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MODERN APPROACHES TO IMPROVING THE EFFICIENCY OF GAS PURIFICATION FROM ACIDIC COMPONENTS

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Abstract. The article presents scientific research on the preparation and application of compositions with the necessary concentration of activators and inhibitors in the absorbent to reduce the corrosion rate and enhance the complete absorption of acidic components from gases. The effectiveness of a new absorption composition containing diethylolamine (DEA), hexamine, and carbohydrazide in purifying gases from acidic components has been determined. According to the studies conducted, the activation of absorbents with activators allows for more complete absorption of acidic components from gases, reduces the formation of heat-resistant salts, thereby reducing the corrosion observed in devices and preventing excessive accumulation in the absorber.

Keywords: gas chromatography, composite absorbent, diethylamine (ДЭА), hexamine ($C_6H_{12}N_4$), carbohydrazide ($OC(N_2H_3)$), carbon dioxide (CO_2), hydrogen sulfide (H_2S), carbon sulfide (COS), carbon disulfide (CS_2), mercaptans (RSH), thiophenes (C_4H_4S).

Аннотация. В статье представлены научные исследования по приготовлению и применению композиций с необходимой концентрацией активаторов и ингибиторов в абсорбенте для снижения скорости коррозии и повышения полноты поглощения кислых компонентов из газов. Определена эффективность новой абсорбционной композиции, содержащей диэтилоламин (ДЭА), гексамин и карбогидразид, при очистке газов от кислых компонентов. Согласно проведенным исследованиям, активация абсорбентов активаторами позволяет добиться более полного поглощения кислых компонентов из газов, уменьшает образование термостойких солей, тем самым уменьшая коррозию, наблюдаемую в устройствах, и предотвращая чрезмерное накопление в абсорбере.

Ключевые слова: газовая хроматография, композитный абсорбент, диэтиламин (DЭА), гексамин (C6H12N4), карбогидразид (OC(N2H3)), диоксид углерода (CO2), сероводород (H2S), сульфид углерода (COS), сероуглерод (CS2), меркаптаны (RSH), тиофены (C4H4S).

Annotatsiya. Maqolada korroziya tezligini pasaytirish va gazlardan kislotali komponentlarning to'liq so'rilishini kuchaytirish uchun changni yutish vositasida

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aktivatorlar va ingibitorlarning kerakli konsentratsiyali kompozitsiyalarni tayyorlash va qo'llash bo'yicha ilmiy tadqiqotlar keltirilgan. Tarkibida dietilolamin (DEA), geksamin va karbogidrazid bo'lgan yangi absorbsion kompozitsiyaning gazlarni kislotali komponentlardan tozalashdagi samaradorligi aniqlandi. O'tkazilgan tadqiqotlarga ko'ra, absorbentlarni aktivatorlar bilan faollashtirish gazlardan kislotali komponentlarni to'liqroq singdirish imkonini beradi, issiqlikka chidamli tuzlarning hosil bo'lishini kamaytiradi, shu bilan qurilmalarda kuzatiladigan korroziyani kamaytiradi va absorberda ortiqcha to'planishning oldini oladi.

Kalit so'zlar: gaz xromatografiyasi, kompozit absorbent, dietilamin (RSH), geksamin (C6H12N4), karbogidrazid (oC(N2H3)), karbonat angidrid (CO2), vodorod sulfidi (H2S), uglerod sulfidi (cos), uglerod disulfidi (CS2), merkaptanlar (RSH), tiofenlar (c4h4s).

1. INTRODUCTION.

At present, due to the rapid development of industry and manufacturing, it has become evident that the direct combustion of hydrocarbon gases is an inefficient use of resources. Modern trends in the oil and gas industry focus on the deep processing of gases to obtain valuable products, which in turn ensures the efficient utilization of raw materials considered highly economically advantageous and meets contemporary requirements [1].

Like any other raw material, hydrocarbon gases cannot be used directly — their application requires preliminary purification, processing, and separation into fractions using appropriate technologies [2–3].

