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ZEOLITE SEPARATION PROCESS – DESCRIPTION OF THE PHENOMENON, TAKING INTO ACCOUNT RESEARCH ON A NATIONAL AND GLOBAL SCALE

PROCES SEPARACJI Z WYKORZYSTANIEM ZEOLITÓW – OPIS ZJAWISKA Z UWZGLĘDNIENIEM BADAŃ PROWADZONYCH W SKALI KRAJOWEJ I GLOBALNEJ

Summary: This article presents the properties of zeolites, their applications in separation, and examples of pressure swing adsorption studies. The article highlights the potential development of zeolite separation and utilization technologies for heat storage applications.

Keywords: zeolites, separation, thermal swing adsorption, vacuum pressure swing adsorption, electric swing adsorption, rapid cycles

Streszczenie: W niniejszym artykule przedstawiono właściwości zeolitów, ich zastosowania w procesach separacji oraz przykłady badań dotyczących adsorpcji z przemianą ciśnienia. W artykule zwrócono uwagę na potencjał rozwoju technologii separacji i wykorzystania zeolitów w zastosowaniach związanych z magazynowaniem ciepła.

Słowa kluczowe: zeolity, separacja, adsorpcja z przemianą temperatury, adsorpcja z przemianą ciśnienia próżniowego, adsorpcja z przemianą elektryczną, szybkie cykle

Introduction

The term "zeolite" (from the Greek: "ξέω" –boiling, "λίθος" – stone) was introduced in 1765 by the Swedish mineralogist Freiherr Axel von Cronstedt. He referred to the phenomenon of the release of large amounts of water by certain minerals. The ability to store water (called zeolitic water) in the channels of the crystal structure is a characteristic feature of zeolites. Dipole bonds occur between polar water molecules and the zeolite skeleton. Water can be removed by heating and then reabsorbed or replaced by other substances. If water is lost too rapidly, the structure is irreversibly damaged. Desorption of zeolitic water is a function of time and temperature.

Currently, the group of natural zeolites includes approximately 40 minerals, the most commonly found and used of which are:

- clinoptilolite $\text{Na}_6[(\text{AlO}_2)_6(\text{SiO}_2)_{30}]\cdot 24\text{H}_2\text{O}$,
- chabazite $\text{Ca}_2[(\text{AlO}_2)_4(\text{SiO}_2)_8]\cdot 13\text{H}_2\text{O}$,

- mordenite $\text{Na}_8[(\text{AlO}_2)_8(\text{SiO}_2)_{40}]\cdot 24\text{H}_2\text{O}$.

In natural zeolites, the silicon-to-aluminum molar ratio is 1-6.

According to Loewenstein's rule, tetrahedra AlO_4 they can only bond with SiO_4 tetrahedra, but these can also bond with each other. The zeolite skeleton is formed by alternating AlO_4 and SiO_4 tetrahedra. The Si/Al ratio determines the classification of zeolites into: – low-silica: Si:Al = 1-1.5, e.g., A, X; – medium-silica: Si:Al = 2-5, e.g., natural zeolites – mordenite, erionite, synthetic zeolites – X, Y; – high-silica: Si/Al = 10-100, e.g., obtained by modifying the high-silica network – Y zeolites, mordenite, erionite, obtained by synthesis with crystallization-directing agents – ZSM-5; – silica molecular sieves: Si/Al silicalites >100, SiO_2 , silica with a zeolite structure [1]. The varying Si/Al ratios in zeolites determine their properties. Low-silica zeolites are characterized by increased acid resistance, stability at higher temperatures, and hydrophilicity. High-silica zeolites, on the other hand, are characterized by a high degree of ion exchange and hydrophobicity. Natural zeolites are stable at pH > 6. Extreme

pH values cause structural damage, especially in strongly acidic environments. Zeolites with a high Si:Al ratio are more resistant to acidic environments. The primary structural elements of the zeolite network are aluminum tetrahedra (AlO_4) and silicon tetrahedra (SiO_4), in which an Al or Si atom is surrounded by four oxygen atoms [1].

Zeolites are characterized by the presence of pores, and are classified as narrow-pore (zeolite A), medium-pore (ZSM-5 and ZSM-11), and wide-pore (zeolite X, zeolite Y). The channel systems present in zeolites are diverse. They can be one-, two-, or three-dimensional and influence the sorption properties of zeolites. Water molecules occur in the voids of the crystal lattice. In the dried state, zeolites exhibit sorption, catalytic, and molecular-sieve properties [1].

