An Insight into Investigations on Intensification of Adsorbent Beds

Sunil Kulkarni[1,*], Ajaygiri Goswami[2]

[1]Chemical Engineering Department, Gharda Institute of Technology, Khed, Maharashtra 415708, India
[2]Chemical Engineering Department, K.B.C. North Maharashtra University, Jalgaon, Maharashtra 425001, India

***Corresponding author: E-mail: suniljayantkulkarni@gmail.com**

DOI: 10.5185/amp.2020.040411

Many pollutants like acid gases, organic gases and inorganic gases are removed from gas mixtures by using adsorption. Regeneration of the adsorbent bed is an important aspect of successful adsorption technology. Adsorption-desorption cycle is governed by changes in pressure or temperature. These methods are termed as pressure swing (PSA) and temperature swing adsorption (TSA). For intensification of the process, it is envisaged to have an equal duration of the adsorption and desorption phases. Temperature swing adsorption (TSA) process can be intensified by employing several methods such as thermal conductivity promoter, thermoelectric elements and cyclic operating mode. Poor conductivity of the adsorbents is also one of the reasons for extended cycle time. Use of conducting materials in the form of composite fins for increasing the heat transfer through porous beds can reduce the adsorption-desorption cycle time. Also use of electrothermal swing adsorption (ESA) with thermoelectric element can intensify the adsorption-desorption process. Thermoelectric element converts current to temperature and vice versa. When there is difference in temperature, electric current of proportional magnitude is generated. Pressure swing (PSA) technology can also be intensified by using process engineering tools. Circulation fluidized beds can be used for dehumidification and also adsorption of volatile organic compounds (VOCs). Uniform bed temperature and mass transfer are advantages of circulation fluidized beds. In this paper, investigations on intensification of adsorption beds are discussed.

Introduction

Adsorption is one of the most important separation methods for solid-fluid systems. Fixed bed adsorption is commonly used contacting pattern for adsorption. Fixed bed adsorption is unsteady state method. The development of adsorption wave and its movement in downward direction are highlights of this adsorption processes. Purification of air, recovery of carbon dioxide from flue gases, recovery of sulphur oxides are few applications of adsorption. Regeneration of adsorbent bed and desorption of solute from adsorbent bed is very important for economical and environment friendly operation. Temperature swing adsorption (TSA) involves manipulation of temperature for adsorption and desorption. This is a cyclic process. In the simplest two bed operation, one bed is used for adsorption while other is regenerated. After one cycle, second bed is used for adsorption and first is regenerated. Poor heat conductivity of the materials is limitation of this method. Longer cycle times are required as more time is required for heating and cooling the bed. In pressure swing adsorption, pressurization and depressurization are used for adsorption and desorption of solute. Pressure swing adsorption is energy insensitive method. Loss of adsorbent capacity and low purity can be overcome by using hybrid method with combination of both, pressure and temperature swing methods. Also, investigations are reported on methods to increase the conductivity of porous materials and use of thermoelectric heating and cooling methods for rapid heating and cooling of the beds. This paper sheds light on various investigations aimed at intensification of pressure and temperature swing adsorption operations. Also, selection and preparation of adsorbent material is discussed. The important parameters for a packed bed are solute, feed rate, height, pH, particle size of adsorbent and temperature [**1**].

Pressure swing adsorption

Pressure swing adsorption and temperature swing adsorption are cyclic methods for fixed bed adsorption. Efficiency of an operation depends on the ease with which the regeneration or desorption can be facilitated. Pressure, temperature and valve coefficient play important role in pressure and temperature swing adsorption operation. Sharma *et al.*, investigated pressure swing adsorption of carbon dioxide [2]. They optimized the process of carbon dioxide synthesis from gas flow stream. For producing high purity carbon dioxide stream, they proposed compression followed by condensation of the carbon dioxide stream from pressure swing adsorption system. Long term storage of carbon dioxide needs supercritical conditions. This method meets that purpose also. All the process parameters were optimized for the pressure swing adsorption system in this work. They used a software to optimize the process. They pressurized the product gases to moderate pressure and then condensed them and ensured that the gases coming out of the condenser have same composition as that of the feed. This reduced the computation time considerably. Their adsorption model assumed isothermal conditions with axial plug flow. By decreasing the product flow, adsorption can be increased even at constant blowdown up to specific energy region of 5.2 (kWh/ kmol CO_2). After this, increase in CO_2 capture becomes insignificant. Decrease in blowdown pressure has positive effect on the CO_2 capture for pressure less than 3 bar (a) [**2**].

