

NATURAL GAS DESULFURIZATION: TECHNOLOGICAL TRANSFORMATION AND ENVIRONMENTAL SUSTAINABILITY ISSUES

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ANNOTATSIYA

Tabiyy gazga bo'lgan global talabning ortib borishi va ekologik me'yorlarning tobora qat'iylashuvi korroziyani keltirib chiqaruvchi hamda iqlim o'zgarishiga hissa qo'shuvchi H₂S, SO₂, CO₂ va suv bug'i kabi kislotali aralashmalarni samarali olib tashlashni talab etadi. Alkanolamin eritmaları, ayniqsa DEA va MDEA asosidagi kimyoviy adsorbsiya usuli eng keng qo'llaniladigan usul bo'lib qolmoqda, biroq uzoq muddatli ekspluatatsiya korroziya, ko'piklanish, oksidlovchi degradatsiya, termik barqaror tuzlarning hosil bo'lishi va ekspluatatsion xarajatlarning ortishiga olib keladi. Ushbu muammolarni bartaraf etish maqsadida adsorbsiya sig'imini oshirish, korroziyani kamaytirish va ko'piklanishni nazorat qilish uchun azot saqlovchi qo'shimchalar, suvda eruvchan polimerlar, ammoniy gidroksid (NH₄OH) hamda amin promouterlari bilan modifikatsiyalangan DEA va MDEA asosidagi kompozit adsorbentlar ishlab chiqildi. Bundan tashqari, mahalliy mineral xomashyalardan foydalanishga asoslangan yopiq siklli ishlab chiqarish tizimlari taklif etildi. LTA turidagi 4Å va 5Å seolitlar Angren kaolinidan sintez qilinib, SEM va XRD usullari yordamida tavsiflandi. Sintez qilingan materiallar tijorat analoglariga nisbatan yuqori H₂S adsorbsiya sig'imini namoyon etdi, bu esa yuqori samaradorlikka ega, import o'rini bosuvchi adsorbentlar va ekologik barqaror gaz tozalash texnologiyalarini ishlab chiqish imkonini berdi.

Kalit so'zlar: Tabiiy gazni tozalash, vodorod sulfididan tozalash, aminli adsorbsiya, membranali ajratish, oltingugurtni qayta tiklash, gazni nordon komponentlardan tozalash, gibridd texnologiyalar.

АННОТАЦИЯ

Растущий мировой спрос на природный газ и ужесточение экологических требований требуют эффективного удаления кислых примесей, таких как H₂S, SO₂, CO₂ и водяной пар, которые вызывают коррозию и способствуют изменению климата. Химическая абсорбция с использованием растворов алканоломинов, в частности DEA и MDEA, остаётся наиболее широко применяемым методом, однако длительная эксплуатация приводит к коррозии, пенообразованию, окислительной деградации, образованию термостабильных солей и увеличению эксплуатационных затрат. Для решения этих проблем были разработаны композитные сорбенты на основе DEA и MDEA, модифицированные азотсодержащими добавками, водорастворимыми полимерами, гидроксидом аммония (NH₄OH) и аминовыми промоторами, с целью повышения абсорбционной способности, снижения коррозии и контроля пенообразования. Кроме того, были предложены замкнутые производственные системы с использованием местных минеральных ресурсов. Цеолиты типа LTA 4Å и 5Å были синтезированы из ангренского каолина и охарактеризованы методами SEM и XRD. Синтезированные материалы продемонстрировали более высокую адсорбционную способность по отношению к H₂S по сравнению с коммерческими аналогами, что позволило разработать высокоэффективные, импортозамещающие адсорбенты и экологически устойчивые технологии очистки газа.

Ключевые слова: Очистка природного газа, удаление сероводорода, аминовая абсорбция, мембранное разделение, извлечение серы, очистка газа от кислых компонентов, гибридные технологии.

ABSTRACT

The increasing global demand for natural gas and stricter environmental regulations require efficient removal of acidic impurities such as H₂S, SO₂, CO₂, and water vapor, which cause corrosion and contribute to climate change. Chemical absorption using alkanolamine solutions, particularly DEA and MDEA, remains the most widely used method, although long-term operation leads to corrosion, foaming, oxidative degradation, heat-stable salt formation, and increased operating costs. To address these challenges, composite adsorbents based on DEA and MDEA modified with nitrogen-containing additives, water-soluble polymers, ammonium hydroxide (NH₄OH), and amine promoters were developed to enhance absorption capacity, reduce corrosion, and control foaming. Additionally, closed-cycle production systems using local mineral resources were proposed. LTA-type 4A and 5A zeolites were synthesized from Angren kaolin and characterized by SEM and XRD. The synthesized materials showed higher H₂S adsorption capacity than commercial analogs, enabling the development of high-performance, import-substituting adsorbents and environmentally sustainable gas purification technologies.

Keywords: Natural gas purification, hydrogen sulfide removal, amine absorption, membrane separation, sulfur recovery, gas sweetening, hybrid technologies.

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Introduction

Sulfur removal processes

Modern innovative technologies play an important role in improving energy efficiency and reducing environmental impacts. Considerable research has been focused on improving deep absorption purification of natural gas using alkanolamines [1-5]

In recent years, natural gas production has increased significantly, reaching approximately 50 billion m³ per year in Uzbekistan. At the same time, the hydrogen sulfide content in natural gas has also increased, and the proportion of high-sulfur gases continues to grow [1]. In addition to H₂S, natural gas contains CO₂, thiols, mercaptans, and alkyl sulfides, which must be removed at the initial stage of processing [2-4]. These compounds are toxic and highly corrosive, causing equipment corrosion and catalyst deactivation. Moreover, recovered sulfur compounds represent valuable raw materials for industrial applications [6,7].