Natural zeolites vary in their structural structure and physicochemical properties. The most commonly used in industry and environmental protection are mordenite, clinoptilolite, chabazite, and analcite. Natural zeolites are formed primarily as hydrothermal formations. They most often occur in fractures and voids within extrusive rocks (e.g., basalts) or as products of the transformation of feldspars and scapolites. Under natural conditions, the process of creating zeolites from volcanic ash, at elevated temperatures and pressures, takes several thousand years. Synthesis of zeolites from fly ash, however, takes few to several hours. In addition to natural zeolites, modified and synthetic zeolites are also used. Zeolite modification is most often used to enhance their ion exchange capacity, increase their capacity, and obtain a more selective material [2, 3, 4, 5, 6].

It is estimated that the global use of natural zeolites is approximately 3.6 megatons per year. The main producers are Cuba, Germany, Japan, South Korea, Australia, Indonesia, and New Zealand. In Poland, they occur in Lower Silesia, the Kraków region, and the Pieniny Mountains [1].

The use of zeolites in the context of separation

Zeolites are distinguished by a number of unique physicochemical properties, including high adsorption capacity, molecular sieving capacity, selectivity, ion exchange capacity, and resistance to acids and elevated temperatures. Zeolites have a highly developed surface area, reaching up to $1,500 \text{ m}^2 \cdot \text{g}^{-1}$. This makes them an attractive material for use in processes involving sorption and ion exchange. Agriculture is the dominant area of application for natural zeolites [1, 7].

Zeolites are used for gas drying and purification, as well as for gas separation. These materials are characterized by selectivity and significant sorption capacity for water due to its dipole properties. Therefore, zeolites are used, for example, for drying neutral gases and for drying atmospheric air. The variety of pore sizes and the presence of cations in the zeolite structure also make them suitable for the selective adsorption of components of gas mixtures. Research into their application in the removal of CO_2 , SO_2 , and nitrogen oxides has yielded positive results, including for zeolites synthesized from fly ash [8]. One of the latest developments in the use of fly ash is its use as

a substrate [9] for the synthesis of zeolites [10, 11], which, due to their properties, are used in many fields of engineering and environmental protection, including oil refining [12], the removal of ammonium ions [13, 14] and heavy metals [15] from wastewater, or radionuclides from mine waters [16], as well as in the separation/adsorption of gases such as CO_2 [17, 18, 19], SO_2 [20], or gaseous forms of mercury [21, 22].

Methods for separating carbon dioxide from gas mixtures using zeolite adsorption. Gas separation and purification methods based on physical adsorption have been considered the most promising development direction for over a decade [23]. Pressure swing adsorption (PSA) is based on the selective adsorption of gases on solid adsorbents [24].

Pressure swing adsorption is an example of an adsorption method. This process is based on the selective adsorption of gases on solid sorbents (zeolites). The driving force of the process is the pressure difference between the successive cycles of adsorption (occurring at elevated pressure) and desorption (occurring at reduced pressure). Pressure swing adsorption involves selecting process conditions and the type of adsorbent so that one of the gas mixture components can selectively adsorb onto the bed. Then, after the adsorbent bed is saturated, the pressure is reduced, desorbing the adsorbed component. This can, of course, be either a desired product or a waste product. Technologies based on pressure swing adsorption are now commonly used in plants producing hydrogen from natural gas, separating carbon dioxide (a waste released into the atmosphere) from hydrogen. Similarly, PSA systems are increasingly being used to produce oxygen and nitrogen from air, replacing cryogenic methods. Pressure swing adsorption systems are characterized by low capital and maintenance costs and low failure rates. They can be quickly started and shut down. They are flexible in terms of load and depreciate slowly. The systems operate essentially at ambient temperature and do not require thermal insulation. Pressure swing adsorption is an appropriate technique for separating gaseous components present in low concentrations. The physical adsorption of gases on solid adsorbents is associated with a low energy effect, which is most important during the adsorbate desorption stage. In most systems, the process is close to isothermal. A common feature of all adsorbents used for the separation of gas mixtures by the PSA method is the cyclical change in pressure and gas flow direction in the column. A single device operates periodically, alternating adsorption and desorption, so to ensure process continuity, two or more columns with a phase-shifted operating cycle are used. More advanced solutions typically use six-column rather than four-column systems and incorporate multiple columns operating at different stages of the cycle. Additional operations primarily involve "rinsing" the columns between specific cycle stages to separate individual gas streams to obtain a product with maximum concentration, high recovery, and high system efficiency. Selecting the appropriate adsorbent and optimizing the system's operating system can significantly minimize process costs, by up to 40% [24].