An investigation was carried out by Ko *et al.*, for sequestration of carbon dioxide from the gas mixture [3,4]. The steps in pressure swing adsorption are, pressurization, adsorption, depressurization and desorption. They used dynamic simulation for optimizing the operating parameters. In this work, they obtained optimum parameters for the cyclic operation. In another investigation, Ko *et al.*, optimized cyclic adsorption operation for the gases containing carbon dioxide and nitrogen. They used pressure swing adsorption and fractionated vacuum swing adsorption [**5**]. They carried of experimental work to increase purity of carbon and nitrogen in the product gases. They carried out experimental work on high temperature fractional vacuum swing adsorption for increasing purity of carbon dioxide. Determination of cyclic steady state was the objective of their investigation. The third step in conventional pressure swing adsorption, depressurization was replaced by the product purge step. In this step nitrogen was purged at moderate pressure of 101.325 kPa. In the last step, counter current blowdown for regeneration at sub-atmospheric pressure condition is facilitated. There is no need for longer beds for PSA optimization. According to them, Longer beds are advisable if simultaneous recovery of nitrogen and carbon dioxide is envisaged. For nitrogen purification, according to this research, contact time is more important than the feed pressure at normal temperature. However, at very high temperature, increase in contact time and pressure have adverse effect on nitrogen purity. According to them, reduction in the pressure during regeneration can improve carbon dioxide regeneration. They concluded that fractional vacuum pressure swing adsorption with 95 percent carbon dioxide purity and 99 percent nitrogen purity is much better than pressure swing adsorption. The carbon dioxide recovery was however slightly lower than the pressure swing adsorption. Nitrogen recovery of this process was higher by about 15 percent than the pressure swing adsorption.

Parameters, namely cycle time, feed pressure, feed to purge ratio and velocity of flow are important parameters for optimization in pressure swing adsorption [**6**]. Adsorption is also used for separation of moisture and other impurities from air. In cryogenic operations, removal of these components is very important. Wright *et al.*, carried out investigation on advanced thermal distillation operation for purification of air [**7**]. Adsorption is accompanied by heat generation. The heat pulse is generated at adsorption wave and it moves down with the wave. It is envisaged to start the regeneration before the pulse leaves the column. The heat required for regeneration of the column is equal to amount of heat liberated during desorption. In temperature swing, the temperature required for desorption is 150 to 200ºC. Pressure swing adsorption overcomes this disadvantage. However, it has its own limitations like switch losses and transient variation in flows. Thermally enhanced pressure swing adsorption was proposed in his research by Wright *et al.*, [**7**]. Also, in this work, attempt was done to overcome the disadvantages of pressure and temperature swing adsorption. Pressure swing system was supplied with mild thermal pulse. This lowers switch losses and increases economy. In another modification of pressure swing adsorption, namely temperature pressure swing adsorption, some adsorbate was removed by using unheated regeneration gas. In this process, lower heat pulse was used and temperature needed was below 100 ºC only against 150ºC for temperature swing adsorption. It is then followed by removal of remaining adsorbate by using inert career gas. Power requirement was 25 percent less in Temperature pressure swing adsorption than Temperature swing adsorption [**7**].

Grande studied advancements in pressure swing adsorption process for its use in gas separation [**8**]. According to him, the most important aspect of pressure swing adsorption is development of strategies for reducing cycle time. Tools namely gPROMS, PROSIM, ASPEN, COMSOL have yielded good results in investigations [**9-11**]. Potential use of the pressure swing adsorption for amines needs more fundamental and experimental studies.