Currently, more than 20 processes are used for the removal of sulfur compounds from gases. These processes depend on gas composition, temperature (T), pressure (P), and the physicochemical properties of sulfur-containing components. They are generally classified into two main groups (Table 1):

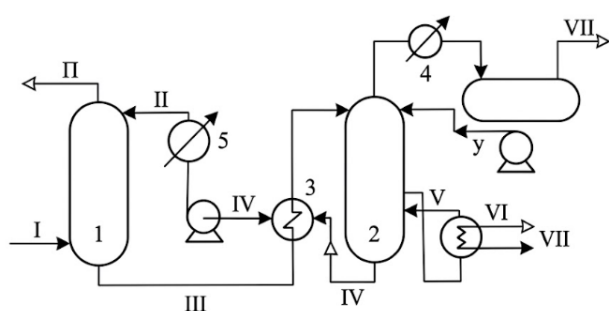
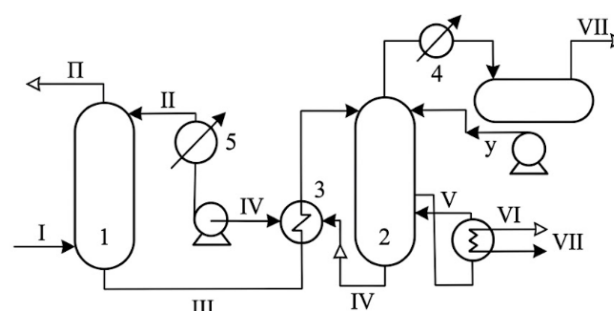
- removal of hydrogen sulfide;
- simultaneous removal of hydrogen sulfide and organic sulfur compounds.

The first group is mainly based on chemical absorption processes, whereas the second group is based on physical absorption methods. In some technologies, organic sulfur compounds are first converted into hydrogen sulfide and subsequently removed.

These processes can be applied individually or in combination, and the selection of a technological scheme is based on technical and economic criteria.

Table 1. Physical and chemical properties of sulfur compounds [8, 9].

Substance	Molecular weight	t_m , °C melting point	t_b , °C boiling point	T_c , °C critical temperature	P_c , MPa critical pressure	ρ , g/cm ³ density	ρ_c , g/cm ³ critical density	n_D refractive index	μ , D dipole moment	$K_d \times 10^{11}$ dissociation constant
H ₂ S	34	-85,6	-60,8	100,4	9	1,538 ²⁵	0,349	—	0,93	$6 \cdot 10^3$ $1 \cdot 10^{-3}$
COS	60	-138,2	-50,2	31,3	65,1	g. 1,072 ⁰ s. 1,24 ⁻⁸⁷	—	—	0,65	—
CS ₂	76	-112	46,2	279	7,9	1,263 ²⁰	0,44	1,6295 ¹⁸	0	—
CH ₃ SH	48,1	-123	6	196,8	71,4	0,8599 ²⁵	0,323	—	1,26	4,20 4,70
C ₂ H ₅ SH	51,6	-147,9	35	226	54,2	0,8314 ²⁵	0,300	1,4278 ²⁵	1,38	2,52
C ₃ H ₇ SH	76,2	-111,5	67,8	—	—	0,8357 ²⁵	—	1,4351 ²⁵	1,33	2,26
C ₄ H ₉ SH	90,2	-115,7	98,2	—	—	0,8365 ²⁵	—	1,4401 ²⁵	1,38	2,21

**Figure 1.** Methods for Purification of Natural and Waste Gases Generated in Industrial Production**Figure 2.** Process Flow Diagram for Gas Purification Using Amine Solutions

When selecting a gas purification method, several factors are considered, including gas flow rate, temperature, pressure, gas composition, sulfur compound concentration, presence of CO₂, and required product quality. The degree of purification depends on the intended application of the treated gas.

The classification of purification methods for natural and waste gases is presented in Figure 1 [8–14].

Cyclic Processes for Natural Gas Purification from Sulfur Compounds

The most widely used methods for hydrogen sulfide removal from natural gas are chemisorption processes, in which aqueous solutions of organic bases (amines such as MDEA, MEA, DEA, DIPA, DGA, and TEA), composite absorbents, and other reactive solutions are applied [15–20].

The process flow scheme is generally similar and is presented in Figure 2. In this system, raw gas is purified in an absorber, while the rich (saturated) solution is sent to a regeneration unit. Regeneration is carried out in a reboiler at temperatures of 70–150 °C, resulting in the desorption of acid gases. The released gases are further processed either for sulfuric acid production or for elemental sulfur recovery via the Claus process.

If CO₂ is present in the gas composition, it is also removed together with H₂S; however, its absorption rate is lower compared to hydrogen sulfide.

The process consists of several main units: absorber (1), desorber (2), heat exchanger (3), condenser-cooler (4), and cooler (5). The incoming gas (stream I) enters the absorber, where it comes into contact with the amine solution and acidic components are removed. The purified gas (stream II) leaves the top of the absorber, while the saturated absorbent (stream III) is directed to the desorber for regeneration.

In the desorber, the absorbent is heated using steam (stream VI), releasing acid gases (stream VII). The regenerated absorbent (stream IV) is cooled in the heat exchanger and cooler before being recycled back to the absorber. Water (stream V) is used in the cooling and condensation stages to improve process efficiency.

