EXPERIMENTAL STUDIES ON RECOVERY OF CO2 FROM FLUE GASES BY USING PRESSURE SWING **ADSORPTION (PSA)**

A Project Work Submitted in the Partial Fulfillment of the Requirements For the Degree of

MASTER OF TECHNOLOGY

IN

REFINING & PETROCHEMICAL ENGG

Under Guidance of

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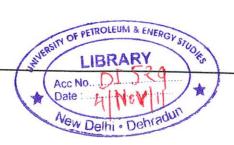


BY

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MAY 2007



CERTIFICATE

This is to certify that the project work entitled "Experimental Studies on recovery of CO₂ from flue gases by using Pressure Swing Adsorption(PSA) "submitted by "K Chaitanya Jonnavitula" in partial fulfillment of the requirements for the award of the degree of Master of Technology(Refining & Petrochemical Engineering), at College of Engineering, University of Petroleum & Energy Studies, is a record of the work carried out by him at Indian Institute of Petroleum(IIP), Dehradun under the guidance of "Dr.D.N.Saraf, Professor,COE,UPES" and "Dr.Amar N.Goswami, Scientist'F', Refining Technology Division, Indian Institute of Petroleum, Dehradun".

To the best of my knowledge, the contents of this project work did not form a basis of the award of any previous degree or published material by any one else.

The work is comprehensive of sufficient standard and here by recommended for the award of the degree of M.Tech in Refining & Petrochemical Engineering.

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CERTIFICATE

This is to certify that the project work entitled "Experimental Studies on recovery of CO₂ from flue gases by using Pressure Swing Adsorption(PSA) "was carried out by K Chaitanya Jonnavitula, M.Tech(Refining & Petrochemical Engineering), University of Petroleum & Energy Studies, Dehradun under our guidance of Dr.D.N.Saraf, Professor, College of Engineering, UPES and Dr.Amar N.Goswami, Scientist'F', Refining Technology Division, Indian Institute of Petroleum(IIP), Dehradun in the partial fulfillment for the degree of Master of Technology in Refining & Petrochemical Engineering. No part of this project has been submitted else where for any other degree.

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भारतीय पेट

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Date: 26th April 2007

TO WHOMSOEVER IT MAY CONCERN

This is to certify that the project titled "Experimental Studies on recovery of CO₂ from flue gases by using Pressure Swing Adsorption (PSA) "was carried out by K Chaitanya Jonnavitula, M.Tech (Refining & Petrochemical Engineering), University of Petroleum & Energy Studies, Dehradun under my guidance during the period March 2007 to April 2007 as partial fulfillment for the degree of 'Master of Technology in Refining & Petrochemical Engineering'. No part of this project has been submitted else where for any other degree.

Shri K Chaitanya has impressed as a sincere, hardworking student with deep interest in the science behind his project work. Within a short period he has been able to grasp the subject, gather a considerable amount of experimental data on CO2 recovery and give a reasonable interpretation to the results.

I wish him all success in the future

Dr Amar N.Goswami

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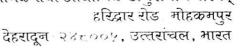
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TO WHOMSOEVER IT MAY CONCERN

This is to certify that Mr. K Chaitanya Jonnavitula, student of M.Tech. (Refining & Petrochemical Engg.), U.P.E.S., Dehradun has completed his vocational training from March 5 to April 26, 2007, both days inclusive in Refining Technology Division of this Institute. The topic on which he has worked is "EXPERIMENTAL STUDIES ON RECOVERY OF CO2 FROM FLUE GASES BY USING PRESSURE SWING ADSORPTION (PSA)". During this period, he has been punctual, sincere to his job and has undergone the learning process with responsibilities and sense of purpose.

This certificate has been issued to student for partial fulfillment of his degree.

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ABSTRACT

Typical flue gases from various industries using fossil fuels (steel, cement, power plants, etc) contain around 17% CO_2 , the balance being N_2 (79%) and O_2 (4%). CO_2 is a greenhouse gas and there are increasing concerns worldwide on its impact on global warming. Recovery or removal of CO_2 by separation processes plays a vital role in alleviating the problem of environmental pollution.

There are many technologies available for capturing of carbon dioxide from flue gases. Based on CO₂ solubilities in amines, most commonly used processes in commercial plants worldwide use amine absorption for CO₂ removal. However amine absorption processes have associated problems of corrosion and foaming. Membrane separation is also an alternative, but it is not commercially well established for flue gas application. Upcoming newer plants use polybed pressure swing adsorption processes. The PSA process for CO₂ recovery generally uses solid micro porous adsorbents like activated carbon, molecular sieves, etc. PSA technology for CO₂ recovery has been in commercial operation in Korea and Japan.

Commercial PSA processes generally employ multiple beds. Due to high equipment costs associated with multiple bed operation, the new trend is to develop single bed PSA processes. The present study describes the experimental work taken up to develop a single bed PSA process for removal of CO₂ from mixtures with nitrogen. The experiments were carried out in a single column PSA unit with PC based data acquisition and control and online gas analysis. To optimize purity and product recovery in a PSA cycle, N₂ Purge flow rates and cycle times were varied between 175-315 seconds. The results obtained from the experimental runs showed CO₂ in product gas could be reduced to 1 % from approximately 12% in the feed and a CO₂ recovery of above 90% could be obtained.

CHAPTER 1 LITERATURE REVIEW ON RECOVERY OF CO₂ FROM FLUE GASES

1. RECOVERY OF CO₂ FROM FLUE GASES

The recovery of CO₂ from flue gases emitted by power plants, steel mills, cement kilns, and fermentation processes is becoming increasingly viable. In its purified form, CO₂ has found many uses in the chemical industry. It can be used in solid form (dry ice), liquid form (e.g. refrigeration equipment) or gaseous form (e.g., for carbonated beverages, fire extinguishing equipment). It can also be used as a reactant in many important chemical reactions or as an inert blanketing gas to prevent oxidation (e.g., for food packaging). Today CO₂ is produced as a by-product of fermentation and lime-kiln operations and by separation from flue gases by gas-liquid absorption processes. Typical flue gases contain around 17 % CO₂, the balance being N₂ (79 %) and O₂(4 %). Trace amounts of SO₂ and NO, can also be found, but they are usually much less than 1% in total, K. T. Chue⁽⁵⁾.

Due to CO₂'s ability to trap solar radiation in the earth's atmosphere and the sheer quantity emitted, CO₂ is considered the principal greenhouse gas responsible for global warming by many in the scientific community. Approximately 7 giga (7 trillion) tons/year (tpy) of CO₂ is emitted worldwide and this is forecast to double between 2030 and 2050. Legislative action is anticipated to impose stricter emission standards, financial penalties and/or incentives to sequester CO₂ emissions.

CO₂ emissions sources include:

- Automobiles (> 500 million vehicles worldwide)
- Normal CO₂ content in natural gas (~2% to ~30%) and coal (~8% to ~20%)
- Coal/gas-based boilers/turbines in large industrial and power plants
- Large diesel/gas engine driven generators for remote areas
- Gas turbine-driven machinery, including compressors and generators in refineries, petrochemical and chemical facilities, oil and gas plants, offshore platforms, floating production units and marine transport

Currently, carbon credits are trading \sim \$30/ton.Values from \sim \$25/ton to \sim \$80/ton are quoted as the cost of CO₂ capture and sequestration. All the technologies necessary for CO₂ capture and sequestration are mature and have been in operation for more

than 30 years.CO₂ concentration in flue gases depend on fuel properties. Coal from various global sources has an 18% to 20% CO₂ content and ash content between 2% and 30% by weight. Many natural gas reservoirs have a CO₂ content ranging from 2% to 30%.Pipeline quality gas specifications typically limit CO₂ content to less than 3% with treatment or mixing often required to meet this criterion. This CO₂ content of the fuel has to be added to the equipment emissions in order to arrive at total plant emissions.

Economics for separating CO_2 from flue gases is affected by the amount of CO_2 in the fuel and the air-fuel ratio used in that particular equipment. This air fuel ratio is ~2:1 in coal-fired boilers and ~45:1 in gas turbine-driven generators. Depending on the plant configuration typically, ~30 %(~60% in simple cycle gas turbine/engine) or more fuel energy is lost through the stack. In coal or gas-fired power plants with heat recovery steam generation, exhaust flue gas temperatures, which typically range between ~175°c to ~220°c (> 400°c for gas turbines) depend on:

- ➤ Flue gas dew point. The exhaust gas temperature leaving the stack must be greater than the dew point temperature to prevent SO₂ and CO₂ forming corrosive acids combined with water vapor in the flue gas
- Minimum stack height required to meet local emission standards for dispersion including SO_x,NO_x and soot
- > Heat recovery process availability

Typical CO₂ separation processes also require pretreatment to remove SO₂ and soot precipitation, thus reducing the allowable flue gas dew point and permitting additional heat recovery from the exhaust stream. For example, in a flue gas with 3% oxygen content, reducing the sulfur content by 0.5% can reduce the flue gas dew point by 15°c-35°c.By removing soot and the heavier CO₂ molecule from the flue gas, the discharge temperature and stack height required to achieve a desired dispersion as per local environmental regulations is reduced, thereby supporting greater flue gas heat recovery, M.N.Saxena⁽¹¹⁾.

The sorption of pollutants on adsorbents is receiving increasing attention both from the view of removal and recovery of pollutants from gas mixtures, especially the ones produced through combustion processes. The generation and discharge of carbon dioxide into the atmosphere due to the consumption of large quantities of fossil fuels has emerged as a significant pollution problem for the environment. Thus studies are in progress increasingly to address this issue. Further, the removal of carbon dioxide

is also important in several gas purification operations, such as, the production of hydrogen gas, landfill and natural gas treatment and in the purification of hydrocarbons. Various separation techniques are applicable for the removal of carbon dioxide, such as, adsorption, absorption, membrane separation and other variants, Vincent G. Gomes⁽⁷⁾.