Use evolutionary algorithms (EAs) is one of the effective methods for optimization of pressure swing adsorption [**9**]. Advantage of this method is the fact that, these are cheaper than computational methods. Beck proposed a method based on Kriging surrogate model [**12**]. In their investigation, Grande *et al.*, treated a mixture of 15 percent carbon dioxide and 85 percent nitrogen in pressure swing adsorption [**13**]. In their work, they used activated carbon and zeolite-based adsorbents. They used two cycle configurations. In one, they used usual four step process including pressurization, feed, blowdown and purge and in second one, 5-step cycle with pressurization, feed, rinse, blowdown and purge. In rinse step, a part of carbon dioxide was compressed and recycled. By using this method, carbon dioxide purity was increased to 80 percent from 65 to 70 percent in four step process. Many studies of pressure swing adsorption are directed towards carbon dioxide recovery [**14,15**]. Carbon dioxide capture and its storage can have significant effect on climate change [**15**]. Large amount of carbon dioxide emitting from power plants, distilleries, boilers cause severe effect on climate. Pressure swing adsorption has potential to solve this carbon dioxide capture problem. According to Warmuzinski, because of low concentration of carbon dioxide in flue gases, it is difficult to get high recovery and high concentration of carbon dioxide in the product [**16**]. A two-stage process with adsorption or membrane separation can be used for adequate results. Hybrid method consisting of adsorption and membrane separation can overcome disadvantages of these two methods, when used separately. Carbon dioxide concentration can be increased from average 65 percent after two stage adsorptions to average 95 percent after membrane process. Aspen plus modelling can be used for modelling and optimization of adsorbent beds [**17**].

For hydrogen production in shift converters, pressure and vacuum swing adsorption methods are used [**18**]. The shift gases are available at high pressure which aids the pressure swing method. This method has disadvantage in terms of high purity carbon dioxide capture. Purity of 99 percent for hydrogen and 96 percent for carbon dioxide can be obtained by using superstructure optimization. In this, various connections to the beds were manipulated to give optimized conditions. Many investigators have proposed reduced order models, surrogate models, nonlinear equations for optimization, finite volume methods for modelling and simulation of adsorbent beds [**19-21**]. Reduced order modelling has found wide acceptance among investigators for simulation and optimization of adsorption beds [**22,23**]. This model meets stringent boundary conditions while maximizing the product purity. It helps to overcome the computational challenge arising through differential algebraic equations. Most of the models consider finite mass transfer rate [**23**]. Heat effect, flow velocity, pressure drop, heat generations are other parameters considered by researchers for modelling.

Oxygen production from air was investigated by Biyani *et al.*, [**24**]. They carried out parametric study of the process. For zeolite-based catalyst beds, they used software simulation. They observed that, with pressure, there was increase in purity and recovery. According to these studies optimum pressure for this process is 2 to 3 atmospheres. At above optimum pressure, there is not considerable increase in purity and recovery compared to operating cost. 40 percent of the cycle time was required for adsorption and purging and 60 percent for blowdown and pressurization. Higher feed rate adversely affects the purity according to this research. It has more adverse effect on the recovery. By using this method more than 95 percent purity and 75 percent recovery was obtained. Hybrid processes combining pressure swing and cryogenic processes are also effective. Pressure swing adsorption can be combined with membrane separation for more effective results [25-28]. Ho *et al.*, used sensitivity analysis for reducing carbon dioxide capture cost [**29]**. Additional steps like pressure equalization, depressurization can reduce the cost of pressure swing adsorption. Many combinations of the steps and their sequences are proposed by various investigators [30-32]. Improved adsorption system proposed by Ho *et al.*, consists of seven steps [29]. These steps are pressurization, adsorption, depressurization, pressure equalization, purging, pressure reduction and evacuation. Modification of adsorbent and additional stages can reduce the cost of operation. Selective literature on intensification of PSA is tabulated in **Table 1**.

Table 1. Literature Study on Intensification of Pressure Swing Adsorption.