Energy consumption during gas purification can be reduced by controlling CO₂ absorption. The selectivity of H₂S absorption is relatively

low for primary and secondary amines, since they rapidly react with CO₂ to form carbonates. Selectivity decreases with increasing temperature and irrigation density. The removal efficiency of organic sulfur compounds typically does not exceed 30%; mercaptans and CS₂ are mainly removed through physical dissolution, while COS removal depends on the hydrolysis rate in alkaline media and increases with rising pH. The optimal operating temperature is 60–80 °C. The absorption capacity of chemisorbents mainly depends on temperature and concentration, whereas the influence of partial pressure is relatively small. Therefore, energy consumption increases proportionally with the concentration of sulfur compounds in the gas [17–21].

In industrial practice, physical absorption-based processes are widely applied, including Fluor (propylene carbonate), Purisol (N-methylpyrrolidone), Rectisol (methanol at –30 °C), and Selexol (polyethylene glycol dimethyl ether) [22, 23]. These methods are economically efficient for high-pressure gases with high concentrations of acid components; however, high hydrocarbon solubility in the absorbent negatively affects the quality of the recovered gas. Physicochemical solvents (amine + physical solvent + water) combine chemisorption of CO₂ and H₂S with physical dissolution of organic compounds.

Adsorption processes are carried out in fixed, moving, and fluidized beds and proceed through cyclic adsorption–desorption stages. Zeolites are widely used for gas drying, purification, deep dehydration, molecular sieve separation, and ion-exchange processes.

Type A zeolite adsorbents based on kaolin have been developed, enabling the formation of polycrystalline structures with high mechanical strength [24]. In addition, high-purity zeolite A is obtained using systems containing pre-synthesized gel additives [4, 7]. These adsorbents are produced through thermal activation and hydrothermal crystallization.

The properties of zeolite adsorbents depend on the volume and radius of secondary pores, where an optimal structure enhances their dynamic activity [9, 11]. Pore size distribution is also a key factor, and dominance of pores within a specific range improves adsorption efficiency [10].

The efficiency of adsorbents is additionally influenced by the presence of transition metals and acidic sites. Matrix composition and pore structure affect both adsorption and catalytic properties; optimal pore

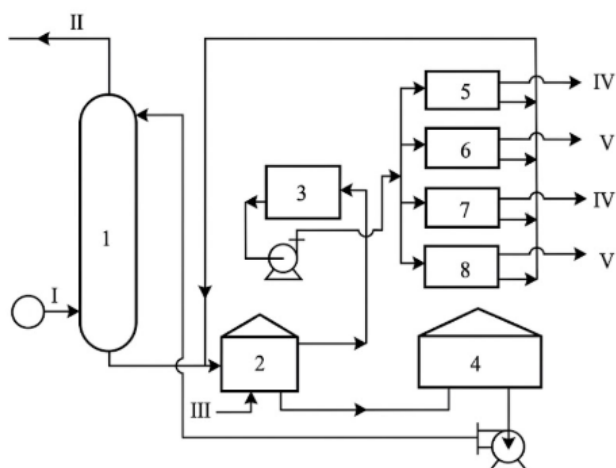


Figure 3. Gas purification scheme using polyethylene glycol dimethyl ether ("Selexol" process)

sizes reduce catalytic activity while maintaining adsorption performance [11]. Thermal treatment leads to the formation of $\gamma\text{-Al}_2\text{O}_3$, which increases dynamic activity [9]. Increasing acidity through chlorination or fluorination enhances catalytic activity, whereas alkali treatment produces the opposite effect [12].

To reduce hydrocarbon losses in acid gas streams, multistage degassing and gas recompression with recycle are applied (Figure 3), although this increases energy consumption. Physical absorption processes become more energy-efficient than amine-based systems when partial pressure exceeds 4–6 atm.

The process includes the following main units: absorber (1), high-, medium- and low-pressure degassing vessels (2–4), desorber (5), and reboiler (6). The raw gas (stream I) enters the absorber, where acid gases are selectively removed by the Selexol solvent. The purified gas (stream II) exits the top of the absorber, while part of the gas may be recycled (stream III) to improve separation efficiency.

The loaded solvent is then directed through a series of degassing vessels operating at decreasing pressures, where dissolved gases are progressively released. The solvent is further regenerated in the desorber, supported by the reboiler, which provides the necessary heat for stripping. Acid gases are removed as stream IV, while water vapor (stream V) is generated during the regeneration stage.

Among the proposed solvent systems, the most effective is a mixture of tetrahydrothiophene dioxide (sulfolane), DIPA, and water, which is used as the absorbent in the "Sulfinol" process. The typical composition of the solution is (mass %): sulfolane 35 %, DIPA 50 %, and water 15 % [25].

The disadvantages of this absorbent system include the high cost of organic components, increased solvent consumption due to DIPA degradation under CO_2 influence, and relatively high solubility of heavy hydrocarbons, particularly aromatic compounds.

Nevertheless, in the presence of organic sulfur compounds in the gas stream, the "Sulfinol" process is considered economically more efficient than amine-based purification processes.

Oxidative processes for purification of natural gas from sulfur compounds

Oxidative processes are intended for the removal of hydrogen sulfide from gases and are based on its oxidation in the liquid phase. Depending on conditions and catalyst type, oxidation of H_2S leads to the formation of elemental sulfur, thiosulfates, or sulfates.

Oxidative processes are divided into three types:

Processes where the absorption solution simultaneously performs both absorption and oxidation functions (e.g., "Thylox", "Giammarco-Vetrocoke", "Manchester" processes), in which sodium/ammonium thioarsenates, arsenate–arsenites, or iron oxide suspensions are used.