C0₂ recovery has been achieved by gas absorption employing solutions of carbonates and alkanolamines. However, this process is energy-intensive for regeneration of solvent and is also plagued by corrosion problems. Recently, the PSA (pressure swing adsorption) process treating high CO₂ concentration flue gases (25% CO₂) has become an alternative to the conventional absorption process. For the recovery of high purity CO₂ from low concentration flue gases (e.g., 15% CO₂), further work such as development of better adsorbents and more efficient PSA cycle is needed to improve the PSA performance.

Prior to the design of a PSA process, the choice of a preferred adsorbent is most important. The fundamental properties of an adsorbent such as selectivity, effective adsorption amount (or working capacity), mass transfer kinetics, and heat of adsorption should be included for consideration. The effective purge amount to be defined in the next section should be also considered when a PSA process involves a product purge step, Byung-Ki Na⁽⁴⁾.

In C0₂ bulk separation from low- and high-CO₂ concentration flue gases, AC (activated carbon), CMS (carbon molecular sieve), and synthetic zeolites are

Candidate adsorbents. PSA cycle for C0₂ separation from a low CO₂ concentration flue gas containing 17% CO₂ has been studied using activated carbon and carbon molecular sieve as the adsorbents, Kikkinides⁽⁸⁾. It was pointed out that the equilibrium selectivity for C0₂ in activated carbon dominates the PSA separation, and the kinetic selectivity in favor of CO₂ in carbon molecular sieve is less important. However, the wide-pore carbon molecular sieve (CMS-W) was successfully tested in pilot scale plant for recovery of C0₂ from flue gases containing 11% CO₂; over 98.9% CO₂ could be produced at over 53%-72% recovery, with energy consumption of 0.8-3.0 kW-h/kg CO₂ (Pilarczyk and Schroter, 1990). In general, zeolites have higher adsorption capacities for C0₂ as well as higher equilibrium selectivities for C0₂ over N₂ than activated carbon. On the other hand, the heat of adsorption of C0₂ on activated carbon is lower than on zeolite, so that the use of activated carbon in a PSA

process may result in less severe heat effect on the PSA performance. The temperature excursion due to heats of adsorption and desorption is detrimental to the separation performance of a PSA process (Cen and Yang, 1986; Lu et al., 1993). Pressure swing adsorption (PSA) is a process of substantial interest in gas processing industry due to its low energy requirements and cost advantages. PSA is well suited to the removal and subsequent recovery of CO2 from gases due to its ease of applicability over a relatively wide range of temperature and pressure conditions and its energy efficiency. PSA is a cyclic batch process where adsorption is carried out at a relatively higher pressure and desorption (regeneration) is accomplished at a lower pressure, generally using part of the product from the adsorption step. A key aspect in separating CO₂ is the identification of a suitable adsorbent. Although several types of adsorbents may be employed for the adsorption of CO2, an important factor for an efficient process is choosing an adsorbent that has strong affinity for carbon dioxide with good sorption capacity as well as desorption capability. However, data on efficient PSA separation of carbon dioxide with a suitable adsorbent in the literature is scarce, Y.Takamura⁽⁶⁾.

CHAPTER 2 INTRODUCTION

2. INTRODUCTION

2.1 ADSORPTION

Adsorption is a process that occurs when a gas or liquid or solute (called adsorbate) accumulates on the surface of a solid or more rarely a liquid (adsorbent), forming a molecular or atomic film (adsorbate). It is different from absorption, where a substance diffuses into a liquid or solid to form a "solution". The term sorption encompasses both processes, while desorption is the reverse process.

Adsorption is operative in most natural physical, biological, and chemical systems, and is widely used in industrial applications such as activated charcoal, synthetic resins and water purification. Adsorption, ion exchange and chromatography are sorption processes in which certain adsorptives are selectively transferred from the fluid phase to the surface of insoluble, rigid particles suspended in a vessel or packed in a column.

Similar to surface tension, adsorption is a consequence of surface energy. In a bulk material, all the bonding requirements (be they ionic, covalent or metallic) of the constituent atoms of the material are filled. But atoms on the (clean) surface experience a bond deficiency, because they are not wholly surrounded by other atoms. Thus it is energetically favorable for them to bond with whatever happens to be available. The exact nature of the bonding depends on the details of the species involved, but the adsorbed material is generally classified as exhibiting physisorption or chemisorption, McCabe⁽³⁾.

2.2 BREAKTHROUGH MEASUREMENTS

In planning new processes it is best to determine the break point and breakthrough curve for a particular system experimentally under conditions resembling as much as possible those expected in the process. Breakthrough experiments are performed to obtain reasonable adsorption time in a PSA process.

Consider a binary solution, either gas or liquid, containing a strongly adsorbed sloute. The fluid is to be passed continuously down through a relatively deep bed of adsorbent initially free of adsorbate. The uppermost layer of solid, in contact with the

strong solution entering, at first adsorbs solute rapidly and effectively, and what little solute is left in the solution is substantially all removed by the layers of solid in the lower part of bed. As solution continues to flow, the adsorption zone moves downward as a wave, at a rate ordinarily very much slower than the linear velocity of the fluid through the bed. At a later time, roughly half the bed is saturated with solute, but the effluent concentration is still substantially zero. At a certain time, the concentration of solute in effluent suddenly rises to appreciate value. Then the system is said to have reached the breakpoint.

The shape and time of appearance of the breakthrough curve greatly influence the method of operating a fixed bed adsorber. The curves generally have an S shape, but they may be steep or relatively flat and in some cases considerably distorted.

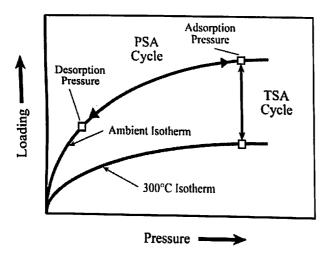
The actual rate and mechanism of the adsorption process, the nature of the adsorption equilibrium, the fluid velocity, the concentration of solute in the feed and the length of the adsorbed bed(Particularly if the solute concentration in the feed is high) all contribute to the shape of the curve produced for any system. Generally breakpoint time decreases with decreased bed height, increased particle size of adsorbent, increased rate of flow of fluid through bed and increased initial solute content of feed, Robert E.Treybal⁽²⁾.

2.3 PRESSURE SWING ADSORPTION

Pressure swing adsorption (PSA) is not a new process and, like most good inventions with the advantage of hindsight the principle appears obvious. As in all adsorption separation processes, the essential requirement is an adsorbent that preferentially adsorbs one component (or one family of related components) from a mixed feed. This selectivity may depend on a difference in adsorption equilibrium or on a difference in sorption rates(kinetic selectivity). In certain cases the difference in rates may be so great that the slower-diffusing species is in effect totally excluded from the adsorbent(size-selective sieving), and in this situation a very efficient separation can obviously be achieved.

All adsorption separation processes involve two principal steps:(1) adsorption, during which the preferentially adsorbed species are picked up from the feed;(2) regeneration or desorption, during which these species are removed from the adsorbent ,thus

"regenerating" the adsorbent for use in the next cycle. It is possible to obtain useful products from either the adsorption or regeneration steps or from both steps. The effluent during the adsorption step is purified "raffinate" product from which the preferentially adsorbed species have been removed. The desorbate that is recovered during the regeneration step contains the more strongly adsorbed species in concentrated form (relative to the feed) and is sometimes called the "extract" product. The essential feature of a PSA process is that, during the regeneration step, the preferentially adsorbed species are removed by reducing the total pressure, rather than by raising the temperature or purging with a displacing agent(although a low-pressure purge step is commonly included in the cycle). The process operates under approximately isothermal conditions so that the useful capacity is the difference in loading between two points, corresponding to the feed and regeneration pressures, on the same isotherm. The feed step is normally terminated before the more strongly adsorbed component breaks through the bed, while the regeneration step is generally terminated before the bed is fully desorbed. At cyclic steady state the profile therefore oscillates about a mean position in the bed.



A major advantage of PSA, relative to other types of adsorption process such as thermal swing, is that the pressure can be changed much more rapidly than the temperature, thus making it possible to operate a PSA process on much faster cycle, thereby increasing the throughput per unit of adsorbent bed volume. The major limitation is that PSA processes are restricted to components that are not too strongly adsorbed. If the preferentially adsorbed species is too strongly adsorbed, an

uneconomically high vacuum is required to effect desorption during the regeneration step.

General Features of a PSA Process

There are five general features of a PSA system that to a large extent explain both the advantages and limitations of the technology and hence determine the suitability for a given application:

- 1. **Product Purity**: The raffinate product (the less strongly adsorbed or slower diffusing species) can be recovered in very pure form, whereas the extract product (the more strongly adsorbed or faster-diffusing species) is generally discharged in impure form as a byproduct. Various modifications to the cycle are possible to allow recovery of the preferentially adsorbed species. However, these all add complexity to the cycle; so the process fits best where a pure raffinate product is required.
- 2. Yield or fractional recovery: In a PSA process, the fractional recovery (i.e., the fraction of the feed stream that is recovered as pure product) is generally relatively low compared with processes such as distillation, absorption, or extraction. The recovery can be increased by including additional steps in the cycle and by increasing the number of adsorbent beds, but both these modifications increase the capital cost. A PSA process therefore fits best when the feed is relatively cheap so that a high product yield is not a matter of primary concern.
- 3. Concentration of trace impurities: Where a highly selective adsorbent is available a PSA process can provide a valuable means of concentrating trace impurities, but this application has not yet been developed to any significant extent.
- 4. Energy Requirements: Like most separation processes, the energy efficiency of a PSA process is relatively low. The first law efficiency (separation work relative to energy consumed) is in fact comparable with that of processes such as distillation or extraction, but a PSA system uses mechanical energy, which is in general more expensive than heat. The power cost is the major component of the operating cost for a PSA system. However, if the feed is already available at high pressure, these costs may be greatly reduced. A PSA system is therefore especially useful where the feed is available at elevated pressure.