Globally, during the purification of hydrocarbon gases from acidic components, alkanolamine-based absorbents play a leading role. Aqueous solutions of diethanolamine (DEA) of various concentrations are considered particularly effective and are widely used for the removal of acidic compounds, especially hydrogen sulfide, from gases. However, gas compositions also contain other acidic components such as carbon dioxide (CO_2), carbonyl sulfide (COS), carbon disulfide (CS_2), mercaptans (RSH), and thiophenes [4–5].

2. MATERIALS AND RESEARCH METHODS.

At present, in order to enhance the complete absorption of acidic components from gases, scientific research aimed at developing and applying compositions consisting of mixtures of activators, corrosion inhibitors, and antifoaming additives within absorbents is of great importance.

Activators added to alkanolamine absorbents include hexamine, piperazine, methanol, phenol, ethanolamine, arsenic acid, glycine, and substituted amines. Corrosion inhibitors that slow down the corrosion rate include carbohydrazide, glycols (MEG/TEG), methyldiethanolamine (MDEA), as well as mixtures of

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glycols (MEG/TEG) with tertiary amines. The introduction of these components into the absorbent further enhances its absorption capacity and stability, reduces concentration losses, and decreases corrosion rates and excessive foaming in the absorber [15].

The study began with an analysis of the composition of the raw gas supplied to the Shurtan Gas Chemical Complex. The composition of the feed gas was determined based on the average result of gas chromatographic analysis over one month. According to the obtained data, the CO_2 content was 3.36%, and H_2S — 0.093%. These indicators exceed the standard values established by the process regulations, which negatively affect the efficiency of amine absorption purification.

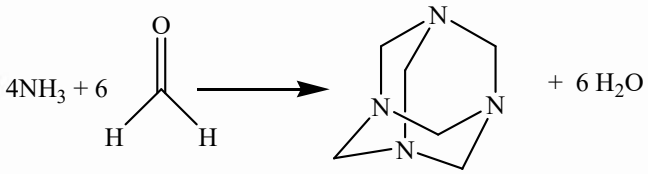
The gas purified at the Shurtan Gas Chemical Complex is subsequently directed as follows: the methane fraction of hydrocarbons is used as feedstock at the GTL (Gas-to-Liquid) plant to produce synthetic fuels, while the ethane fraction serves as raw material for polymer production within the complex itself. The purity of these gas compositions is of primary importance for their chemical and catalytic processing.

To improve the technical and economic performance of hydrocarbon gas purification processes from acidic components by reducing operating costs, a modified version of the absorption process using diethanolamine (DEA) was developed.

Accordingly, a composite absorbent was formulated based on industrially used diethanolamine (DEA) and activators such as hexamine and carbohydrazide.

Diethanolamine (DEA) — $NH(C_2H_2OH)_2$ — possesses the following physicochemical properties: it is a liquid ranging from colorless to yellowish in appearance, with a characteristic odor. It is well soluble in both water and fats. The hydrogen ion index (pH) is 11 at a concentration of 100 g/L in water at 20 °C. The density at 20 °C is 1.0959 g/cm³.

Hexamine (HMTA) — $C_6H_{12}N_4$ — is a white crystalline powder formed by the reaction of 4 moles of ammonia with 6 moles of formaldehyde [45; 48–62 pp.]. In terms of structural organization, the compound resembles adamantane, which gives it high thermal stability and chemical resistance.

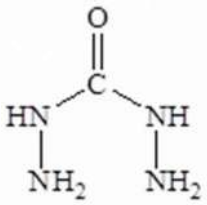


Based on toxicological assessments of hexamine exposure, it has been established that under various application methods, no long-term health risks have been identified, and the current level of exposure to the substance is considered

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safe. Hexamine functions as a corrosion inhibitor, preventing hydrogen-induced corrosion of equipment.

Carbohydrazide (hydrazine, 1,3-diaminourea; carbonyl dihydrazide) — molecular formula $CO(NHNH_2)_2$ — is a white crystalline powder with a purity of 98.0%. It is typically used as a 10% aqueous solution with a pH of 8.45. Carbohydrazide is highly soluble in water and alcohol and is applied as a corrosion inhibitor, particularly in systems where water and elevated temperatures are present.