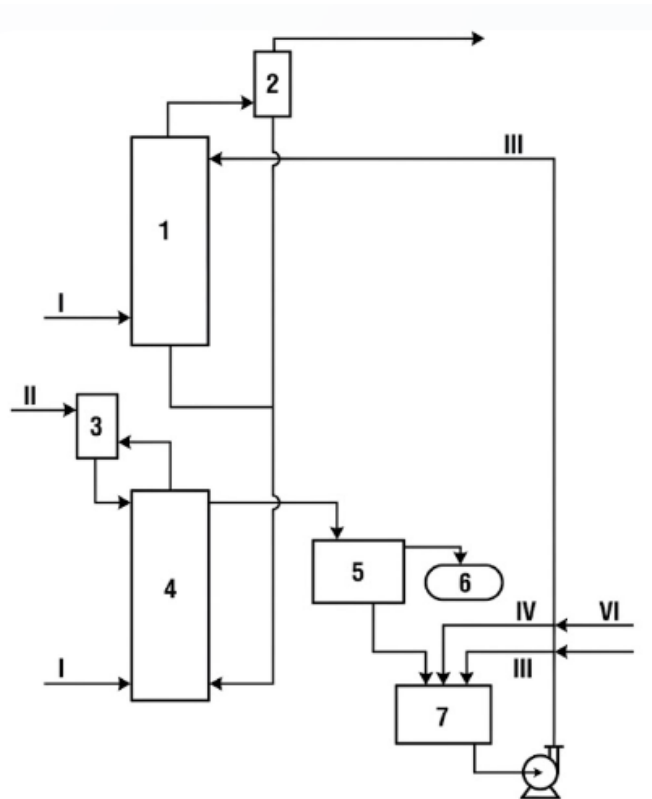


Figure 4. Scheme of hydrogen sulfide removal by the "Stretford" process

Processes using organic oxidation catalysts in weakly alkaline solutions ("Perox", "Stretford").

For both types, the technological scheme is similar (Figure 4), the difference lies in the stage at which H_2S oxidation occurs: in the first case it occurs during regeneration, while in the second case it occurs during the absorption stage. The main products are sulfur (70%) and thiosulfates.

Oxidative processes exhibit high selectivity toward H_2S and are applied for gas streams with a low $\text{H}_2\text{S}:\text{CO}_2$ ratio. Their disadvantages include low sulfur loading capacity of the absorbent and high energy consumption during the product recovery stage. Therefore, they are not practically used when sulfur loading exceeds 10 tons per day.

The third type is based on the use of strong oxidizing agents (potassium dichromate and potassium permanganate), where oxidation of H_2S results in the formation of sulfate. These methods are mainly used in laboratory conditions or in small-scale specialized purification processes.

The process consists of the following main units: contact unit (1), oxidation unit (2), settling tank for slurry separation (3), storage vessel (4), filtration unit (5), filtration and autoclave unit (6), centrifugation unit (7), and centrifugation and heating unit (8). The sulfur-containing gas (stream I) is introduced into the contact unit, where hydrogen sulfide is absorbed and converted into soluble compounds. The purified gas (stream II) exits the system after treatment.

Air (stream III) is supplied to the oxidation unit to regenerate the solution and promote the conversion of sulfide species into elemental sulfur. The formed sulfur is separated in the settling tank and further processed through filtration and centrifugation stages. Solid sulfur (stream IV) is obtained as an intermediate product, which can be further treated to produce molten sulfur (stream V) in the heating unit.

This scheme represents the "Stretford" process, which is based on the oxidation of hydrogen sulfide to elemental sulfur and is considered one of the earliest industrial methods for mercaptan removal from gas streams [26]. The process cannot be applied when the total concentration of H_2S and CO_2 in the gas exceeds 0.1 %, since non-regenerable carbonates and sulfides are formed, leading to increased alkali consumption. As the molecular weight of mercaptans increases, their solubility decreases and

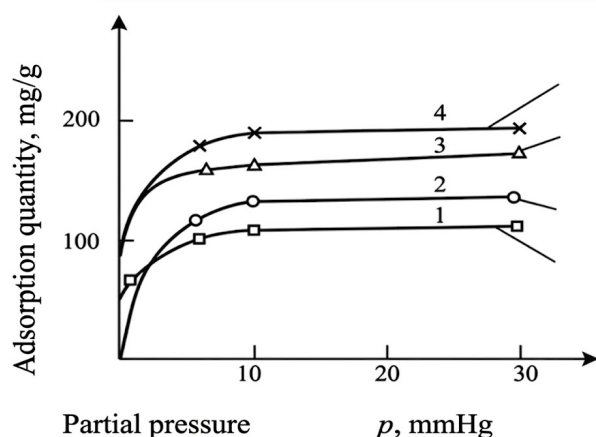


Figure 5. Scheme of alkaline purification of sulfur-containing gas

the purification efficiency declines; therefore, solubilizers are introduced to enhance process performance.

The main conclusions of this study are as follows:

Type A zeolites (4 Å pore size) are widely used in gas drying due to their high adsorption capacity. Kaolin is an important raw material, containing 37–39.5 % Al_2O_3 . Since kaolin reserves are gradually decreasing, the search for alternative raw materials and synthesis routes is of increasing importance.

Zeolite synthesis based on metakaolin recrystallization enables the production of binder-free granular materials.

The radius of secondary pores determines the dynamic activity of adsorbents; control of pore structure, including microwave-assisted treatment, is a key research direction.

Ion exchange of sodium cations with other metal cations allows the preparation of zeolites with tailored properties.

Mercaptan removal processes such as “Yunisol”, “Merox”, and “Solexol” are based on alkaline solutions with organic additives (e.g., methanol and phenols). The efficiency of these processes mainly depends on the regeneration method [27, 28]. The most common regeneration method is steam desorption of mercaptans; however, steam consumption is high (approximately 200 kg/m³) and depends on mercaptan concentration.

