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INVESTIGATION OF ALKANOLAMINE LOSSES IN THE PROCESS OF NATURAL GAS ABSORPTION PURIFICATION

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Abstract: The article discusses the loss of alkanolamines used as absorbents in the process of purifying natural gas from acid components under the influence of various factors, as well as methods to prevent such losses.

Keywords: hydrocarbon gases, acid components, hydrogen sulfide, carbonyl sulfide, mercaptans, sulfides and disulfides, gas purification.

Introduction

The choice of the process for purifying natural gas from sulfur compounds depends on several factors, the main of which include: the composition and parameters of the raw gas, the required degree of purification, the field of application of the commercial gas, the availability and parameters of energy sources, production waste, and others.

Global practice in the field of natural gas purification shows that, for processing large gas flows, the main processes are absorption methods that utilize chemical and physical absorbents or their combinations. Oxidation and adsorption processes are typically used for purifying small gas streams or for fine purification of gases.

The following requirements are imposed on absorbents used in industry [1, 3]:

- high absorption capacity;
- low vapor pressure;
- thermochemical stability under operating conditions;
- low viscosity, thermal conductivity, and toxicity;
- resistance to foam formation;
- selectivity in absorbing certain components when necessary;
- relatively low cost;
- resistance to side reactions with various impurities.

The most commonly used type of chemisorbents are alkanolamines.

The use of chemical solvents is based on a chemical reaction between the chemisorbent and the acid components.

The maximum absorption capacity of aqueous solutions of chemical absorbents is limited by stoichiometry.

Research Methodology

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Installations for purifying hydrocarbon gases with ethanolamine solutions include at least two column-type units – an absorber and an amine solution regeneration column.

In addition, the installation is equipped with the necessary pumps, heat exchangers, filters, fittings, and other equipment. Amine solution regeneration is often carried out in centralized units within oil refining plants, which significantly improves the economic performance of the process.

In the design of purification units, the main decisions focus on selecting the working amine solution or a mixture of amines, determining the equipment and technological parameters that ensure the required degree of gas purification, protecting against corrosion, addressing the issue of solution foaming, and reducing losses caused by solution carryover and degradation [4, 5].

Amine consumption is one of the key indicators in the operation of gas purification units, since absorbents are expensive, and the cost of absorbents constitutes a major part of the total operational expenses.

The main causes of amine losses in installations include: entrainment with gas, thermochemical degradation of amines, and mechanical losses [2,6].

Losses with Gas. Amine losses with gas occur either in the vapor phase or in the form of droplets. The magnitude of the first type is determined by the vapor pressure of amine above the aqueous mixture and depends on temperature, as well as on the concentration and type of amine in the mixture. Even though the vapor pressure of amines is relatively low, due to the large volume of purified gas, the losses caused by evaporation can be significant.

Losses in the form of droplets depend on the load of gas and liquid mixtures, the efficiency of separation devices in the gas streams, and the type of contact elements used in column apparatuses. Droplet losses increase sharply when the absorbing solution foams. Under normal operating conditions of plant equipment, this indicator of amine loss is about $20–30\ \mathrm{mg/m^3}$ of gas, and during foaming of the solution, it can rise up to $100\ \mathrm{mg/m^3}$

The thermal decomposition of amines proceeds at a relatively low rate in the absence of carbon dioxide (CO_2), but it is enhanced by both CO_2 saturation and increasing temperature. Under the influence of CO_2, the loss of diethanolamine (DEA) is relatively minor; at $100\ °C$ and a pressure of $1.2\ \mathrm{MPa}$, it amounts to about 9%. When a $20\ \mathrm{wt\%}$ aqueous DEA solution saturated with CO_2 is maintained at $125\ °C$ and $1.7\ \mathrm{MPa}$ for $8\ hours$, approximately 22% decomposition occurs. In contrast, when the DEA mixture is heated for $8\ hours$ under a nitrogen atmosphere (without CO_2) at a pressure of $4.1\ \mathrm{MPa}$ and a temperature of $205\ °C$, no significant change in DEA concentration or amine degradation is observed [7–10].