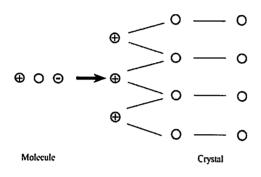
- 5. Scaling Characteristics: The operating costs of most separation processes increase approximately linearly with throughput. The capital cost of a PSA process is also approximately linear with throughput, but for most other processes the capital cost curve is highly nonlinear, with the incremental cost being smaller for the larger units. As a result, when the overall costs are considered, the economics tend to favor PSA at low to moderate throughputs and to favor other processes such as cryogenic distillation for very large-scale operations.
- 6. Pressure range: The term vacuum swing adsorption (VSA) is often used to denote a PSA cycle with desorption at subatmospheric pressure. This is a semantic choice. The performance of any PSA process is governed by the ratio of absolute (rather than gauge) pressures. That desorption at subatmospheric pressure often leads to improved performance is due to the form of the equilibrium isotherm rather than to any intrinsic effect of a vacuum.

PSA depend on physical adsorption rather than on chemisorption, since except for a few rather specialized applications, the capacities achievable in chemisorption systems are too small for an economic process. Since the adsorption forces depend on the nature of the adsorbing molecule as well as on the nature of the surface, different substances are adsorbed with different affinities. It is this "selectivity" that provides the basis for adsorption separation processes.

The role of adsorbent is to provide the surface area required for selective sorption of the preferentially adsorbed species. A high selectivity is the primary requirement, but a high capacity is also desirable since the capacity determines the size and therefore the cost of the adsorbent beds. to achieve a high capacity commercial adsorbents are made from micro porous materials. As a result the rate of adsorption or desorption is generally controlled by diffusion through the pore network, and such factors must be considered in the selection of an adsorbent and the choice of operating conditions .Certain materials (zeolites and carbon molecular sieves) that have very fine and uniformly sized micropores show significant differences in sorption rates as a result of steric hindrance to diffusion with in the micro-pores. Such adsorbents offer the possibility of achieving an

efficient kinetic separation based on differences in sorption rate rather than on differences in sorption equilibrium, D.M.Ruthven⁽¹⁾.

Adsorption of gaseous components on commercial adsorbents such as molecular sieves, silica gel, or activated carbon is determined by several factors, of which volatility and polarity are the most important. As process conditions approach the dew point of a component, a less-volatile component is adsorbed from the gas phase.



Adsorption is further aided by electrostatic forces. A strong preference by polar adsorbents is shown for polar or polarisable molecules. These molecules are attracted by the adsorptive forces of the positively charged cations contained in the crystal lattice of the adsorbent, J.Stocker⁽¹³⁾.

PSA processes may be categorized according to the nature of the adsorption selectivity (equilibrium or kinetic) and whether the less strongly (or less rapidly) adsorbed species (the raffinate product) or the more strongly (or more rapidly) adsorbed species (the extract product) is recovered at high purity, Geoffrey Q.Miller⁽¹⁴⁾.

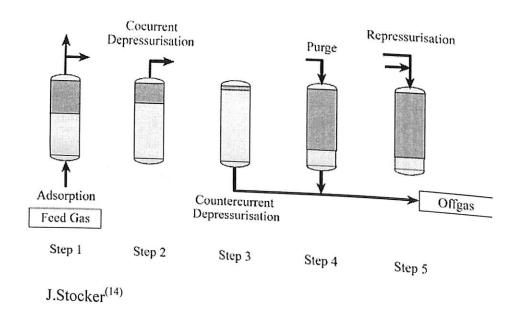
The PSA cycle

PSA can be operated on the basis of equilibrium, steric or kinetic selectivity. For separation based on equilibrium selectivity, the more strongly adsorbed components of a gas mixture are retained within the column, while the exiting high-pressure effluent contains the less strongly adsorbed species. On the other hand, in separation based on kinetic selectivity, the faster diffusing species is retained by the adsorbent and the high pressure product is concentrated in slower diffusing component. The optimum process variables and operating conditions to be used depend on the controlling mechanism for the process.

Any PSA cycle can be considered as a sequence of elementary steps, the most common of which are:

- 1. Pressurization (with feed or raffinate product);
- 2. High-Pressure feed with raffinate withdrawal;
- 3. Depressurization or "blow down" (cocurrent or countercurrent to the feed);
- 4. Desorption at the lower operating pressure; this may be accomplished by evacuation, purging the bed with the raffinate product or, in a kinetically controlled process, by slow equilibration with consequent evolution of the slower-diffusing sorbate;
- 5. Rinse (purging with the preferentially adsorbed species at high pressure following the adsorption step), D.M.Ruthven⁽¹⁾.

PSA Process Steps



To understand a PSA cycle properly it is necessary to know the way in which the concentration profile moves and changes shape during each of the elementary steps. Gas-phase concentration profiles in an adsorption column that undergoes in sequence pressurization, high-pressure adsorption, blow down and low-pressure desorption for equilibrium and kinetic separations. During pressurization the initial gas in the bed is pushed toward the closed product end, where it forms a plateau that is significantly enriched in the less strongly adsorbed species. In the high pressure step the concentration wave front travels down the column, and a raffinate product, enriched in the less strongly adsorbed species, is withdrawn at the product end. In the blow down and purge steps the concentration wave front is pushed back and a relatively clean initial bed condition is established for the next cycle, D.M.Ruthven⁽¹⁾.

CHAPTER 3 EXPERIMENTAL SYSTEMS & PROCEDURES

3. EXPERIMENTAL SYSTEMS & PROCEDURES

3.1 Apparatus and Materials

A schematic diagram of a single column PSA unit is shown in Figure 1. The adsorption column was a 64.4 cm long stainless steel pipe packed with adsorbent. All connecting lines were stainless steel tubing. Solenoid valves were installed to direct the flow in to and out of the column. These solenoid valves were controlled by a computer. Pressure in the bed was controlled by a back-pressure regulator and low pressure used was atmospheric. A pressure gauge at the top of the column was installed to acquire the pressure history of the adsorption column. The axial temperature was measured by using three temperature thermocouples. These temperatures as well as pressure data were recorded by a computer which was interfaced with an A/D converter, Dr.A.N.Goswami^(9,10,12).

Characteristics of adsorbent bed:

Adsorbent = Ba-Zeolite (IIP adsorbent)

Weight of adsorbent = 30 gm

Height of bottom support = 35 cm

Height of adsorbent bed = 10 cm

Height of top support = 19.4 cm

Internal diameter of column = 3.35 cm

Actual length of column = 64.4 cm

Bed diameter = 33.5 mm

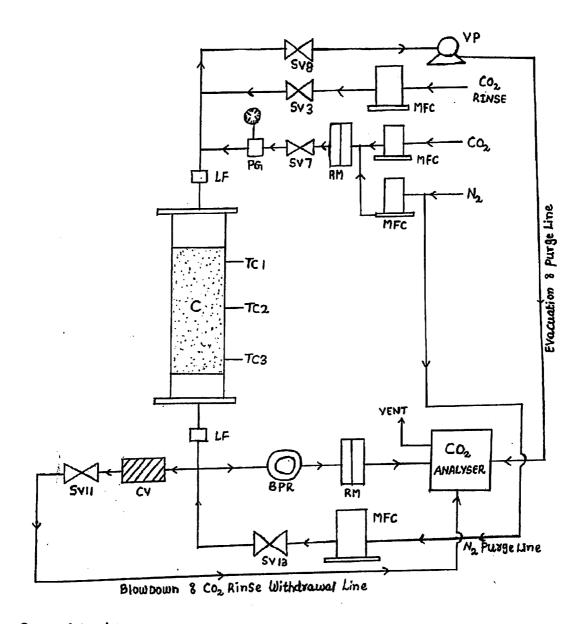
Bed volume = 88.15 cm^3

Bed Weight = 30 gm

Adsorbent packed density = 0.34 gm/cc

The initial feed flow rates are set by using Matheson mass flow controllers. The product flow rates in blown down, rinse withdrawal, evacuation, evacuation + purge are measured by using wet gas meter.

Fig.1 Schematic Diagram of PSA Unit



Sv -> Solenoid valve

MFC -> Mass Flow Controller

RM -> Rotameter

PG -> Pressure Guage

VP --> Vacuum Pump

LF -> Line filter

TC -> Thermocouple

CV -> Check Valve

BPR -> Back Pressure Regulator

C -> ADSORPTION COLUMN

3.2 Experimental Procedures

Moisture-free gas was used. The adsorbent was activated by heating it to a temperature $0f 150^{0}c$ for 2 hours.

3.2.1 Breakthrough Experiment

Before conducting breakthrough experiment, the adsorbent bed was evacuated with N_2 purge (In order to clean the bed completely). After, the adsorption bed was first pressurized up to the desired pressure (3 bar (Abs)) by feed gas (CO_2+N_2). Then, the sample was collected at the end of a column while the feed gas was continuously supplied. The amount of CO_2 adsorbed on adsorbent with respect to time was measured by using CO_2 analyzer. The breakthrough experiment was carried at a temperature 50^0 c.

The breakthrough experiment has been performed to obtain adsorption time in a PSA process.

3.2.2 Regeneration Experiment

Since the adsorption column was saturated with CO_2 , the adsorption bed was heated to 150° c temperature and was purged by N_2 gas followed by evacuation. The amount of CO_2 desorbed from the bed with respect to time is measured.

Regeneration experiment gives an approximate time required to regenerate completely the bed.

3.2.3 Single Column PSA Experiment

The procedure described below was for the separation of $CO_2/N_2(12/88 \text{ Wt \%})$ mixtures by the single column PSA experiment.