When solubilizers are applied, steam consumption increases by 20–30 %. The most efficient approach is catalytic regeneration, where mercaptans are oxidized to disulfides in the presence of air and oxygen-carrying catalysts. Metal phthalocyanines of groups V–VIII exhibit high catalytic activity in this process. The process flow scheme is presented in Figure 5.

The process includes the following main units: alkaline absorber (1), separators (2–3), regenerator (4), settler (5), disulfide storage vessel (6), and alkali storage vessel (7). The sulfur-containing gas (stream I) enters the alkaline absorber, where acidic components are removed through interaction with the alkaline solution (stream III) in the presence of a catalyst (stream IV). The purified gas (stream V) exits the absorber after treatment.

The spent solution is directed to the separators, where phase separation occurs, and then to the regenerator for restoration of its activity. Air (stream II) is supplied to support regeneration reactions. The formed disulfides are separated in the settler and collected in the storage vessel (6). The regenerated alkaline solution is recycled back to the absorber, while air for combustion (stream VI) may be used in auxiliary thermal processes.

Chemisorption purification methods

Chemisorption of hydrogen sulfide is typically carried out using heavy metal oxides such as Fe, Cu, Zn, and Mn [28, 29]. The process occurs in the temperature range of 20–400 °C, leading to the formation of metal sulfides and, in some cases, elemental sulfur. The sulfur ca-

capacity of these sorbents generally does not exceed 20 %, and effective regeneration methods are not available for their reuse. COS and CS₂ form thiocarbonates, while mercaptans form mercaptides; however, the removal efficiency of organic sulfur compounds remains relatively low.

For this reason, organic sulfur compounds are first converted into H₂S at 300–400 °C using Co–Mo or Mo–Ni catalysts and subsequently removed using ZnO. Bifunctional catalysts capable of performing both steps in a single stage have also been developed, where $\gamma\text{-Al}_2\text{O}_3$ serves as the catalytic support and ZnO acts as the sorbent [29]. Additional metal oxides such as Cr, Cu, and Mo enhance catalytic activity and suppress side reactions. Due to high energy consumption and operational costs, these methods are mainly applied for deep (final) gas purification.

In gas drying processes, zeolite adsorbents play a crucial role, enabling dew point temperatures below –30 °C. Type A zeolites (KA–3Å, NaA–4Å, CaA–5Å) are widely used for this purpose. For drying gases containing unsaturated hydrocarbons, KA zeolites with small pore sizes and high ion-exchange capacity are applied [30, 31].

For separation of CO and CO₂, 4 Å (NaA) zeolites are used, while 5 Å (CaA), X, Y, and mordenite zeolites are applied for the removal of SO₂, H₂S, and other acidic components. These adsorbents are also widely used in the production of high-purity gases such as nitrogen and argon [24].

Adsorptive separation of simple gas mixtures (e.g., O₂–N₂ and O₂–Ar) is challenging due to their low polarity. To improve selectivity, ion exchange, dealumination, and pre-adsorption of polar molecules (e.g., water) are applied. These approaches enhance separation efficiency by modifying adsorption heat [25].

Kaolin is the primary raw material for the synthesis of type A zeolites, containing 37–39.5 % Al_2O_3 . Due to the depletion of kaolin reserves, the search for alternative raw materials and improved synthesis routes remains an important research direction.

Adsorption processes and their industrial importance

Adsorption of sulfur-containing compounds has been extensively studied using activated carbons, silica gels, alumina gels, and synthetic zeolites [28, 29, 32–35]. However, except for synthetic zeolites, most adsorbents exhibit low sulfur capacity due to co-adsorption of heavy hydrocarbons.

Modification of activated carbon using alkalis, metal salts, and other reagents has not shown significant industrial effectiveness, as adsorption capacity decreases rapidly [35, 36]. Therefore, synthetic zeolites remain the dominant materials in industrial applications.

Zeolites exhibit high selectivity toward polar molecules and possess molecular-sieve properties. Their porous structure consists of cavities of molecular dimensions connected by narrow channels (“windows”), the size of which depends on the zeolite type (A, X, Y) and the radius of exchangeable cations (Na^+ , K^+ , Ca^{2+}).

The molecular-sieve properties of zeolites are summarized in Table 3, which presents the adsorption capacity of CaA and NaX zeolites for isoamyl mercaptan.

The figure presents the adsorption isotherms of ethyl mercaptan on various zeolite types, including CaX (1), NaA (2), CaA (3), and NaX (4). The results indicate that the adsorption capacity depends significantly on the zeolite structure and cation composition. Among the studied samples, CaX exhibits the highest adsorption performance, which can be attributed to its larger pore size and higher affinity toward sulfur-containing compounds.

In contrast, NaA and CaA show relatively lower adsorption capacities due to their smaller pore structures, which limit the diffusion of ethyl mercaptan molecules. NaX demonstrates intermediate behavior, reflecting differences in cation type and adsorption site availability. These findings highlight the importance of selecting appropriate zeolite grades for efficient removal of mercaptans from gas streams.