The mechanisms of formation of CO_2 compounds with amines that are difficult or impossible to regenerate have not been fully studied. In the reaction of CO_2 with amines, carbonates and carbamates are first formed, which initially produce oxazolidone-2; this, in turn, temporarily

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forms oxyethylimidazolidone-2. The resulting imidazolidone is then hydrolyzed to oxyethylenediamine.

In DEA (diethanolamine) solutions, oxazolidone can, at high temperatures, transform into another thermal compound – oxyethylpiperazine. Oxazolidone-2 may form even at normal temperatures when carbon dioxide is present in the gas composition.

In amine solutions, compounds containing nitrogen and other complex degradation products with structures that are difficult to identify have been detected.

Discussion and Results

The rate of side reactions is generally low. However, the prolonged circulation of the absorbing solution leads to the accumulation of products that are difficult to regenerate. This, in turn, causes a decrease in the concentration of active amine, reduces the efficiency of the absorption process, and increases the viscosity of the solution. In addition, the presence of amine degradation products promotes foaming of the solution and increases its corrosive aggressiveness.

Studies have shown that the solidification products of DEA slow down its decomposition reactions and that the destruction of DEA is reduced under the influence of hydrogen sulfide.

Due to the complexity of amine degradation processes and reaction kinetics, as well as the side reactions of CO_2 with DEA and MDEA, experimental analyses of the thermochemical degradation of DEA and MDEA were carried out under the influence of CO_2 in autoclaves at temperatures up to 250°C and pressures up to 5 MPa.

$$d[A]/d\tau = -K[A] \qquad (1)$$

Figure 1 shows the dependence of the rate constant K on the rate of amine degradation under the influence of CO_2. The presented data indicate that MDEA has higher thermochemical stability compared to DEA, and this stability increases with rising temperature.

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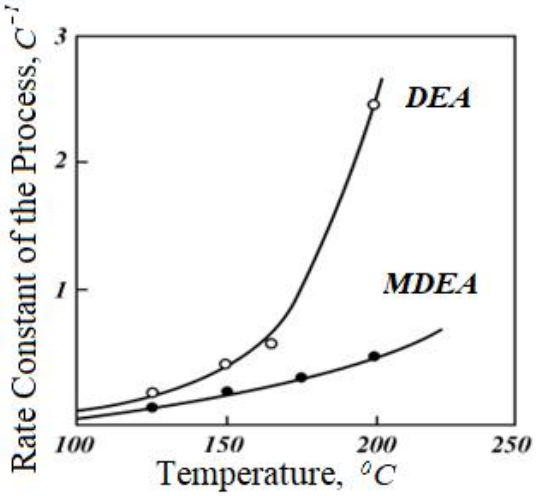

Figure 1. Dependence of the rate constant on temperature during the thermochemical degradation process of alkanolamines at P_{CO_2}>0,1 MPa

The experimental indicators of the dependence of the rate constant of the amine degradation process (K, mol/l·s) on the partial pressure of CO_2 (P_{CO_2}, MPa) and temperature are described by the following empirical equations.

For MDEA at P_{CO_2}>4 MPa:

$$K=-0,0846+0,169\cdot \ln P_{CO_2} \quad (2)$$

t = 185℃;

$$K = \exp(-15,1335+0,0749\cdot t), \quad (3)$$

100 ℃ < t < 165 ℃;

$$K = -0,8505+5,5\cdot 10^{-3}-3t, \quad (4)$$

165 ℃< t < 205 ℃.

MDEA uchun (P_{CO_2}>2 MPa):

$$K = \exp(-12.05+0,03856\cdot t) \quad (5)$$

Using equations (2–5), it is possible to determine the approximate consumption of amines in gas desulfurization and raw material recovery units, taking into account the technological processes involved.