An experimental setup was prepared, with its parameters adapted to industrial operating conditions. The process of gas absorption purification was carried out using various composite absorbents. Tests were conducted with seven samples, during which the purified gas and the saturated absorbent were collected in separate containers for subsequent analysis (Table 1) [16–18].

Table 1.

Composition of absorbent mixtures based on DEA, hexamine, and carbohydrazide for the purification of gases from acidic components

№	Name of absorbent	Water content in the composition, %	Concentration of components, %		
			DEA	Hexamine	Carbohydrazide
1	DEA, Sample 1 (for comparison)	70	30	-	-
2	Sample 2 with DEA	67,975	30	2	0,025
3	Sample 2 with DEA	65,975	30	4	0,025
4	Sample 2 with DEA	65,975	32	2	0,025
5	Sample 2 with DEA	63,975	32	4	0,025
6	Sample 2 with DEA	63,975	34	2	0,025
7	Sample 2 with DEA	61,975	34	4	0,025

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When preparing the above-mentioned absorbent composition, the selection of chemical reagents was primarily based on the local origin of raw materials, their degree of efficiency, as well as on the shortcomings observed in the existing technological scheme.

3. RESULTS AND ANALYSIS

To ensure the efficient performance of the diethanolamine (DEA) absorbent in the process of gas absorption purification, regular laboratory analyses are carried out. This is necessary because, under the influence of process parameters — such as pressure, temperature, environment, chemical reagents, and gas composition — chemical changes may occur in the absorbent, causing it to lose its functional properties.

Taking this into account, the following studies were conducted on DEA-based absorbent compositions used for the purification of natural gases from CO_2 and H_2S:

- determination of carbon dioxide (CO_2) content;
- determination of hydrogen sulfide (H_2S) mass in saturated and regenerated diethanolamine solutions by titration;
- determination of the amount of acid–thermally stable salts;
- determination of the foaming tendency of the absorbents;
- determination of concentration;
- determination of H_2S content in DEA;
- measurement of the pH value of DEA;
- determination of DEA turbidity;
- determination of DEA viscosity.

These analyses made it possible to evaluate the physicochemical stability of the absorbent, the efficiency of its performance, and the changes in its properties during operation and regeneration. The amount of carbon dioxide in the DEA composition samples was determined using an Orsat apparatus (INV 12147) in accordance with Enterprise Standard 39.2-36-201 / ASTM D-513.

Based on the obtained results, it can be concluded that an increase in diethanolamine concentration and the addition of an activator lead to a higher degree of carbon dioxide removal. However, a further increase in activator concentration does not have a significant effect on the amount of absorbed carbon dioxide. The composition containing 2% activator and 32% DEA concentration differed from the others in that during regeneration, minimal absorbent concentration losses were observed [19–21].

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Table 2.

Results of Determining the CO_2 Content in Saturated DEA Solutions in Various Units of Measurement

No	Samples	mg	wt.%	g/L	mole %	mole/ L	mole/mole DEA
	CO_2 Indicator According to Orsat		CO_2	CO_2	CO_2	CO_2	CO_2
1	DEA, Sample 1 (for comparison)	82,0	5,783	64,429	3,324	1,464	0,495
2	Sample 2 with DEA	83,0	5,848	64,429	3,342	1,482	0,499
3	Sample 2 with DEA	84,0	5,929	65,214	3,388	1,501	0,503
4	Sample 2 with DEA	91,0	6,418	70,592	3,392	1,625	0,505
5	Sample 2 with DEA	91,0	6,418	70,592	3,392	1,625	0,505
6	Sample 2 with DEA	91,4	6,446	70,900	3,399	1,642	0,498
7	Sample 2 with DEA	91,9	6,475	71,219	3,423	1,660	0,499

The main issue lies in the degree of CO_2 absorption by the absorbent, while the analysis of the absorption degree with the activator was compared and illustrated in the graph (Figure 1).