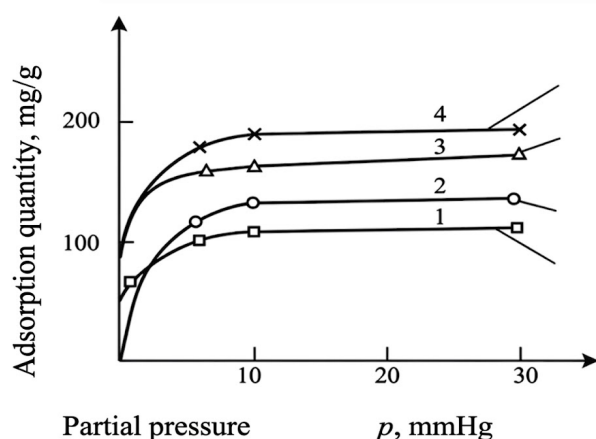
Absorption processes are economically efficient for purification of H₂S, CO₂, and organic sulfur compounds in high-pressure gases, and are particularly advantageous when the partial pressure of acidic components exceeds 4–6 atm. However, the high solubility of hydrocarbons

Table 2. Adsorption capacity of natural gas components on different types of zeolites

Zeolite type	Pore size, Å	Adsorbed molecules	Non-adsorbed molecules
KA	3	H ₂ O, CH ₄	Ethane and larger molecules
NaA	4	The above, CO ₂ , C ₂ H ₄ , CH ₃ SH	Propane and larger molecules
CaA	5	The above, n-hydrocarbons, n-thiols	Iso-compounds
NaX	9	The above, iso-hydrocarbons, iso-thiols	—

Table 3. Isoamyl mercaptan adsorption on CaA and NaX zeolites at 25°C

Partial pressure of isoamyl mercaptan, mmHg	Equilibrium adsorption capacity, mg/g (CaA)	Equilibrium adsorption capacity, mg/g (NaX)
$5 \cdot 10^{-3}$	—	17,7
$2 \cdot 10^{-2}$	—	124
4	2,8	198
10	6,94	209
15	16,5	219

**Figure 6.** Adsorption isotherms of ethyl mercaptan on zeolites of different grades

complicates the regeneration process and reduces the quality of the sulfur product.

Oxidative and alkaline processes are based on the conversion of H₂S into elemental sulfur and mercaptans into disulfides, and are carried out in the liquid phase in the presence of catalysts (phthalocyanines). These are applied for low-concentration acidic gases.

In adsorptive dehydration of gases, 3 Å, 4 Å, and 5 Å (CaA) zeolites are widely used in Uzbekistan. KA and zeolon (mordenite) are effective for nitrogen drying, while reduced diffusion has been observed in KA zeolite [2]. NaA zeolite can adsorb up to 19 % water and achieves a dew point of -72 °C [30].

In the separation of argon–oxygen mixtures, NaA, X, and Y zeolites are applied, and cation exchange significantly increases selectivity. Zn-, K-, and NaA-form zeolites do not adsorb N₂ and Ar, but show adsorption toward n-butane. CuY and (Cu,Na)-Y zeolites exhibit strong adsorption of O₂ [39]. The reducibility of cations follows the order Ni > Co > Cr [40].

Zeolites are also used in ion-exchange processes for water softening, purification of solutions, and dehydration of alcohols in CO₂-containing systems. NaA, NaX, and NaP zeolites effectively bind Ca²⁺ ions [41].

In agriculture, zeolites improve soil fertility due to their ability to retain water and slowly release ammonia. Studies on clinoptilolite and mordenite have demonstrated a positive effect on grain protein content [42, 43].

Specific features of zeolite adsorbents, their production and modification

Synthetic zeolite NaA has the composition Na₂O·Al₂O₃·2SiO₂·4.5H₂O, and its ideal crystal structure is represented as Na₁₂[(AlO₂)₁₂(SiO₂)₁₂] · 27H₂O. Its production is typically carried out by crystallization of hydrogels or by synthesis of metakaolinite in an alkaline medium in granular form. Both methods are accelerated by seed crystals; for example, NaY zeolite crystallizes rapidly in the presence of NaX seed crystals [7].

The main raw materials in the synthesis mixture are sodium silicate and sodium aluminate, while the SiO₂/Al₂O₃ and Na₂O concentrations determine the type of zeolite formed. A-type zeolite forms at a ratio of approximately 0.33:2.0, whereas X and Y zeolites are formed under higher SiO₂ content conditions [44]. The hydrogel product is obtained as 2–4 μm powder, which is subsequently granulated into zeolite adsorbents with the addition of 15–20 % binder [40].

Separation of powder zeolites is difficult and requires centrifugation. Sodium silicate is available as a raw material, but sodium aluminate is lacking in Uzbekistan; therefore, kaolins and galloysites are the main sources.

In kaolins, if kaolinite is highly dispersed ($\leq 2 \mu\text{m}$) and low in quartz, in primary kaolins quartz and cristobalite are more abundant (particle size $\leq 5 \mu\text{m}$). Secondary kaolins are characterized by low crystallinity and higher impurities (for example, gypsum up to 5%).

Structure, composition, physicochemical properties, and application areas of natural zeolites

Natural zeolites are framework aluminosilicates expressed by the general formula (M₂, M')O·Al₂O₃·nSiO₂·mH₂O, where M represents Na⁺, K⁺, Li⁺ ions and M' represents Ca²⁺, Mg²⁺, Ba²⁺ ions; n ≥ 5 and m corresponds to the amount of crystalline water.

Zeolites consist of interconnected SiO₄ and AlO₄ tetrahedra, in which exchangeable cations compensate for the negative framework charge. More than 30 types of natural zeolites have been identified [44, 45].

The main characteristic of zeolites is the molecular-sieve effect, where only molecules smaller than the pore openings can be adsorbed [46]. Natural zeolites have also formed the basis for the development of synthetic zeolite technologies [47].

X-ray diffraction (XRD) methods developed in recent years have enabled the identification of volcanic-sedimentary rocks containing zeolite phases up to 90 %. Large natural zeolite deposits have been identified in the USSR, USA, Japan, Hungary, Bulgaria, Cuba, Yugoslavia, Italy, and France [48].

Zeolite deposits in the territory of the former USSR were extensively studied by A.E. Fersman and are located in Transcaucasia, Crimea, Siberia, Kamchatka, Sakhalin, and the Ural regions [49]. Georgian zeolites were thoroughly investigated in the works of L.A. Tvalchrelidze and G.V. Gvakharia [50, 51]. Although the total reserves have not been fully evaluated, clinoptilolite and mordenite reserves in the Transcaucasian region are estimated to reach hundreds of millions of tons [52].