PSA experiments are carried out with purge cycle and without purge cycle strategies. The steps involved in the PSA experiments are

- STEP 1→ Pressurization, was initiated by opening the top valve (SV 7) as shown in Figure 1. and the bed was pressurized to the adsorption pressure, 3 bar (Abs). The desired column pressure was controlled by a pressure regulator.
- STEP 2→ Adsorption started when the top valve (SV 7) as shown in the figure 1 was Open. In the adsorption, as pressure reaches to desired pressure, feed gas Starts adsorbing on the adsorbent.
- STEP 3→ Blow down, was achieved by simultaneously opening the bottom valve (SV 11) as shown in the figure and closing the valve (SV 7) as shown in

the Figure 1.In this blown down step the pressure is suddenly decreased

- STEP $4 \rightarrow CO_2$ Pressurization, was started when the valve (SV 3) is open as shown in Figure 1 and remaining valves are kept closed.
- STEP $5 \rightarrow CO_2$ Rinse Withdrawal is achieved by opening the valve (SV 11) along With valve (SV 3) as shown in the figure 1.
- STEP 6→ Evacuation, is achieved by opening the valve (SV 8). Evacuation is created By using vacuum pump.
- STEP 7 \rightarrow Evacuation + Purge is achieved by opening valve (SV 8) and the valve (SV 13).N₂ gas was used as purge.

Few PSA experiments are carried out with the above 6 steps (Without Purge step→ Strategy 1) and remaining experiments are carried out with all the 7 steps (With Purge step→ Strategy 2).

The Valve sequence used in the process was summarized as

VALVE SEQUENCE									
STEPS	SV3	SV7	SV8	SV11	SV13				
FEED	CLOSED	OPEN	CLOSED	CLOSED	CLOSED				
PRESSURIZATION					CEOSED				
ADSORPTION	CLOSED	OPEN	CLOSED	CLOSED	CLOSED				
BLOW DOWN	CLOSED	CLOSED	CLOSED	OPEN	CLOSED				
CO_2	OPEN	CLOSED	CLOSED	CLOSED	CLOSED				
PRESSURIZATION									
CO ₂ RINSE	OPEN	CLOSED	CLOSED	OPEN	CLOSED				
WITHDRAWAL									
EVACUATION	CLOSED	CLOSED	OPEN	CLOSED	CLOSED				
EVACUATION +	CLOSED	CLOSED	OPEN	CLOSED	OPEN				
N ₂ PURGE									

After the every PSA experiment was done, the bed is completely regenerated at a temperature of 150^{0} c with N_{2} purge followed by evacuation in order to leave the bed with no traces of CO_{2} .

The amount of CO_2 (Wt %) in product, rinse withdrawal, blow down, evacuation, Evacuation + Purge steps was measured by using Carbon dioxide gas analyzer. The flow rates in Adsorption + Pressurization, Product, evacuation, evacuation + purge, rinse withdrawal and blow down steps was measured by using wet gas meter. The total by-pass flow was also measured by using wet gas meter.

The PSA experiments were designed to study the effects of purge flow rates and purge cycle time on CO_2/N_2 mixture. Different cycle times were used for CO_2/N_2 separation. The experimental conditions used are summarized as

Cycle No.→	Cycle1	Cycle2	Cycle 3	Cycle 4	Cycle 5	Cycle 6
Cycle Step 1	Time	Time	Time	Time	Time	Time
	(sec)	(sec)	(sec)	(sec)	(sec)	(sec)
Pressurization	5	5	5	5	5	5
Adsorption	150	150	80	80	80	80
Blow Down	5	5	5	5	5	5
CO_2	5	5	5	5	5	5
Pressurization						
CO ₂ Rinse	30	30	30	10	10	10
Evacuation	120	120	50	70	60	60
Evacuation +	X	X	X	X	10	10
Purge						
Parameter	-	Rinse	Temp	Rinse +	Addition	N ₂
Varied		Flow		Evac	Of	Purge
		Rate	1	Cycle	Purge	Flow
				Time	Step	rate
Temp(°C)	26	26	50	50	50	50

X-Cycle Step Not Used

Cycle No.→ Cycle Step	Cycle 7 Time (sec)	Cycle 8 Time (sec)	Cycle 9 Time (sec)	Cycle 10 Time (sec)	Cycle 11 Time (sec)	Cycle 12 Time (sec)
Pressurizati on	5	5	5	5	5	5
Adsorption	80	80	80	80	80	80
Blow Down	5	5	5	5	5	5
CO ₂ Pressurizati on	5	5	5	5	5	5
CO ₂ Rinse	10	10	10	10	10	10
Evacuation	60	60	60	60	60	60
Evacuation + Purge	10	20	30	40	60	40
Parameter Varied	N ₂ Purge Flow Rate	Purge Cycle Time	Purge Cycle Time	Purge Cycle Time	Purge Cycle Time + Purge Flow rate	Purge Cycle Time + Purge Flow rate
Temp(⁰ C)	50	50	50	50	50	50

The cycle step times greatly affected the performance of a PSA process such as purity and recovery.

RESULTS & DISCUSSIONS

3.3 RESULTS AND DISCUSSIONS

The main objective of the study was to understand the function of each step in a PSA process packed with the adsorbent which could eventually be used to optimize the PSA cycle for CO_2/N_2 separation. The main parameters that described the performance of a PSA process were purity as well as the recovery of CO_2 product.

3.3.1 Breakthrough Measurements

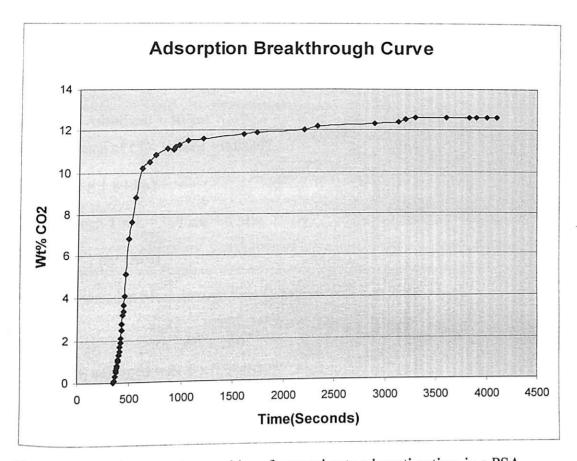
The adsorption bed was first pressurized up to the desired pressure (3 bar (Abs)) by feed gas (CO_2+N_2) . Then, the sample was collected at the end of a column while the feed gas was continuously supplied. The amount of CO_2 adsorbed on adsorbent with respect to time was measured by using CO_2 analyzer. The breakthrough experiment was carried at a temperature 50° c.

The breakthrough experiment has been performed to obtain adsorption time in a PSA process.

Channel Setting CH (CO₂) = 1(0.13 lpm) CH (N₂) = 15.3(1.97 lpm) Column Pressure = $1.98 \text{ Kg/cm}^2(\text{Abs})$

Feed flow = 1.8 NLPM Product flow = 0.5 NLPM Adsorp Temp = 24.4° c(Min) Feed Concentration = 12.6 Wt% CO₂ = 29.4° c(Max)

Time(Sec)	Wt% CO₂	Time(Sec)	Wt% CO
350	0	550	8.8
360	0.1	610	10.2
365	0.3	681	10.5
370	0.5	740	10.82
375	0.6	850	11.11
380	0.7	910	11.1
385	0.8	930	11.2
390	1	970	11.3
395	1.1	1050	11.5
400	1.3	1200	11.6
405	1.5	1600	11.8
410	1.7	1730	11.9
415	1.9	2200	12
420	2.1	2330	12.2
425	2.5	2900	12.3
430	2.8	3130	12.4
435	3.2	3200	12.5
440	3.4	3300	12.6
445	3.7	3600	12.6
450	4.1	3830	12.6
460	5.1	3900	12.6
490	6.8	4000	12.6
510	7.6		



The breakthrough curve gives an idea of approximate adsorption time in a PSA process.

CALCULATION FOR APPROXIMATE ADSORPTION LOADING CAPACITY AT BREAKTHROUGH TIME

Weight of Adsorbent = 30 gm

Mole Fraction of CO_2 in feed = 0.08403

Flow Rate = 1.8 LPM

Breakthrough Time = 360 sec = 6 Min

Pressure = 2 bar (Abs)

Vol.Feed passed at breakthrough time (6 Min) = 10.8 L at 2 bar

= 21.6 L at STP

Vol of CO_2 adsorbed = 21.6 x 0.08403

= 1.82 L

1.82 L at 1 atm & 2980K

At 1 atm & 273° K = 1.82 x 273

298

= 1.66 L

22.4 L at 1 atm,273°K contains 1 mole

At 1 atm, 273° K contains = 1.66/22.4 = 0.074 mol CO₂

30 gm adsorbs 0.074 moles CO₂

1 Kg of adsorbent adsorbs = $\underline{0.074 \times 1000}$

30

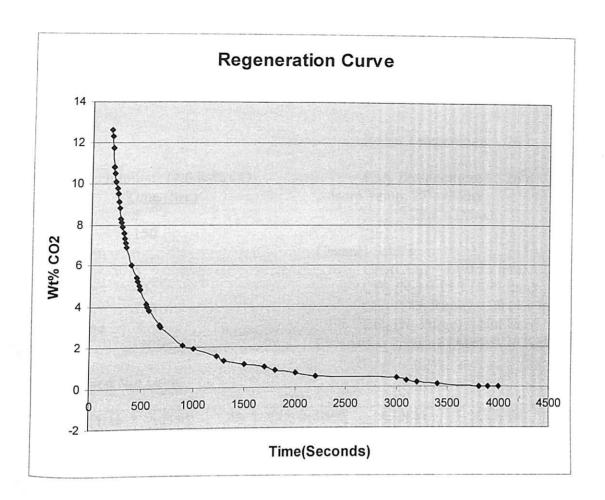
= 2.46 moles CO₂/ Kg of Adsorbent

3.3.2 REGENERATION ANALYSIS

After the breakthrough experiment was performed the bed is completely regenerated at a temperature of 150^{0} c with N_{2} purge followed by the evacuation.

Results and the plot analyses the amount (Wt %) of CO₂ desorbed from the bed with respect to time.