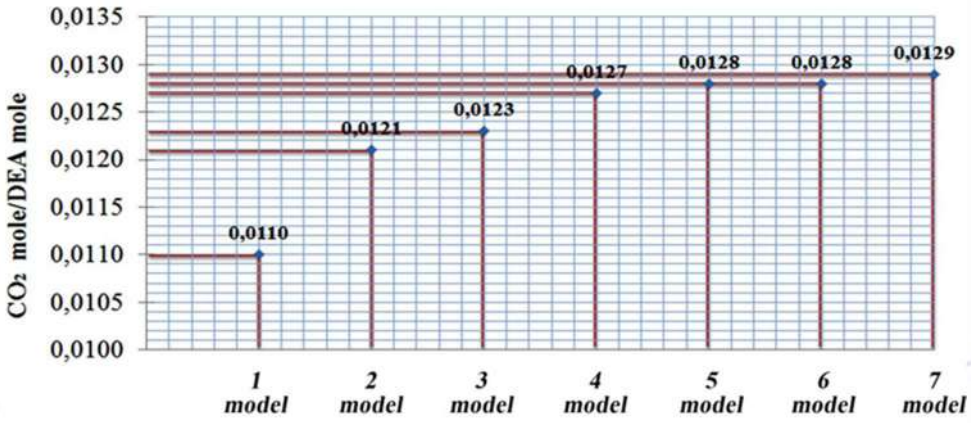


Figure 1. Degree of CO_2 Absorption by Saturated DEA Samples, CO_2 mole/mole DEA

Sample No. 1 represented the DEA solution used at the plant without the addition of any activators. According to the results obtained for Sample No. 1 and other composite samples, in compositions with an increased concentration of

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diethanolamine and the addition of an activator, the degree of carbon dioxide absorption increased.

Another characteristic feature of amines is the concentration of carbon dioxide remaining in the DEA composition after regeneration. In practice, a decrease in DEA concentration is observed with each regeneration cycle. To maintain the required concentration in the technological process, regular addition of fresh amine solution is provided. In composite absorbents containing an activator (Sample No. 4), such losses were significantly reduced.

The carbon dioxide content in the desorbed (regenerated) composite DEA samples was also determined and calculated by laboratory methods in various units, similar to the saturated amine solutions (Table 3).

Table 3.

Determination of Carbon Dioxide Content in Desorbed Composite DEA Samples in Various Units of Measurement

No.	Samples	mg	wt.%	g/L	mole %	mole/L	mole/mole DEA
	CO_2 Indicator According to Orsat		CO_2	CO_2	CO_2	CO_2	CO_2
1	DEA, Sample 1 (for comparison)	5,9	0,219	2,121	0,114	0,052	0,017
2	Sample 2 with DEA	5,8	0,214	2,161	0,117	0,051	0,016
3	Sample 2 with DEA	5,5	0,202	2,161	0,117	0,049	0,016
4	Sample 2 with DEA	5,3	0,194	2,157	0,113	0,047	0,014
5	Sample 2 with DEA	5,3	0,193	2,153	0,112	0,047	0,015
6	Sample 2 with DEA	5,3	0,193	2,153	0,112	0,047	0,014
7	Sample 2 with DEA	5,4	0,196	2,158	0,113	0,048	0,014

The amount of carbon dioxide in the desorbed DEA composition sample indicates that the absorbent has been regenerated.

Analyses showed that the carbon dioxide content in the regenerated absorbent samples was lower compared to the control sample. It was found that the added activator influences the rapid and efficient absorption and subsequent release of acidic components by the amine (Figure 2).