The major portion of natural zeolite reserves consists of clinoptilolite. Clinoptilolite tuffs exhibit high water vapor adsorption capacity under conditions of P/Ps = 0.4 and t = 20 °C (see Table 6).

Table 4. Classification of natural zeolites according to structural characteristics

Structural groups and zeolites	Idealized chemical composition	Number of tetrahedra forming the window	Window size (Å)
Analcime group			
Analcime	$\text{Na}(\text{AlSi}_2\text{O}_6) \cdot \text{H}_2\text{O}$	-	-
Natrolite group			
Natrolite	$\text{Na}_2\text{Al}_2\text{Si}_3\text{O}_{10} \cdot 2\text{H}_2\text{O}$	8	2,6-3,9
Thomsonite	$\text{NaCa}_2(\text{Al}_5\text{Si}_5\text{O}_{20}) \cdot 6\text{H}_2\text{O}$	8	2,6-3,9
Chabazite group			
Chabazite	$\text{Ca}(\text{Al}_4\text{Si}_8\text{O}_{24}) \cdot 13\text{H}_2\text{O}$	8	3,7-3,5
Gmelinite	$\text{Na}_2(\text{Al}_2\text{Si}_4\text{O}_{12}) \cdot 6\text{H}_2\text{O}$	12	6,9
Erionite	$(\text{Ca}, \text{K}_2, \text{Mg})_4 \cdot 5(\text{Al}_9\text{Si}_{27}\text{O}_{72}) \cdot 27\text{H}_2\text{O}$	8	3,6-4,8
Phillipsite group			
Phillipsite	$(\text{K}, \text{Na}_5)(\text{Al}_5\text{Si}_{11}\text{O}_{32}) \cdot 10\text{H}_2\text{O}$	8	2,8-4,8
Geylandite group			
Stilbite	-	8	2,4-6,1
Mordenite group			
Mordenite	$\text{Na}(\text{AlSi}_5\text{O}_{12}) \cdot 3\text{H}_2\text{O}$	12	6,7-7,0
Faujasite group			
Faujasite	$(\text{Na}_2, \text{Ca})_{30}(\text{Al}_{60}\text{Si}_{132}\text{O}_{384}) \cdot 260\text{H}_2\text{O}$	12	7,4

Table 5. Amount of water vapor adsorption by clinoptilolites from different deposits

Adsorbate (adsorbed substance)	0 (Initial state)	0,25 n.	1,0 n.	2,0 n.	5,0 n.	10,0 n.
H ₂ O (Water)	12,30	12,20	11,40	11,10	9,90	8,40
C ₆ H ₆ (Benzene)	1,24	4,13	4,66	4,09	4,13	2,93
n-C ₆ H ₁₄ (n-Hexane)	1,28	4,16	5,23	4,70	4,60	3,14

Kaolinite ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) is a natural aluminosilicate mineral formed as a result of the weathering of feldspar- and mica-rich rocks. Its typical chemical composition is $\text{Al}_2\text{O}_3 - 39.50\%$, $\text{SiO}_2 - 46.55\%$, and $\text{H}_2\text{O} - 13.95\%$. The crystal structure corresponds to a 1:1 layered silicate, in which tetrahedral and octahedral sheets are linked by hydrogen bonding [40]. At temperatures of 480–590 °C, kaolinite undergoes dehydroxylation with the loss of structural water.

In natural kaolin deposits, quartz content may reach up to 60 %, while iron-containing minerals may be present up to 5 %. The mineralogical composition varies significantly depending on the geological origin of the deposits [53].

Modification of natural zeolites

Natural zeolites are widely used in agriculture to improve the efficiency of mineral fertilizers and reduce nutrient leaching. They are also applied as carriers for pesticides [43].

However, the main limitation of natural clinoptilolite is its relatively small pore opening (≈ 4 Å) and low internal pore volume, which restrict its adsorption capacity. Therefore, zeolites are commonly modified by chemical treatment methods, including alkali, acid, and salt solutions [52, 54–56, 35]. Treatment with NH_4Cl , ZnCl_2 , and MnCl_2 at 90–100 °C for approximately 6 h has shown favorable results.

In current studies, acid modification plays a significant role. During this process, decationization and dealumination occur, leading to the formation of Brønsted and Lewis acid sites, which enhance the catalytic activity of zeolites. These acid sites remain stable in the temperature range of 200–500 °C.

During dealumination, Si–O–Si bonds become more stabilized, while aluminum species transform into under-coordinated states. This mechanism was first proposed by Barrer and Mackie and later confirmed by various experimental techniques. The effects of acid modification on clinoptilolite properties have also been extensively investigated [57, 58].

Clay fraction (<60 μm): 52–60%, Sandy fraction: 40–48%

Beneficiated kaolin typically consists of 85–98 % kaolinite. The main impurities include leucoxene, carbonates, hydromicas, microcline, and iron hydroxides.

Kaolins from the Eluvial Prosyantovsk deposit are characterized by a kaolinite content of 90–95 % in the clay fraction. Minor impurities such as gibbsite, leucoxene, halloysite, and others are present only in individual samples [59]. The chemical composition of these kaolins is given as follows (%):

For the Kishtym deposit, data on the chemical composition of raw and beneficiated kaolin are also available [53].