Time(Sec)	Wt% CO₂	Time(Sec)	Wt% CO
180	12.6	540	4.1
190	12.3	550	4
200	11.7	570	3.8
210	10.8	670	3.1
220	10.5	680	3
230	10.1	900	2.1
240	9.8	1000	1.9
250	9.5	1230	1.5
260	9.1	1300	1.3
270	8.8	1500	1.1
280	8.3	1700	1
290	8.1	1800	8.0
300	7.9	2000	0.7
310	7.6	2200	0.5
320	7.3	3000	0.4
330	7.1	3100	0.3
340	6.9	3200	0.2
390	6	3400	0.1
440	5.4	3815	0
450	5.2	3900	0
470	5	4000	0
480	4.8		



The regeneration curve gives the information on the maximum time required to regenerate the bed.

3.3.3 RESULTS OF SINGLE COLUMN PSA

The PSA Cycle experiments were carried out by varying different parameters. The following tables summarize the conditions and parameters varied in different cycles:

Room Temperature: 26°c

Run.No.1

 26° c Feed Concentration: 12.6 wt% CO₂ PSA Temperature: Adsorp Temp: 27^{0} c (Min) Time (Sec) Cycle $: 29.8^{\circ} c (Max)$ Press 5 150 Adsorp Channel Setting: Blow Down 5 $CH_1(CO_2) = 1 (0.13 lpm)$ CO₂ Press 5 $CH_2(N_2) = 15.3 (1.97 lpm)$ 30 CO₂ Rinse CH_3 (CO_2 Rinse) = 20 (4.1 lpm) Evac 120 CH_4 (N_2 Purge) = Not Used Evac+Purge X Evacuation Pressure (Abs): 0.26 Kg/cm² Total 315(Sec)

X-Cycle Step Not Used

Cycle No.	Feed Flow Lpm	Prod Flow Lpm	Adsorp Press Kg/cm ² (Abs)	CO ₂ In Prod Wt%	CO ₂ in Blow Down		Down			O ₂ in inse	CO ₂ Ev		CO ₂ Evac Pur	c+ ge
					Ti	Wt%	Ti	Wt%	Time	Wt%	Tim	Wt		
					me		me		(s)		е	%		
					(s)		(s)				(s)			
1	1.8	3.8	3.13	7.6	0	92	0	21	0	96.6				
2	1.8	4	3.13	10.4	5	21.5	5	80	10	82				
3	1.8	4	3.00	10.2			10	80.2	20	89				
4	1.8	4	3.00	8.7			15	87.9	30	91				
5	1.8	4	3.00	8.7			20_	93.1	40	92				
6	1.6	7	3.00				25	94.5	50	91				
7							30	95.3	60	88				
8									70	86				
9									80	82				
10									90	78				
11									100	76.5	_			
12									110	73.2				
13									120	71				
14														

Adsorp + Press Flow = 7.3 liters per 155 sec Product Flow = 9.8 liters per 150 sec Evacuation Prod Flow = 2 liters per 120 sec

Rinse Prod Flow = 5.4 liters per 30 sec

Blow down Prod Flow = 1 liter per 5 sec

Total By-Pass Flow: Partially Open = 5 lpm

 N_2 Purity (Wt %) = 91.3

Totally Open = 8 lpm

% CO₂ Recovery = 82.25

SINGLE COLUMN PSA EXPERIMENT

Run.No.2

Room Temperature: 26°c

Feed Concentration: 12.2 wt% CO₂

<u>Cycle Time (Sec)</u>

Press

PSA Temperature: 26°c

Adsorp Temp: 27°c (Min)

: 29.2°c (Max)

Adsorp 150
Blow Down 5 Channel Setting:

CO₂ Press 5 $CH_1(CO_2) = 1 (0.13 \text{ lpm})$ CO₂ Rinse 30 $CH_2 (N_2) = 15.1 (1.94 \text{ lpm})$ Evac 120 $CH_3 (CO_2 \text{ Rinse}) = 5 (0.95 \text{ lpm})$ Evac+Purge X $CH_4 (N_2 \text{ Purge}) = \text{Not Used}$ Total 315(Sec) Evacuation Pressure (Abs): 0.26 Kg/cm²

X-Cycle Step Not Used

<u> </u>	Υ				- 00	-	CO	in	CO	₂ in	CO ₂	in
Cycle	Feed	Prod	Adsorp	CO ₂		₂ in	1	₂ in			Evac	
No.	Flow	Flow	Press	In	Blow	Down	Kii	nse	EV	ac		
}	Lpm	Lpm	Kg/cm ²	Prod	}						Pur	ge
	•		(Abs)	Wt%		177.07	Time	337407	Time	Wt%	Tim	Wt
					Tim	Wt%	Tim	Wt%	Tim	W 170	'	
					e		e	ļ.	е		e	%
					(s)		(s)		(s)		(s)	
1	1.8	2.8	3.01	0.1	0	21	0	9.8	0	28.4		
2	1.8	4.4	3.25	7	5	22.3	5	15.5	5	66.7		
3	2	4.6	3.07	7.9			10	18.9	10	76.6		
4	2	4.6	3.05	8.8			15	20.7	20	84.9		
5		4.0	2.92	8.8			20	21.4	30	85.7		
6	1.8		3.06	8.8			25	21.8	40	85.4		
7	1.8	4	3.05	8.8			30	22.4	50	83.8		
8	1.8	4		8.8					60	81.2		
9	1.8	4	3.02	0.0					70	78.1		
								-	80	74.8		
10									90	71		
11	_								100	67.5		
12												
13									110	64.5		
14					1.6.6				120	61.2		لــــــا

Adsorp + Press Flow = 4.2 liters per 155 sec

Product Flow = 9.8 liters per 150 sec

Evacuation Prod Flow = 0.85 liters per 120 sec

Rinse Prod Flow = 1 liter per 30 sec

Blow down Prod Flow = 1 liter per 5 sec

Total By-Pass Flow: Partially Open = 5.9 lpm

Totally Open = 8 lpm

 N_2 Purity (Wt %) = 91.2

% CO₂ Recovery = 80.3

Run.No.3

Room Temperature: 26°c

Feed Concen	tration: 12.6 wt% CO ₂	PSA Temperature: 50°c
<u>Cycle</u>	Time (Sec)	Adsorp Temp: 47.4 ⁰ c (Min)
Press	5	: 50.4 ⁰ c (Max)
Adsorp	80	
Blow Down	5	Channel Setting:
CO ₂ Press	5	$CH_1(CO_2) = 0.8 (0.103 \text{ lpm})$
CO ₂ Rinse	30	$CH_2(N_2) = 15.6 (2 \text{ lpm})$
Evac	50	CH_3 (CO_2 Rinse) = 5 (0.95 lpm)
Evac+Purge	X	CH_4 (N_2 Purge) = Not Used
Total	175(Sec)	Evacuation Pressure (Abs): 0.35 Kg/cm ²

X-Cycle Step Not Used

Cycle No.	Feed Flow Lpm	Prod Flow Lpm	Adsorp Press Kg/cm ² (Abs)	CO ₂ In Prod Wt%	CO ₂ in Blow Down		CO ₂ in Rinse		CO ₂ in Evac		CO ₂ in Evac + Purge	
					Ti	Wt%	Tim	Wt%	Time	Wt%	Tim	Wt
					me		e		(s)		e (a)	%
ļ		_			(s)		(s)			00	(s)	
1	1.8	4	3.11	-Ve	0	22.9	0	24	0	0.9		
2	1.8	4	3.11	-Ve	5	24	5	21.2	5	0.8		-
3	1.8	4.2	3.12	11.5			10	21.7	10	56		
4	1.8	4.2	3.12	11.6			15	21.8	20	68.2		
5	1.8	2.2	3.12	13.2			20	22.5	30	65.3		
6	1.8	4.2	3.12	14.3			25	24	40	58.1		
7	1.8	4.2	3.12	14.3			30	24.9	50	50.1		
8	1.0	7.2										
9	-											
10												
11												
12												
13												
14												

Adsorp + Press Flow = 4 liters per 85 sec Product Flow = 4.1 liters per 80 sec Evacuation Prod Flow = 1.4 liters per 50 sec Rinse Prod Flow = 1.3 liters per 30 sec Blow down Prod Flow = 1 liter per 5 sec Total By-Pass Flow: Partially Open = 3.6 lpm

Totally Open = 8.1 lpm

 N_2 Purity (Wt %) = 85.7

% CO₂ Recovery = 77.3

Run.No.4

Room Temperature: 26°c

Feed Concentration: 12.3 wt% CO₂ 50° c PSA Temperature: Adsorp Temp: 46.7°c (Min) Time (Sec) Cycle $: 50.4^{\circ} c (Max)$ Press 5

Adsorp 80

Channel Setting: Blow Down 5 $CH_1(CO_2) = 0.8 (0.103 \text{ lpm})$ 5 CO₂ Press $CH_2(N_2) = 15.7 (2.02 lpm)$ CO₂ Rinse 10 CH_3 (CO_2 Rinse) = 5 (0.95 lpm) Evac 70 CH_4 (N_2 Purge) = Not Used Evac+Purge X Evacuation Pressure (Abs): 0.37 Kg/cm² Total 175(Sec)

X-Cycle Step Not Used

Cycle No.	Feed Flow Lpm	Prod Flow Lpm	Adsorp Press Kg/cm ² (Abs)	CO ₂ In Prod Wt%	CO ₂ in Blow Down		CO ₂ in Rinse		CO ₂ in Evac		CO ₂ in Evac + Purge	
					Ti me (s)	Wt%	Ti me (s)	Wt%	Time (s)	Wt%	Tim e (s)	Wt %
1	1.8	4.2	3.14	-Ve	0	9.5	0	10.4	0	6.2		
2	1.8	4.2	3.14	-Ve	5	10.4	5	12.6	5	66.3		
3	1.8	4.2	3.14	-Ve			10	14.3	10	66.4		
4	1.8	4.2	3.10	4					20	61.8		
5	1.8	4.2	3.12	7.9					30	52.8		
6	1.8	4.6	3.10	8.5					40	43.2		
7	1.8	4.6	3.13	8.8					50	34.4		
8	1.8	4.6	3.07	-					60	28.5		
9	1.8	4.6	3.07	8.5					70	25.9		
10	1.8	4.6	3.07	8.5								
11												
12												
13												
14												