Ref. No.	Authors	Paper Title	Results
2	Sharma, I; Hoadley, A.; Mahajani, S.; Ganesh, A.	Optimisation of Pressure Swing Adsorption (PSA) Process for Producing High Purity CO_2 for Sequestration Purposes	An Aspen Adsorption based Multi-Objective Optimisation (MOO) framework for PSA systems was used successfully
3	Ko, D.; Siriwardane, R.; Biegler, L. T.	Optimization of a Pressure-Swing Adsorption Process Using Zeolite 13X for CO_2 Sequestration	The method of finite differences was adopted for the discretization of the spatial domains.
5	Ko, D.; Siriwardane, R.; Biegler, L.	Optimization of Pressure Swing Adsorption and Fractionated Vacuum Pressure Swing Adsorption Processes for CO_2 Sequestration	Fractionated vacuum pressure swing adsorption was developed. And was more effective than conventional methods
6	Rajasree, R.; Moharir, A.S.	Simulation based synthesis, design and optimization of pressure swing adsorption (PSA) processes	Adaptive simulation-based process synthesis and optimization strategies were developed
7	Wright, A.; Kalbassi, M.; Golden, T.	Prepurification of Air using an Advanced Thermal-Pressure Swing Adsorption (TPSA) Cycle	Temperature-pressure swing adsorption was proposed with energy saving in the range of 20-60 percent.
8	Grande, C	Advances in Pressure Swing Adsorption for Gas Separation	Provided an overview of the fundamentals of PSA process
12	Beck, J.	Efficient Targeted Optimisation for the Design of Pressure Swing Adsorption Systems for CO_2 Capture in Power Plants	The design of PSA systems for CO_2 capture was proposed.
14	Khajuria, H.	Model-based Design, Operation and Control of Pressure Swing Adsorption Systems	A detailed mathematical model was developed which captures the hydrodynamic, mass transfer and equilibrium effects in detail.

Table 2. Literature Study on Intensification of Temperature Swing Adsorption.

Ref.No.	Authors	Paper Title	Findings
33	Pahinkar, D.	Temperature Swing Adsorption Processes for Gas Separation, (Dissertation)	They Used adsorbent material as a coating to the inner surface of microchannel monolith and passed the impure feed gas through these microchannel. Heating and cooling were faster.
34	Pahinkar, D.; Garimella S.; Robbins,	Feasibility of Using Adsorbent-Coated Microchannels for Pressure Swing Adsorption: Parametric Studies on Depressurization	Due to good heat transfer characteristics of the microchannel, the heating and cooling becomes faster.
37	Moate, J. R.; M. D. LeVan, M., D.	Optimization of Temperature Swing Adsorption Systems for the Purpose of Claus Tail Gas Clean Up	Used porous adsorbent material with graphite. Effective conductivity of the composite material was several hundred times more than activated carbon alone.
38	Stegmaier, M., Linde A.G;	Nonideal gas simulation of pressure swing adsorption process	Use of active composites with fins can increase the heat transfer considerably.
39	Luo, L	Intensification of Adsorption Process in Porous Media	Thermoelectric element was found to be efficient temperature controller.

Temperature swing adsorption

Pressure swing adsorption processes consume lot of energy. These processes are slower due to resistance between adsorbent particles and slow gas diffusion through the pores [**33**]. Temperature swing adsorption also faces difficulty in heating and cooling of the poor heat conducing adsorbent materials. Pahinakar *et al.*,, in their investigation, used adsorbent material as a coating to the inner surface of microchannel monolith and passed the impure feed gas through these microchannel [**34**]. Due to good heat transfer characteristics of the microchannel, the heating and cooling becomes faster. In another work, they used heat transfer fluids through adjacent channels [**35**]. In related research, they passed the feed gas and the heat transfer fluid through the same channel [**33**]. The process capacity of this method was 100 times better than normal pressure swing system. This design yielded 4 times better results than the arrangement with separate channels for feed and heat transfer fluid [**33**]. Wahedi *et al.*, carried out investigation on temperature swing adsorption for separation of the sulphur gases [**36**]. The processes which need lower pressure (Claus Process) are candidates for temperature swing adsorption. Use of thermal conductivity promotors in adsorbent beds can decrease the cycle time considerably. Mixing of the porous adsorbent material with graphite can improve the heat transfer characteristics [**36**]. Effective conductivity of this composite material is several hundred times more than activated carbon alone [**37**]. Use of active composites with fins can increase the heat transfer considerably [**38,39**]. Thermoelectric element (TE) is a device that provides the direct conversion of temperature differences to electric current or vice versa. It creates an electric current when there is a temperature difference between two sides (Seebeck effect), or conversely, it creates a temperature difference when it is fed by an electrical current (Peltier effect). Application of thermoelectric principle for heating and cooling the bed can also reduce the cycle time. According to Leo, one noticeable feature of the thermoelectric element (TE) is that, the thermal polarity of TE depends on the polarity of applied current. In this, the cold junction and the hot junction can be changed easily by reversing a DC current. As a result, the TEs are efficient temperature controllers [**39**]. Selective literature on intensification of TSA is tabulated in **Table 2**.