Information on mineral impurities in kaolins from the Kishtym deposit is not provided in the literature [53]. From the above data on the composition of kaolins from known deposits widely used as raw materials for the production of A-type zeolites and synthetic faujasites, it can be concluded that, in terms of mineral composition, kaolins from the Prosyantovsk and Glukhovets deposits are the purest. For the unbeneficiated — eluvial and redeposited — kaolins of the Angren deposit, according to various data, the average chemical composition (%) is known:

Currently, information on beneficiated primary Angren kaolins and their chemical and granulometric composition is reported in the literature [60]. The mineral composition of Angren kaolins includes siderite (FeCO_3), barite (BaSO_4), pyrite (FeS_2), goethite (FeOOH), chlorites, feldspars, and other associated minerals. In addition, mixed-layer structures containing montmorillonite and hydromica components are also present [31].

In Uzbekistan, the Angren deposit contains large kaolin reserves estimated at 962.864 million tons [53]. Currently, the AKF-78 concentrate obtained from primary kaolins is used as a raw material for zeolite A synthesis, while secondary kaolins have not yet been fully industrially utilized.

The synthesis of zeolites A and X is typically carried out using kaolinite or sodium aluminate, where the raw material composition, particu-

Table 6. Chemical composition of raw kaolin from the Eluvial Glukhovets deposit (%)

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	ΣR ₂ O	SO ₂	Others
46,09-63,20	24,99-39,26	0,05-4,27	trace- 4,72	0,3- 6,5	Trace- 0,22	Trace- 0,36	Trace - 0,27	13,2 – 13,78

Table 7. Chemical composition of beneficiated Glukhovets kaolin (%)

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	ΣR ₂ O	SO ₂	Others
45,9-49,3	35,9– 39,3	Izlar-4,6	0,3-4,7	Trace - 0,9	0–1,2	Trace - 1,0	-	12,8- 13,8

Table 8. Chemical composition of raw and beneficiated kaolins (%)

Raw kaolin	Raw kaolin	Raw kaolin	Raw kaolin	Raw kaolin	Raw kaolin	Raw kaolin	Raw kaolin	Raw kaolin
SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	ΣR ₂ O	SO ₂	Others
49,60-55,80	24,00-38,41	0,48-0,50	0,21– 0,46	0,20 – 0,68	0,13– 0,68	0,5–0,6	0,03–0,12	8,80–12,8
Beneficiated kaolin	Beneficiated kaolin	Beneficiated kaolin	Beneficiated kaolin	Beneficiated kaolin	Beneficiated kaolin	Beneficiated kaolin	Beneficiated kaolin	Beneficiated kaolin
SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	ΣR ₂ O	SO ₂	Others
46 -47	36 – 38,5	0,1–1,4	0,1– 0,7	0,3– 0,6	-	0,5 – 0,6	-	12,5–14,0

Table 9. Chemical composition of raw and beneficiated kaolins from the Kishtym deposit (%)

Raw kaolin	Raw kaolin	Raw kaolin	Raw kaolin	Raw kaolin	Raw kaolin	Raw kaolin	Raw kaolin	Raw kaolin
SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	K ₂ O	SO ₂	Others
65,0	23,39	0,85	0,59	0,35	-	0,16	-	7,86
Beneficiated kaolin	Beneficiated kaolin	Beneficiated kaolin	Beneficiated kaolin	Beneficiated kaolin	Beneficiated kaolin	Beneficiated kaolin	Beneficiated kaolin	Beneficiated kaolin
SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	K ₂ O	SO ₂	Others
45,52	36,99	0,62	0,46	0,08	0,03	0,17	-	8,65

Table 10. Average chemical composition of kaolin (%)

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	K ₂ O	SO ₂	Others
67,92	20,7	1,4	0,87	0,33	0,67	0,81	0,62	7,98

larly the SiO₂/Al₂O₃ ratio, determines the crystallization pathway [61, 7]. Cation exchange significantly modifies zeolite adsorption properties; for instance, the KA form exhibits negligible adsorption of CO₂, N₂, and hydrocarbons [62].

For zeolite synthesis based on kaolinite, metakaolin suspensions in NaOH solution are used, and under autoclave conditions, crystallization leads to the formation of NaA zeolite, hydrocancrinite, and other phases [63]. Zeolite-hydroxide composite adsorbents have also been reported as effective materials for CO₂ adsorption [7]. Acid modification induces decationization and dealumination in zeolites, resulting in changes in porosity and adsorption properties [52, 54]. Under controlled conditions, porosity increases and adsorption activity improves, whereas high acid concentrations may destroy the crystalline framework.

In general, the application of natural zeolites for gas purification is expanding; however, their performance is still mainly limited to molecules up to 4 Å. Therefore, the development of highly efficient adsorbents through chemical modification remains an important research direction [35, 52]. These findings strongly align with our long-term research on the solubility of acid components (H₂S and CO₂) and the reduction of steel corrosion in advanced composite absorbent formulations [64-70].

Conclusion

According to the literature analysis, there are various methods for purification of natural, secondary, and waste gases from sulfur compounds and moisture, each having its own advantages and disadvantages. The selection of optimal technology depends on economic efficiency, con-

centration of incoming impurities, temperature-pressure conditions, and environmental requirements.

Adsorption methods based on LTA-type 5Å zeolites are considered promising for gas drying and purification due to their high selectivity, large sorption capacity, and stable properties. Their main advantages include technological simplicity, effective adsorption of moisture and harmful components, and the possibility of regeneration and reuse.

Process efficiency depends on the crystal structure of the zeolite, Si/Al ratio, porosity, and regeneration conditions; therefore, the development of highly efficient and low-cost adsorbents and improvement of regeneration technologies are important.

Synthesis of LTA-type zeolites based on local raw materials is environmentally and economically feasible and contributes to the development of import-substituting technologies.

In general, the development of adsorption technologies based on LTA zeolites for natural gas purification is a relevant scientific and practical direction.

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