Adsorp + Press Flow = 3.8 liters per 85 sec

Product Flow = 6.2 liters per 80 sec

Evacuation Prod Flow = 1.3 liters per 70 sec

Rinse Prod Flow = 0.4 liter per10 sec

Blow down Prod Flow = 0.9 liter per 5 sec

Total By-Pass Flow: Partially Open = 7.5 lpm

Totally Open = 8.2 lpm

 N_2 Purity (Wt %) = 91.5

 $\% CO_2 Recovery = 86.32$

Run.No.5

Room Temperature: 27°c

Feed Concent	tration: 12.2 wt% CO ₂	PSA Temperature: 50°c
Cycle	Time (Sec)	Adsorp Temp: 46.5°c (Min)
Press	5	: 50.2°c (Max)
Adsorp	80	GI 10 W
Blow Down	5	Channel Setting:
CO ₂ Press	5	$CH_1(CO_2) = 0.7 (0.09 \text{ lpm})$
CO ₂ Rinse	10	$CH_2(N_2) = 15.1 (1.94 \text{ lpm})$
Evac	60	CH_3 (CO ₂ Rinse) = 5 (0.95 lpm)
Evac+Purge	10	$CH_4 (N_2 \text{ Purge}) = 10 (2.05 \text{ lpm})$
Total	175(Sec)	Evacuation Pressure (Abs): 0.38 Kg/cm ²

Cycle No.	Feed Flow Lpm	Prod Flow Lpm	Adsor p Press	CO ₂ In Prod Wt%	CO ₂ in Blow Down		I.	CO ₂ in CO ₂ in Evac			CO ₂ in Evac + Purge	
			Kg/cm 2 (Abs)	, ,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	Ti me (s)	Wt%	Ti me (s)	Wt%	Time (s)	Wt%	Ti me (s)	Wt%
1	2.0	1.6	3.02	-Ve	0	29.5	0	17.1	0	7.6	0	40.2
2	2.2	4.6	3.03	-Ve	5	17.2	5	17.9	5	7.4	5	38.6
3	1.8	4	2.97	-Ve	_		10	18.2	10	57.5	10	37.5
4	1.8	4	2.97	-Ve					20	65.9		
5	1.8	4	2.98	3.6					30	62.1		
6	1.8	4	3	4.8					40	54.6		
7	1.8	4	2.96	6.0					50	47.6		
8	1.8	4	2.96	6.6					60	40.2		<u> </u>
9	1.8	4	2.97	6.7								
10	1.8	4	2.95	6.8								
11	1.8	4	2.95	6.9								
12	1.8	4	2.95	6.9								
13	1.0		2.,,									
14									<u> </u>			

Adsorp + Press Flow = 4 liters per 85 sec

Product Flow = 5.6 liters per 80 sec

Evacuation Prod Flow = 1.2 liters per 60 sec

Rinse Prod Flow = 0.35 liter per 10 sec

Blow down Prod Flow = 0.9 liter per 5 sec

Purge Prod Flow = 0.65 liter per 10 sec

Total By-Pass Flow: Partially Open = 7.7 lpm

Totally Open = 8.8 lpm

 N_2 Purity (Wt %) = 93.1

% CO₂ Recovery = 84.8

Run.No.6

Room Temperature: 27°c

 $50^{0}c$ PSA Temperature: Feed Concentration: 12.5 wt% CO₂ Adsorp Temp: 47.2°c (Min) Time (Sec) Cycle $: 50.6^{\circ} c (Max)$ 5 Press 80 Adsorp Channel Setting: Blow Down 5 $CH_1(CO_2) = 0.7 (0.09 \text{ lpm})$ 5 CO₂ Press $CH_2(N_2) = 15.4 (1.98 \text{ lpm})$ 10 CO₂ Rinse CH_3 (CO_2 Rinse) = 5 (0.95 lpm) 60 Evac CH_4 (N₂ Purge) = 20 (4.1 lpm) 10 Evac+Purge Evacuation Pressure (Abs): 0.39 Kg/cm² 175(Sec) Total

Cycle No.	Feed Flow Lpm	Prod Flow Lpm	Adsorp Press Kg/cm ² (Abs)	CO ₂ In Prod Wt%	CO ₂ in Blow Down		CO ₂ in Rinse		CO ₂ in Evac		CO ₂ in Evac + Purge	
					Ti me (s)	Wt%	Ti me (s)	Wt%	Time (s)	Wt%	Ti me (s)	Wt%
1	1.8	2.2	2.94	-Ve	0	3.1	0	9.6	0	0.6	5	38.3 40.8
2	1.8	1.8	3.09	-Ve	5	8.7	5	12.1	5	66.4 72	10	35.1
3	1.8	3.8	2.96	-Ve			10	13.4	10	69.4	10	33.1
4	1.8	3.6	3.10	-Ve					30	62.1		
5	1.8	3.6	3.10	0.7					40	52.9		
6	1.8	3.6	3.01	3					50	45.3		
7	1.8	3.6	2.97	4.2						38.3		_
8	1.8	3.6	3.01	4.3					60	36.3		
9	1.8	3.6	3.02	4.3								
10	1.8	3.6	2.98	4.3								
11	1.8	3.6	2.97	4.3								
12	1.8	3.6	2.97	4.3								
13	1.8	3.6	2.97	4.3								
14	1.8	3.6	2.94	4.3	05		<u> </u>			l		

Adsorp + Press Flow = 4.2 liters per 85 sec

Product Flow = 4.3 liters per 80 sec

Evacuation Prod Flow = 1 liters per 60 sec

Rinse Prod Flow = 0.4 liter per 10 sec

Blow down Prod Flow = 0.8 liter per 5 sec

Purge Prod Flow = 0.9 liter per 10 sec

Total By-Pass Flow: Partially Open =5.6 lpm

Totally Open = 7.85 lpm

 N_2 Purity (Wt %) = 95.7

% CO₂ Recovery = 90.73

Run.No.7

Room Temperature: 27°c

Feed Concen	tration: 12.2 wt% CO ₂	PSA Temperature: 50°c
<u>Cycle</u>	Time (Sec)	Adsorp Temp: 46.9 ⁰ c (Min)
Press	5	: 54.1 ⁰ c (Max)
Adsorp	80	
Blow Down	5	Channel Setting:
CO ₂ Press	5	$CH_1(CO_2) = 0.7 (0.09 \text{ lpm})$
CO ₂ Rinse	10	$CH_2(N_2) = 15.4 (1.98 \text{ lpm})$
Evac	60	CH_3 (CO_2 Rinse) = 5 (0.95 lpm)
Evac+Purge	10	CH_4 (N ₂ Purge) = 30 (6.3 lpm)
Total	175(Sec)	Evacuation Pressure (Abs): 0.38 Kg/cm ²

Cycle No.	Feed Flow Lpm	Prod Flow Lpm	Adsorp Press Kg/cm ² (Abs)	CO ₂ In Prod Wt%	CO ₂ in Blow Down		CO ₂ in Rinse		CO ₂ in Evac		CO ₂ in Evac + Purge	
1		ļ			Ti	Wt%	Ti	Wt%	Time	Wt%	Ti	Wt%
Ì					me		me		(s)	1	me	
					(s)		(s)				(s)	17.0
1	2.2	46	2.97	-Ve	0	19.5	0	12.9	0	24	0	45.2
2	1.8	3.6	3.09	-Ve	5	13	5	13.7	5	24.6	5	39.1
3	1.8	3.6	3.06	-Ve			10	14	10	73	10	30.8
4	1.8	3.6	3.03	-Ve					20	71.5		
5	1.8	3.6	3.04	0					30	68		
6	1.8	3.6	3.03	2					40	61.9		
7	1.8	3.6	3.04	3.6					50	56.1		
8	1.8	3.6	3.03	3.8					60	51.6		
9	1.8	3.6	3.03	4.2								
10	1.8	3.6	3.03	4.3								
11	1.8	3.6	3.03	4.3								
12	1.8	3.6	3.03	4.3								
13	1.8	3.6	3.03	4.3								
14					l							

Adsorp + Press Flow = 4.2 liters per 85 sec

Product Flow = 4.2 liters per 80 sec

Evacuation Prod Flow = 0.92 liters per 60 sec

Rinse Prod Flow = 0.2 liter per 10 sec

Blow down Prod Flow = 0.95 liter per 5 sec

Purge Prod Flow = 1.3 liters per 10 sec

Total By-Pass Flow: Partially Open = 6.5 lpm

Totally Open = 7.99 lpm

 N_2 Purity (Wt %) = 95.7

% CO₂ Recovery = 92

Run.No.8

Room Temperature: 26°c

Feed Concen	tration: 12.2 wt% CO ₂	PSA Temperature: 50°c
Cycle	Time (Sec)	Adsorp Temp: 46°c (Min)
Press	5	: 46.8°c (Max)
Adsorp	80	
Blow Down	5	Channel Setting:
CO ₂ Press	5	$CH_1(CO_2) = 0.8 (0.103 \text{ lpm})$
CO ₂ Rinse	10	$CH_2 (N_2) = 16 (2.06 \text{ lpm})$
Evac	60	CH_3 (CO_2 Rinse) = 5 (0.95 lpm)
Evac+Purge	20	CH_4 (N ₂ Purge) = 10 (2.05 lpm)
Total	185(Sec)	Evacuation Pressure (Abs): 0.38 Kg/cm ²