Examples of pressure swing adsorption studies

Modifications to the classic pressure swing adsorption process, primarily related to the method of adsorbate desorption, have led to the division of the basic process into several distinctive varieties:

- 1) TSA (Thermal Swing Adsorption). Desorption is assisted by increasing the temperature of the adsorbent bed, achieved without a diaphragm, using a gas stream heated in an exchanger. This ensures rapid and deep desorption, but it is necessary to add a cooling operation after the desorption process, which extends the operating cycle and, consequently, the system's efficiency [24].
- 2) VPSA or VSA (Vacuum Pressure Swing Adsorption). Desorption of the adsorbed gas occurs under the reduced pressure, typically to 0.01–0.05 bar. This allows the adsorption process to proceed at atmospheric pressure, avoiding the need for gas compression. Low pressure, in turn, effectively desorbs the gas, increasing the bed's working capacity and, consequently, the system's daily efficiency [24].
- 3) ESA (Electric Swing Adsorption). This refers to adsorption processes on conductive carbon materials, nanotubes, and nanofibers [25]. Applying a voltage to the adsorbent bed causes rapid desorption of the adsorbed gas – regeneration efficiency reaches 85% (20% more than using vacuum techniques). This is a new method, currently undergoing intensive research. The price of carbon nanomaterials is a barrier to its practical application.
- 4) "Rapid Cycles" systems [23]. This system uses wide, low columns and extremely short cycle times:
 - filling 0.3–1.0 s;
 - pause 0.5–3.0 s;
 - emptying approximately 2 s.

This operating regime does not provide high recovery, but it does provide an approximately five-fold increase in efficiency.

Characteristics of sorbents (zeolites) – molecular sieves are crystalline aluminosilicates of divalent and alkali metals. Zeolites are composed of SiO_4 and AlO_4 tetrahedra linked by oxygen ions. Their main advantage is a narrow pore size range of 0.3–1.1 nm [24].

Adsorbents for industrial gas purification, in addition to having an appropriate porous structure, must meet a number of features that allow them to be used as packing in adsorption columns [26]. Requirements for these materials:

- 1) Porous structure:
 - large specific surface area (SBET 1000–2500 m^2/g),
 - high micropore content.
- 2) Process conditions:
 - mechanical strength,
 - good selectivity,
 - low resistance to the gas flow,
 - high adsorption capacity per unit volume,
 - complete desorption of adsorbed gases at elevated temperature and reduced pressure,
 - high adsorption affinity for gases in the presence of water vapor.

Zeolites are also porous materials that store hydrogen – hydrated aluminosilicates that contain small-diameter tunnels in their structure [27]. The most well-known representative of this material group is germanosilicate – ITQ-33 [28]. Its mass capacity (when filled with liquid hydrogen) is 2.5% by weight [29].

There is a method combining PSA (Pressure Swing Adsorption) and TSA (Temperature Swing Adsorption) – PTSA (Pressure Temperature Swing Adsorption), in which desorption occurs by varying both pressure and temperature. In the PTSA method, adsorption is carried out at high pressure and low temperature, while desorption occurs by decreasing pressure and increasing temperature [30].

Na-A zeolite, synthesized from fly ash, was used to capture CO_2 in the PTSA system. This solution is one of the methods for neutralizing ash generated by coal combustion. The exhaust gases leaving the boiler have a pressure of 1 bar and a temperature of 123°C, while the exhaust gas flow rate is 483.2 kg/s. The first step of the simulation analysis was to select the appropriate adsorption and desorption parameters and the factors necessary for proper system operation.

The effect of exhaust gas pressure on sorbent demand – as the adsorption pressure increases, the sorbent's sorption capacity increases, resulting in a reduction in sorbent demand [31]. An increase in pressure from 1.2 bar to 2 bar reduces sorbent demand by approximately 83% compared to the demand at 1.2 bar. A compressor powered by reheated steam, drawn from the power plant's steam-water cycle, was used to compress the exhaust gases. Although compression to higher pressures further reduces sorbent demand, the energy inputs required for this process do not justify compressing the exhaust gases to higher pressures. The analysis assumed a constant temperature of 110°C for the exhaust gases entering the PTSA unit. Compression of the exhaust gases increases their temperature to maintain a constant temperature before entering the PTSA unit, requiring cooling via an exchanger. Condensate collected at a pressure of 22 bar and a temperature of 35°C was used to cool the exhaust gases. After heating it in the heat exchanger to a temperature of 152.6°C, it was recycled to the main circuit and introduced between the heat exchanger and the deaerator. Based on the analysis, it was determined that the exhaust gases would be introduced into the PTSA unit at a pressure of 2 bar and a temperature of 110°C.