Adsorbent material

Selection of the adsorbent and evaluation of its efficiency can lead to optimization of the process. Many times, even slight modification can result in large improvement in performance. Generally, isotherm and kinetic studies, selectivity, capacities are evaluated to characterise the adsorbent. Rajagopalan carried out screening of adsorbents [**40**]. He investigated four adsorbents namely Mg-MOF-74, Zeolite 13X, UTSA-16 and activated carbon. Zeolite adsorbents are aluminosilicates which are crystalline in nature. For Application of zeolite for carbon dioxide capture, the flue gas needs to be de-moisturized as the adsorption ability of zeolite decrease in presence of moisture [**41**]. Different starting materials and activation methods can be used for activated carbon. Nowadays many investigations focus on synthesis of adsorbent from waste materials [**42-53**]. Metal organic frameworks (MOFs) are also investigated for carbon dioxide capture [**53**]. At low pressure, MOFs exhibit large change in adsorption for very small change in pressure [**54**]. According to Broom, gas composition, pressure and temperature are important factors for adsorbent performance [**55**]. For pressure swing method, working capacity is the difference between uptake pressure in pressurization and depressurization. It is difference between corresponding temperatures for temperature swing adsorption. Working capacity is a function of isotherm. Internal particle and intraparticle voids affect the adsorption [**56**]. Stefański *et al.*, discussed adsorption bed for cooling operation [**57**]. They discussed different configurations for the same. In their review, they discussed loose grain beds and fixed beds. According to them, a granular bed demonstrates higher intensity of adsorption and desorption process for the same layer thickness. The advantages of coated and granular bed can be obtained by using combined bed.

Contacting pattern

Fixed beds are commonly used as contacting equipment for gas-solid systems. Fixed beds have limitations in terms of the bed efficiency, cycle time and capacity. Fixed bed adsorption is unsteady state operation. The driving force for the mass transfer is not evenly divided in fixed beds. The counter current adsorption with moving bed can be considered as better option for normalizing the driving forces. Large columns, long heating and cooling times are disadvantages of fixed bed, (temperature and pressure swing) methods. Moving bed adsorption with amine supported catalyst has been investigated by Krutka and Sjostrom [**58**]. Simulated moving bed adsorption involves manipulating input streams for feed, regeneration to ensure counter current pattern [**59,60**]. Difficulties such as conveying of adsorbent, attrition of adsorbent, capacity loss have limited the use of moving bed absorbers. Circulation fluidized beds can be used for dehumidification and adsorption of volatile organic compounds (VOCs). Uniform bed temperature and mass transfer are advantages of circulation fluidized beds [**61**].

Conclusion

Many investigators have investigated intensification of pressure swing adsorption. However, there is limited literature available on intensification of temperature swing adsorption. In this paper intensification of pressure and temperature swing adsorption along with selection of adsorbent and contacting pattern is discussed. Regeneration of adsorbent bed and desorption of solute from adsorbent from the bed is very important for economical and environment friendly operation. In pressure swing adsorption, pressurization and depressurization are used for adsorption and desorption of solute.