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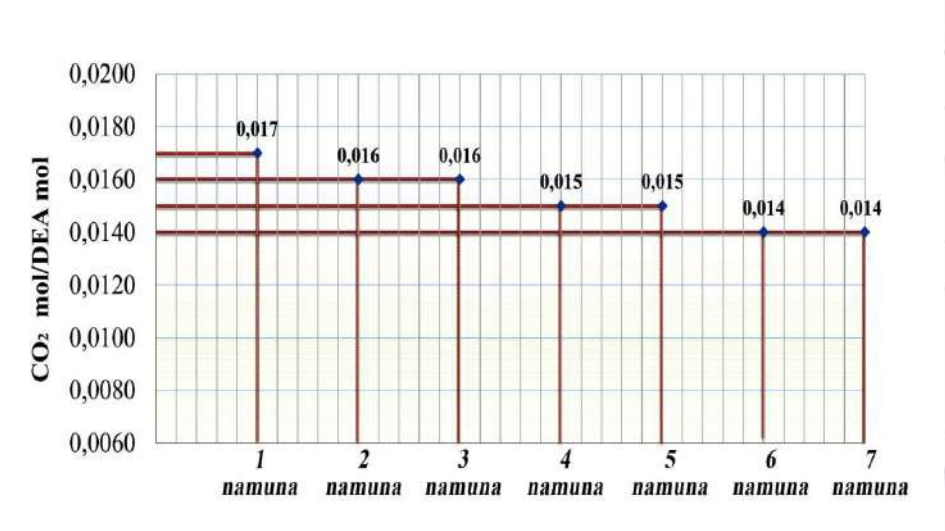


Figure 2. CO_2 Content in Regenerated DEA Composition Samples, CO_2 mole/mole DEA

The essence of the analysis lies in comparing the absorbent's capacity based on its hydrogen sulfide absorption performance. After repeated regeneration of the absorbent, analyses were conducted to determine the amount of hydrogen sulfide remaining in its composition.

The next key indicator is the determination of the hydrogen sulfide content. It can be observed that the absorption capacity of the absorbent compositions containing activators toward H_2S also increased. This is evident from the results obtained for Sample No. 4.

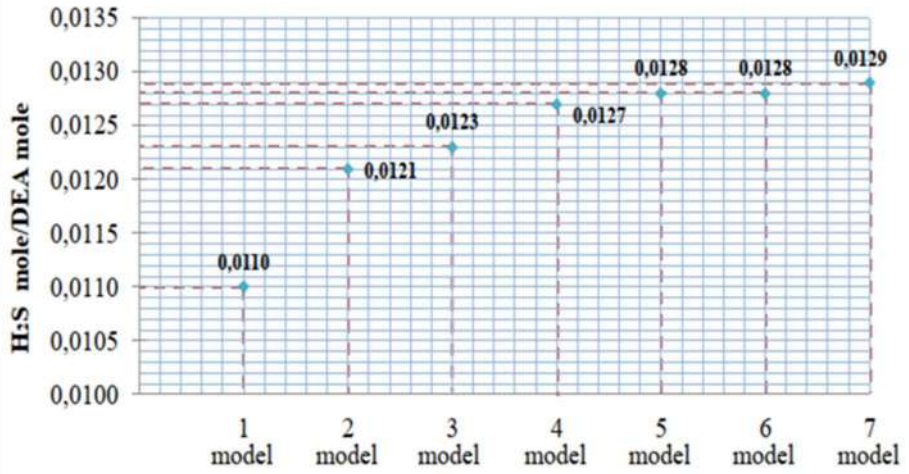


Figure 3. Amount of Absorbed H_2S by Saturated DEA Samples, H_2S mole/mole DEA

The determination of hydrogen sulfide content in the regenerated DEA samples was carried out using the above-mentioned method. However, during the analysis of regenerated DEA, the amine samples were diluted as follows: 10 ml of DEA was mixed with 40 ml of water, after which the resulting 50 ml solution was titrated (Table 5).

Table 5.