Cycle No.	Feed Flow Lpm	Prod Flow Lpm	Adsorp Press Kg/cm ² (Abs)	CO ₂ In Prod Wt%	CO ₂ in Blow Down		CO ₂ in Rinse		CO ₂ in Evac		CO ₂ in Evac + Purge	
					Ti	Wt%	Ti	Wt%	Time	Wt%	Ti	Wt%
					me		me		(s)		me	}
					(s)		(s)				(s)	
	1.8	4	2.87	-Ve	0	13.2	0	11.1	0	6.1	0	34
2	1.8	4	2.91	-Ve	5	11.1	5	11.6	5	67.7	10	33.7
3	1.8	4	2.91	-Ve			10	12.5	10	68	20	29.1
4	1.8	4	2.84	-Ve					20	63.4		
5	1.8	4	2.80	-Ve					30	56.4		
6	1.8	4	2.96	1.8					40	49.6		-
7	1.8	4	3	2.4					50	41.1		
8	1.8	4	2.98	3					60	34		
9	1.8	4	2.91	3.5								
10	1.8	4	2.87	3.6								
11	1.8	4	2.87	3.7								
12	1.8	4	2.87	3.7								
13												
14										1		

Adsorp + Press Flow = 4.6 liters per 85 sec

Product Flow = 5.5 liters per 80 sec

Evacuation Prod Flow = 1.1 liters per 60 sec

Rinse Prod Flow = 0.2 liter per 10 sec

Blow down Prod Flow = 0.8 liter per 5 sec

Purge Prod Flow = 1.75 liter per 20 sec

Total By-Pass Flow: Partially Open = 8 lpm

Totally Open = 8.3 lpm

 N_2 Purity (Wt %) = 96.3

% CO₂ Recovery = 92

Run.No.9

Room Temperature: 27°c

Feed Concen	tration: 12 wt% CO ₂	PSA Temperature: 50°c
Cycle Cycle	Time (Sec)	Adsorp Temp: 46.5°c (Min)
Press	5	: 50.4 ⁰ c (Max)
Adsorp	80	
Blow Down	5	Channel Setting:
CO ₂ Press	5	$CH_1(CO_2) = 0.8 (0.103 \text{ lpm})$
CO ₂ Rinse	10	$CH_2(N_2) = 15.2 (1.95 \text{ lpm})$
Evac	60	CH_3 (CO_2 Rinse) = 5 (0.95 lpm)
Evac+Purge	30	CH_4 (N ₂ Purge) = 10 (2.05 lpm) Evacuation Pressure (Abs): 0.38 Kg/cm ²
Total	195(Sec)	Evacuation Pressure (Abs): 0.38 Kg/cm ²

Cycle	Feed Flow	Prod Flow	Adsorp Press	CO ₂ In		₂ in Blow Down	1	O ₂ in inse	CO ₂ in Evac		CO ₂ in Evac +	
No.	Lpm	Lpm	Kg/cm ² (Abs)	Prod Wt%	Down						Purge	
					Ti	Wt%	Ti	Wt%	Time	Wt%	Ti	Wt%
					me		me		(s)		me	
					(s)_		(s)				(s)	
1	2.4	4.6	3.12	-Ve	0	43	0	11.3	0	57.2	0	35.6
2	2.4	4.6	3.12	-Ve	5	11.6	5	11.4	5	68.5	5	38.9
3	1.8	3.4	3.04	-Ve			10	12.1	10	68.7	10	37.3
4	2.4	3.6	3.06	-Ve					20	63.4	20	30.6
5	2.4	3.6	3.05	0.4					30	55.9	30	25.3
6	2.4	3.6	3.04	1.4					40	47.6		
7	1.8	4.2	3.05	1.9					50	40		
8	1.8	3.6	3	2.2					60	37.1		
9	1.8	3.6	3.03	2.5								
10	1.8	3.6	2.99	2.6								
11	1.8	3.6	2.98	2.6								
12	1.8	3.6	2.98	2.6								
13												
14												

Adsorp + Press Flow = 4.6 liters per 85 sec

Product Flow = 3.15 liters per 80 sec

Evacuation Prod Flow = 1 liters per 60 sec

Rinse Prod Flow = 0.35 liter per 10 sec

Blow down Prod Flow = 0.75 liter per 5 sec

Purge Prod Flow = 2.15 liters per 30 sec

Total By-Pass Flow: Partially Open = 7.5 lpm

Totally Open = 7.9 lpm

 N_2 Purity (Wt %) = 97.4

% CO₂ Recovery = 93.6

Run.No.10

Room Temperature: 28°c

Feed Concen	tration: 12.2 wt% CO ₂	PSA Temperature: 50°c
<u>Cycle</u>	Time (Sec)	Adsorp Temp: 46.2 ⁰ c (Min)
Press	5	: 49.5 ⁰ c (Max)
Adsorp	80	
Blow Down	5	Channel Setting:
CO ₂ Press	5	$CH_1(CO_2) = 0.8 (0.103 \text{ lpm})$
CO ₂ Rinse	10	$CH_2 (N_2) = 15.1 (1.94 \text{ lpm})$
Evac	60	CH_3 (CO_2 Rinse) = 5 (0.95 lpm)
Evac+Purge	40	$CH_4 (N_2 Purge) = 10 (2.05 lpm)$
Total	205(Sec)	Evacuation Pressure (Abs): 0.38 Kg/cm ²

Cycle No.	Feed Flow Lpm	Prod Flow Lpm	Adsorp Press Kg/cm ² (Abs)	CO ₂ In Prod Wt%	l .	₂ in Blow Down	l .	O ₂ in inse	CO ₂ Ev		Ev	O ₂ in vac + urge
					Ti	Wt%	Ti	Wt%	Time	Wt%	Ti	Wt%
					me		me		(s)		me	
					(s)_		(s)				(s)	20.0
1	1.8	3.4	3.01	-Ve	0	19.2	0	7.1	0	1	0	38.8
2	1.8	3.2	3.05	-Ve	5	7.3	5	7	5	69.1	5	39.8
3	1.6	3	3.16	-Ve			10	7.4	10	74.5	10	35.2
4	1.6	3	3.05	-Ve					20	72.6	20	28
5	1.6	3.2	3	-Ve					30	65.6	30	23.9
6	1.6	3.2	3.02	-Ve					40	56.1	40	22.1
7	1.8	3.2	2.97	-Ve					50	47.9		
8	1.8	3.2	2.99	0.2					60	41.5		
9	1.8	3.2	2.95	0.6								
10	1.8	3.2	2.90	0.8								
11		3.2	2.88	0.9								
12	1.8	3.2	2.91	0.9								
13	1.8		2.87	1.1								
	1.8	3.2		1.1								
14	1.8	3.2	2.85	1.1	06						•	

Adsorp + Press Flow = 3.8 liters per 85 sec

Product Flow = 3.55 liters per 80 sec

Evacuation Prod Flow = 0.90 liters per 60 sec

Rinse Prod Flow = 0.2 liter per 10 sec

Blow down Prod Flow = 0.7 liter per 5 sec

Purge Prod Flow = 2.7 liters per 40 sec

Total By-Pass Flow: Partially Open = 6.75 lpm

Totally Open = 7.8 lpm

 N_2 Purity (Wt %) = 98.9

% CO₂ Recovery = 93.5

Run.No.11

Room Temperature: 28°c

Feed Concen	tration: 12.1 wt% CO ₂	PSA Temperature: 50°c
Cycle	Time (Sec)	Adsorp Temp: 46.5°c (Min)
Press	5	: 50.4°c (Max)
Adsorp	80	
Blow Down	5	Channel Setting:
CO ₂ Press	5	$CH_1(CO_2) = 0.8 (0.103 \text{ lpm})$
CO ₂ Rinse	10	$CH_2(N_2) = 15.2 (1.95 \text{ lpm})$
Evac	60	CH_3 (CO_2 Rinse) = 5 (0.95 lpm)
Evac+Purge	60	CH_4 (N_2 Purge) = 5 (0.95 lpm)
Total	225(Sec)	Evacuation Pressure (Abs): 0.43 Kg/cm ²

Cycle No.	Feed Flow Lpm	Prod Flow Lpm	Adsorp Press Kg/cm ² (Abs)	CO ₂ In Prod Wt%	1	₂ in Blow Down		O ₂ in inse	CO ₂ Eva		Ev	O ₂ in vac + urge
					Ti	Wt%	Ti	Wt%	Time	Wt%	Ti	Wt%
					me		me		(s)		me	
					(s)		(s)				(s)	
1	1.8	4	3	-Ve	0	14.9	0	11.7	0	1.4	0	3.5
2	1.8	4	3.01	-Ve	5	12.1	5	12	5	60.9	5	4.7
3	1.8	4	3.05	-Ve			10	12.2	10	72.4	10	14.6
4	1.8	4	3.02	-Ve					20	72.6	20	39.1
5	1.8	4	2.91	0.8					30	67.6	30	38.3
6	1.8	4	2.88	2.2					40	59.2	40	35.6
7	1.8	4	2.85	2.6					50	52.3	50	31.9
8	1.8	4	2.83	2.6					60	43.5	60	29.3
9	1.8	4	2.97	2.6								
10	1.8	4	2.90	2.6								
11												
12												
13												
14												

Adsorp + Press Flow = 3.9 liters per 85 sec

Product Flow = 4.55 liters per 80 sec

Evacuation Prod Flow = 0.90 liters per 60 sec

Rinse Prod Flow = 0.2 liter per 10 sec

Blow down Prod Flow = 0.75 liter per 5 sec

Purge Prod Flow = 2.45 liters per 60 sec

Total By-Pass Flow: Partially Open = 7.85 lpm

Totally Open = 8 lpm

 N_2 Purity (Wt %) = 97.4

 $% CO_2 Recovery = 93.75$

Run.No.12

Room Temperature: 28°c

Feed Concen	tration: 12.1 wt% CO ₂	PSA Temperature: 50°c
<u>Cycle</u>	Time (Sec)	Adsorp Temp: 47.6 ⁰ c (Min)
Press	5	: 50.2 ⁰ c (Max)
Adsorp	80	
Blow Down	5	Channel Setting:
CO ₂ Press	5	$CH_1(CO_2) = 0.8 (0.103 \text{ lpm})$
CO ₂ Rinse	10	$CH_2(N_2) = 15.1 (1.94 \text{ lpm})$
Evac	60	CH_3 (CO_2 Rinse) = 5 (0.95 lpm)
Evac+Purge	40	CH_4 (N ₂ Purge) = 5 (0.95 lpm)
Total	205(Sec)	Evacuation Pressure (Abs): 0.43 Kg/cm ²

