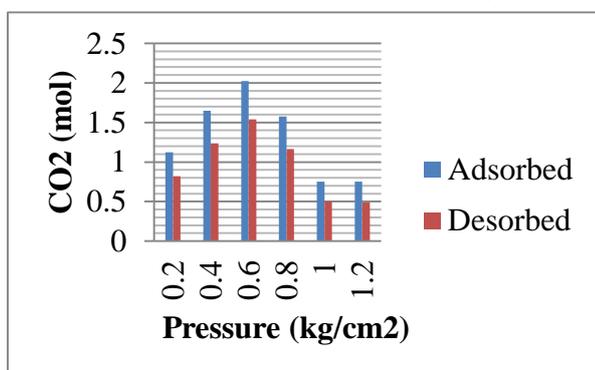

Fig10. Amount of CO2 adsorption and desorption

F. Temperature variation and adsorption performance

Adsorption process is associated with heat of adsorption. Hence an obvious increase in temperature is observed throughout the adsorption cycles. Fig 8 summarizes temperature observations during 6 adsorption cycle.
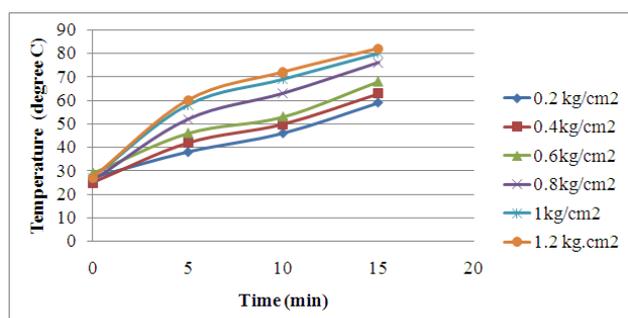

Fig 8. Temperature profiles during adsorption cycles

The decrease in adsorption can be justified by increase in temperature with increase in pressure. Due to temperature increase the adsorbed gases tend to desorb. This also justifies corresponding increase in outlet CO_2 concentration.

G. Effect of flow rate on adsorption

The effect of flow rate on CO_2 removal is assessed at constant pressure of 0.6 kg/cm² . The adsorption cycle is run for 10 minutes. Five adsorption cycles are conducted by flow varying the flow rate between 1-5 lpm. Fig 9 shows variation in CO_2 removal at variable flow rates. The bell shape curve indicates maximum CO_2 removal at 2.0 lpm. The declining trend for higher flow rate due to reduced pore volume due to progressive adsorption. Fig. 10 indicates increase in adsorption capacity with increase in flow rate. However the rate is increase reduces at higher flow rate indicating saturation flow rate.
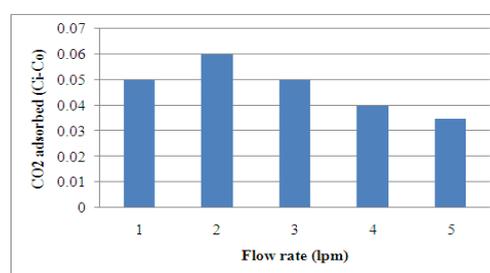

Fig. 9 Variation in CO2 removal with flow rate

VII. CONCLUSION

Adsorption is one of the promising mechanisms of carbon capture. The regenerable solid sorbent from Zeolite Na A family removes CO_2 through molecular sieving as well as adsorption in the narrow channels throughout the three dimensional cage like framework.

The adsorbent has a capture potential minimum of 0.17 mol/kg and maximum of 2.5 mol/kg. The minimum and maximum values are obtained at pressure of 1.2 and 0.6 kg/cm². The optimum working pressure is 0.6 kg/cm². Promising adsorption capacities are obtained in the temperature range of 60-80 ⁰C.

Removal efficiency of the zeolite 4A is obtained in the range of 6% to 33 %. The rise in temperature with time limits adsorption capacity and removal efficiency.

The recovery of CO2 ranges from 65-76% at constant flow of 1.0 lpm. Maximum removal of CO_2 is obtained at 2.0 lpm rate.

ACKNOWLEDGMENT

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