Influence of desorption pressure on sorbent demand – the desorption process occurs at a pressure lower than the adsorption process, and the amount of CO_2 produced is the difference between the adsorption capacity of the sorbent under adsorption and desorption conditions. This means that as the pressure decreases (and thus the sorption capacity decreases), the sorbent demand decreases, as a larger CO_2 stream can be recovered from the same amount. As the desorption pressure decreases from 0.5 bar to 0.15 bar, the sorbent demand decreases by approximately 30%. The pressure at which desorption will be conducted was assumed to be 0.15 bar.

The influence of steam parameters on the separation process – the CO_2 separation process requires heat in the form

of heating steam. The influence of heating steam parameters on the demand for the Na-A sorbent was studied. Steam parameters were varied within the following ranges: pressure 10–30 bar and temperature 200–500°C. The selected parameter range includes the steam bleeds from which the heating steam will be drawn. As steam parameters increase, the demand for sorbent decreases. At a pressure of 30 bar, desorption occurs at a temperature of 231°C, and the sorbent demand is lowest, which is due to the nature of the process itself. Therefore, in this case, steam with higher parameters was selected as the heating medium, i.e., steam from the bleed at a pressure of 26.81 bar and a temperature of 478.7°C. This will reduce sorbent consumption. For the mentioned steam parameters, the temperature at which desorption occurs is 231.8°C. Sorbent cooling – After the desorption process, the sorbent reaches a temperature of 225°C. For reuse, it must be cooled to 121°C. For this process, condensate at 22 bar pressure and 35°C is used, collected downstream of the condensate pump. The condensate is heated to 119°C in the PTSA unit and then recycled to the main circuit between the exchangers [31].

Zeolite performance

Due to their unique porous structure, zeolites are widely used as adsorbents, catalysts, and ion exchangers. In the energy sector, a key area is sorptive thermal energy storage, where the heat of water adsorption and desorption is utilized [32].

Table 1 presents a comparison of zeolite types, including their main properties and applications.

The efficiency of systems based on the water-zeolite system depends on the charging temperature and ambient conditions [34, 37, 38]. Zeolite systems achieve densities of 150–200 kWh/m³. In practical (full-scale) applications, values ranging from 19.7 to 50.1 kWh/m³ have been achieved. Laboratory and demonstration units demonstrate charge/discharge cycle efficiencies exceeding 90% under optimal conditions. The desorption process (storage charging) typically requires temperatures in the range of 175–250°C.

The energy storage process in zeolites is a thermochemical cycle [39, 40] and involves a phase-wise mechanism:

- 1) Charging phase (desorption):
 - Heat (e.g., solar or waste heat) is supplied to the wet zeolite;
 - Water is evaporated and removed from the porous structure;
 - Energy is stored in the form of adsorption potential (dry material) with virtually no loss over time.
- 2) Discharge Phase (Adsorption):
 - Introducing water vapor into the dry zeolite;
 - Water binds in the pores, causing a rapid temperature increase (exothermic reaction).
 - Heat recovery for domestic hot water or space heating.
- 3) Storage Applications [34, 37]:
 - Seasonal heat storage: Due to the lack of energy loss over time (as long as the zeolite remains dry), this technology allows for storing summer heat for winter use;
 - Mobile Energy Storage (m-TES), transporting charged zeolite from waste heat generation sites (e.g., factories) to end users;
 - Integration with Renewable Energy Sources (RES), working with solar collectors to stabilize the thermal energy supply in residential buildings. Taking into account the research on zeolites carried out in the Łukasiewicz Research Network – Institute of Artificial Intelligence and Cybersecurity, Research Centre for Innovative Technologies, Katowice, the paper presents an analysis of the literature in the context of directing potential activities aimed at developing a heat storage model.

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Table 1. List of the most commonly used zeolite types and their key properties [own study]


Zeolite Type	Pore Diameter [nm]	Si/Al Ratio Main	Main Properties and Applications
LTA (e.g., 4A) [33]	~0.4	Low (~1.0)	Highly hydrophilic, ideal for gas drying and calcium ion removal.
FAU (e.g., 13X, NaY) [34]	~0.7 - 0.8	Medium (1.2 - 2.5)	Very high adsorption capacity; Type 13X is the standard in heat storage.
MFI (e.g., ZSM-5) [35]	~0.5	High (>10)	Hydrophobic, high thermal stability; used primarily in petrochemical catalysis.
Clinoptilolite [36]	~0.4	Variable	Natural zeolite; less expensive, used in water treatment and agriculture.

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