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Determination of H_2S Content in Desorbed (Regenerated) DEA Composition Samples in Various Quantities

No.	Samples	mg	wt.%	g/L	mole %	mole/L	mole/mole DEA
	H_2S titrator indicator		H_2S	H_2S	H_2S	H_2S	H_2S
1	DEA, Sample 1 (for comparison)	10,3	0,071	0,0074	0,0071	0,003	0,00062
2	Sample 2 with DEA	9,7	0,067	0,0070	0,0056	0,003	0,00059
3	Sample 2 with DEA	8,7	0,061	0,0062	0,0051	0,002	0,00053
4	Sample 2 with DEA	8,5	0,058	0,0061	0,0043	0,002	0,00052
5	Sample 2 with DEA	8,4	0,058	0,0060	0,0042	0,002	0,00051
6	Sample 2 with DEA	8,3	0,057	0,0059	0,0042	0,002	0,00050
7	Sample 2 with DEA	8,3	0,057	0,0059	0,042	0,002	0,00050

From the analyses determining the amount of hydrogen sulfide remaining in the absorbent after repeated regeneration, it can also be seen that the desorption process has improved (Figure 3.4).

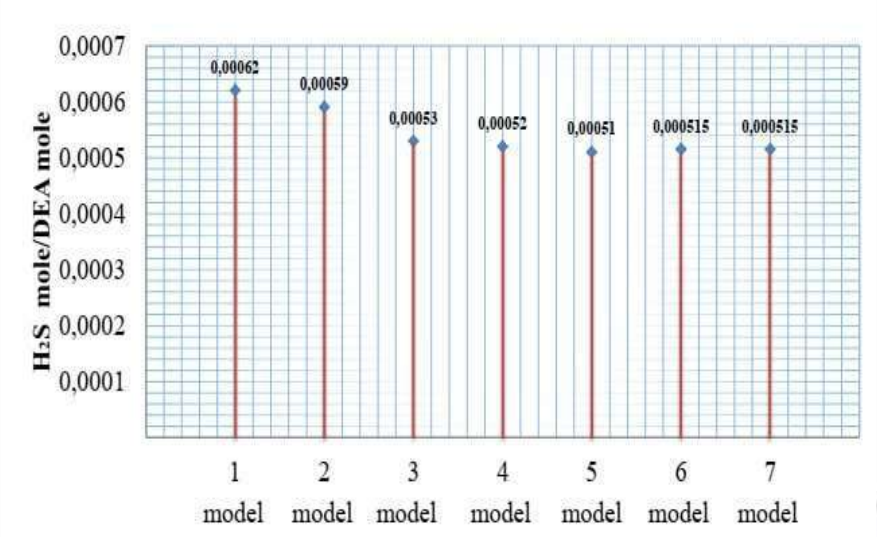


Figure 4. Amount of Residual H_2S in Regenerated DEA Samples, H_2S mol/mol DEA

The titrator readings and the obtained results also showed a decrease in the H_2S content in the DEA composition samples. This was particularly evident in Sample No. 3 and subsequent ones.

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Determination of Thermally Stable Salts in the Absorbents

Thermally stable salts are components formed during the absorption process as a result of the interaction between the absorbent and acidic components, which are not decomposed or removed during desorption (regeneration).

The study was carried out only for the desorbed DEA samples. The amount of thermally stable salts in the DEA samples was calculated using data on the sample density and the volume of alkali consumed for titration (Table 6) [22–23].

Table 6.

Content of Thermally Stable Salts in Regenerated DEA Samples

No.	Absorbent Samples	Sample Density, g/cm³	V, Volume of Alkali Used for Titration, mL	Content of Thermally Stable Salts in DEA Samples, wt.%
1	DEA, Sample 1 (for comparison)	1,055	5,9	0,1118
2	Sample 2 with DEA	1,062	5,8	0,1092
3	Sample 3 with DEA	1,068	5,5	0,1030
4	**Sample 4 with DEA**	**1,073**	**5,4**	**0,1006**
5	Sample 5 with DEA	1,075	5,4	0,1004
6	Sample 6 with DEA	1,077	5.3	0,0984
7	Sample 7 with DEA	1,079	5,4	0,0955

The content of thermally stable salts in the desorbed DEA samples decreased compared to the initial control sample. This is clearly illustrated in the following graph (Figure 5).

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