Pressure swing adsorption is energy insensitive method. Loss of adsorbent capacity and low purity can be overcome by using hybrid method with both pressure and temperature swing methods. For pressure swing method, working capacity is the difference between uptake pressure in pressurization and depressurization. It is difference between corresponding temperatures for Temperature swing adsorption. Temperature swing adsorption (TSA) involves manipulation of temperature for adsorption and desorption. This is a cyclic process. In the simplest two bed operation, one bed is used for adsorption while other is regenerated. After one cycle, second bed is used for adsorption and first is regenerated. The low heat conductivity of the materials is limitation of this method. Longer cycle times are required as more time is required for heating and cooling the bed. Application of thermoelectric principle for heating and cooling the bed can also reduce the cycle time. Working capacity is a function of isotherm. Internal particle and intraparticle voids affect the adsorption. Investigations are also reported on methods to increase the conductivity of porous materials and use of thermoelectric heating and cooling methods for rapid heating and cooling of the beds. Simulated moving bed adsorption involves manipulating input streams for feed and regeneration to ensure counter current pattern. Difficulties such as conveying of adsorbent, attrition of adsorbent, capacity loss have limited use of moving bed absorbers. Circulation fluidized beds can be used for dehumidification and adsorption of volatile organ compounds (VOCs). Uniform bed temperature and mass transfer are advantages of circulation fluidized beds.

Acknowledgements

Authors acknowledge the support of their affiliated institutes and their Principals.

Conflicts of interest

There are no conflicts to declare.

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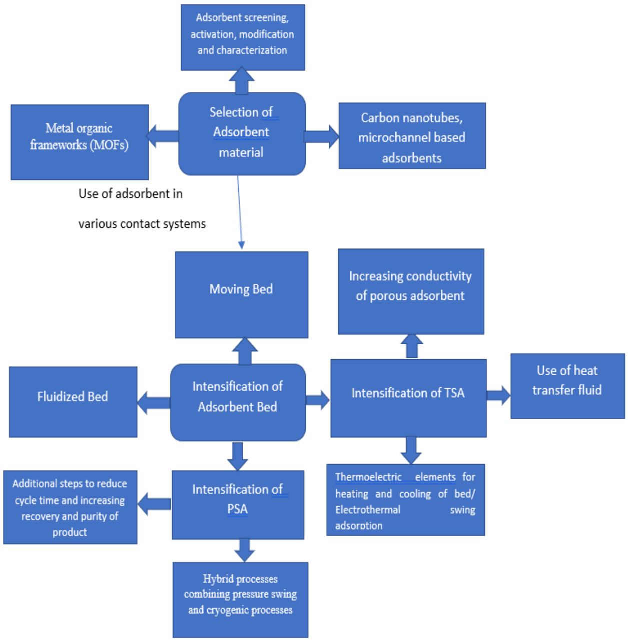
Authors biography

Dr. Sunil J. Kulkarni has completed his Ph.D. in Chemical Technology and Masters in Chemical Engineering. He is working as Associate Professor in Chemical Engineering Department of Gharda Institute of Technology, Khed, India. He has 19 years of teaching and research experience. Dr. Sunil is involved with different academic activities across the universities in Maharashtra, India. His area of investigation in doctoral studies is phenol and heavy metal removal from waste water.

Dr. Ajaygiri K. Goswami has completed his Masters in Chemical Technology from Nagpur University and Ph.D in Chemical Tech. from University Institute of Chemical Technology, Jalgaon, India. He has presented and published more than 15 international research papers. His area of interest includes biodiesel and process control. He has about 21 years of teaching experience. He is working as Associate professor in chemical engineering at University Institute of Chemical Technology, Jalgaon, India.

Graphical abstract

Intensification of adsorbent beds can be carried out based on adsorbent material, contacting equipments and sequencing of the steps. Selection and proper activation of the adsorbent can increase the recovery. PSA can be intensified by reducing cycle time and electricity cost by modifying the processes with some additional steps. For TSA, increasing thermal conductivity and conduction in the bed by using heat transfer fluids and addition active composites in the beds can improve the performance.



Intensification of Adsorbent Beds