Cycle No.	Feed Flow Lpm	Prod Flow Lpm	Adsorp Press Kg/cm ² (Abs)	In Prod Wt%	í	₂ in Blow Down	1	O ₂ in inse	,		Ev	O ₂ in vac + urge
					Ti me (s)	Wt%	Ti me (s)	Wt%	Time (s)	Wt%	Ti me (s)	Wt%
1	2.4	4.8	3.04	-Ve	0	2.4	0	8.1	0	1.8	0	42.3
2	1.8	3.2	3.10	-Ve	5	6.3	5	10.3	5	66.8	5	43.0
3	1.8	3.2	3.14	-Ve			10	12.3	10	73.8	10	41.7
4	1.8	3.2	3.09	-Ve					20	73.8	20	38.2
5	1.8	3.2	3.09	-Ve					30	71.1	30	33.2
6	1.8	3.2	2.97	0.9					40	67.8	40	29.4
7	1.8	3.2	3.07	1.4					50	64.8		
8	1.8	3.6	3.01	2					60	45.2		
9	1.8	3.6	3.01	2								
10	1.8	3.6	2.91	2.4								
11	1.8	3.2	2.85	2.2								
12	1.8	4	2.82	2.2								
13												
14												

Adsorp + Press Flow = 1.7 liters per 85 sec

Product Flow = 5 liters per 80 sec

Evacuation Prod Flow = 1 liter per 60 sec

Rinse Prod Flow = 0.25 liter per 10 sec

Blow down Prod Flow = 0.8 liter per 5 sec

Purge Prod Flow = 1.8 liters per 40 sec

Total By-Pass Flow: Partially Open = 7.75 lpm

Totally Open = 7.9 lpm

 N_2 Purity (Wt %) = 97.8

% CO₂ Recovery = 85.6

MODEL CALCULATIONS FOR FINDING % OF CO2 RECOVERY

	WT % CO ₂	WT % N ₂	MOL.FRAC.CO ₂
FEED	12.2	87.8	0.08124
PRODUCT	1.1	98.9	0.00702
RINSE	7.1	92.9	0.04637
WITHDRAWAL			
BLOW DOWN	7.3	92.7	0.0477
EVACUATION	60.36	39.64	0.4921
EVAC + PURGE	24.3	75.7	0.16962

 CO_2 in Rinse withdrawal Input = 0.95 x 0.16

$$= 0.152$$

Feed Input = Total bypass flow - Bypass during Adsorption

$$= 7.8 - 3.8$$

 CO_2 in feed Input = 4 x 0.08124

$$= 0.32$$

Total CO_2 Input = CO_2 in feed Input + CO_2 in Rinse withdrawal Input

$$= 0.32 + 0.152$$

$$= 0.472$$

CO₂ in evacuation Output = Evacuation product flow x CO₂ in evacuation

$$= 0.90 \times 0.49$$

$$= 0.441$$

% CO_2 Recovery = CO_2 in evacuation output

$$= 0.441$$

$$= 0.935 \times 100$$

$$= 93.5\%$$

A total of 12 experimental runs were carried out in order to optimize PSA cycle and to get better purity and recovery. The initial four PSA cycles were carried out without purge step and remaining eight PSA cycles are carried out with purge step.

During the study of PSA cycle, mainly three parameters were studied. There are

- Product Purity
- Effect of Purge flow rate on N₂ purity and CO₂ recovery
- Effect of Purge Cycle time on N₂ purity and CO₂ recovery

PRODUCT PURITY

It has been investigated that under the following conditions, a maximum N_2 purity of 98.9 % (Wt %) is achieved.

Feed Concent	tration: 12.2 wt% CO ₂ Time (Sec)	PSA Temperature: 50°c Adsorp Temp: 46.2°c (Min) : 49.5°c (Max)
Press	5	. 49.5 C (Wax)
Adsorp	80	Channel Setting:
Blow Down	5	$CH_1(CO_2) = 0.8 (0.103 \text{ lpm})$
CO ₂ Press	5	$CH_1(CO_2) = 0.8 (0.105 \text{ lpm})$ $CH_2 (N_2) = 15.1 (1.94 \text{ lpm})$
CO ₂ Rinse	10	$CH_2(N_2) = 13.11(1.54 \text{ lpm})$ $CH_3(CO_2 \text{ Rinse}) = 5 (0.95 \text{ lpm})$
Evac	60	$CH_4 (N_2 \text{ Purge}) = 10 (2.05 \text{ lpm})_2$
Evac+Purge	40	Evacuation Pressure (Abs): 0.38 Kg/cm ²
Total	205(Sec)	Dydoudion 1 1000010 (1100). 010 0 1-8 4

 CO_2 in Product = 1.1 Wt%

 N_2 Purity = 98.9 %(Wt %)

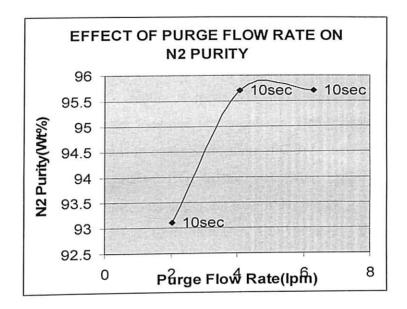
EFFECT OF PURGE FLOW RATE ON N2 PURITY AND CO2 RECOVERY

The effect of flow rate of purge on N_2 purity and CO_2 recovery was investigated. Purge flow rates are varied between 2-6 lpm and purge cycle time (10 seconds) is kept constant. Figure-2-a and 2-b shows the effect of purge flow rate on N_2 purity and CO_2 recovery. It is observed from the figure that as the purge flow rate is increased, N_2 purity increases and CO_2 concentration decreases in the product. The reason for decrease in CO_2 is, as purge flow rate increases column is more efficiently regenerated and mass transfer front is shifted towards the feed end of the column. Better regeneration and shifting of mass transfer front leads to better adsorption of CO_2 in a column i.e. more CO_2 removal and hence purer product (N_2) in the succeeding cycle.

It is observed that as purge flow rate increases, CO₂ recovery also increases gradually.

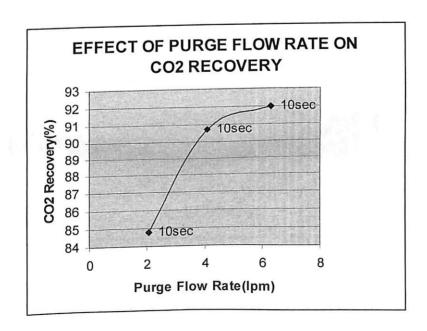
Purge Flow	N ₂ Purity
rate(lpm)	(Wt %)
2.05	93.1
4.1	95.7
6.3	95.7

Fig-2-a



CO_2
Recovery (%)
84.8
90.73
92

Fig-2-b

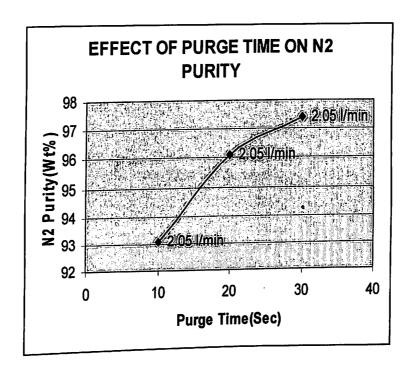


EFFECT OF PURGE TIME ON N2 PURITY AND CO2 RECOVERY

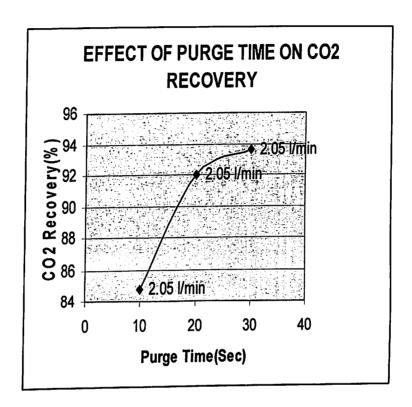
The effect of purge time on N₂ purity and CO₂ recovery was investigated. Purge cycle times are varied between 10-30 Seconds and purge flow rate (2.05 l/min) is kept constant. Figure-3-a and 3-b shows the effect of purge time on N₂ purity and CO₂ recovery. It is observed from the figure that as the purge time is increased,N₂ purity increases and CO₂ concentration decreases in the product. The reason for decrease in CO₂ is, as purge time increases column is more efficiently regenerated and mass transfer front is shifted towards the feed end of the column. Better regeneration and shifting of mass transfer front leads to better adsorption of CO₂ in a column i.e. more CO₂ removal and hence purer product (N₂) in the succeeding cycle. It has been investigated that purge time also plays an important role on N₂ purity.

It has been observed that, as purge time increases, CO₂ recovery also increases gradually.

Purge	N ₂ Purity
Time(Sec)	(Wt %)
10	93.1
20	96.3
30	97.4



Purge	CO ₂ Recovery
Time(Sec)	(%)
10	84.8
20	92
30	93.6



It has been observed that, increase in purge cycle time rather than purge flow, there is more N_2 Purity as well as CO_2 Recovery. So purge cycle time plays a major role on N_2 Purity as well as on CO_2 Recovery.

CONCLUSIONS

CONCLUSIONS

Pressure Swing Adsorption (PSA) can be used to separate CO₂ from mixtures with nitrogen. The process can be carried out with a single column containing adsorbent. Among the various cycles studied, optimum results are obtained when a vacuum is used to assist the purging of the column.

To optimize purity and product recovery in a PSA cycle, N₂ Purge flow rates and cycle times were varied between 175-315 seconds. The results obtained from the experimental runs showed CO₂ in product gas are reduced to 1 % from approximately 12% in the feed i.e., N₂ purities of 99% and a CO₂ recovery of above 90% are obtained.

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