It should be noted that under industrial conditions, the zones of intensive amine degradation are usually found in areas with high CO_2 saturation and elevated temperatures. Such zones are

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located along the line of the saturated solution, where the temperature ranges from 80 to 110°C.

In the production cycle, liquid ethanolamine (EA) solutions used for gas purification undergo intense thermal effects during absorption, and even stronger ones during desorption, where the temperature can reach 120–130°C. Under these conditions, even chemically stable ethanolamine undergoes additional changes, especially oxidation under the influence of oxygen.

Oxygen is present in trace amounts both in the water used to prepare the EA solution and in the natural gas being purified [2-7]. Industrial DEA and MDEA solutions have been found to contain up to 1.3% by mass of such oxidation products. Along with EA, salts of oxalic, acetic, and formic acids are also present.

Conclusion and Recommendations

Such thermally stable salts are not regenerated, which leads to a decrease in ethanolamine concentration and causes corrosion. Ethanolamine (EA) reacts reversibly with hydrogen sulfide (H_2S). However, in the presence of oxygen, during the desorption of acid gases from EA solutions, ethanolamine does not decompose but instead forms additional thiosulfate compounds - $(EA)_2 \cdot S_2O_3$. Thiosulfates, which form micro-impurities, are oxidized to produce EA and other thiosulfonic acid derivatives [8]. As a result, the formation of thiols, mono- and dialkyl sulfides, and thiophenes has been observed.

COS and CO_2 react irreversibly with primary ethanolamine to form (hydroxyethyl-N-thiocarbamic) and (hydroxyethyl-N-dithiocarbamic) acids [9], as well as their corresponding salts.

Under partial pressures of 0.3–1.17 MPa and heating up to 120–180 °C, the degradation of DEA solutions was observed through the formation of ethyleneamine, imidazolidone, and piperazine compounds [10].

When the absorbing solution contained an aqueous solution of 4.28 mol/L MDEA and was heated to 120–180°C, the following degradation products were identified: 2-(dimethylamino)ethanol, 1,4-dimethylpiperazine, N-(hydroxyethyl)-methylpiperazine, N,N'-bis(hydroxyethyl)piperazine, TEA (triethanolamine), trimethylamine, ethylene glycol, and methanol.

The degradation rate constant of MDEA was found to be approximately 10 times lower than that of DEA. Studies of changes in MDEA+DEA and MDEA+MEA mixtures under the influence of CO_2 (partial pressure 2.6 MPa, temperature 120–180°C) [9] showed that the rate of by-product formation follows the kinetic sequence:

MDEA < MEA < DEA (Equations 6–8).

$$\ln k_{MDEA} = 20{,}34 - 96{,}420/RT \qquad (6)$$

$$\ln k_{MEA} = 19{,}36 - 85{,}476/RT \qquad (7)$$

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$$\ln k_{DEA} = 12{,}60 - 58{,}212/RT \qquad (8)$$

The activation energy **(Ea)** during the interaction with **CO_2** decreases.

Table 2 shows the main degradation products identified for the studied amine mixtures.

Table 1 – Degradation Products of Amine Solutions

Alkanolamine	Components of Acid Gases	Reaction Conditions	Reaction Products	Properties of Degradation Products
Primary:				
-MEA	H_2S	Presence of Oxygen	tiosulfat $(EA)_2 \cdot S_2O_3$	EA does not decompose during the desorption of acid gases
	COS, CS_2	Reaction is partially reversible	Salts of N-(hydroxyethyl)-thiocarbamic acid	
	O_2		α-Aminoaldehyde → Glycine → Glycolic Acid → Oxalic Acid → Formic Acid	Forms iron salts that cause corrosion
	CO_2	Aqueous solution t=165-200 °C	Oxazolidone, N-(hydroxyethyl)-imidazolidone, and ethylenediamine base	
	Oxazolidone-2		N,N'-Diethanolamine → 1-(2-hydroxyethyl)-imidazolidone-2 → Ethylenediamine base	The equipment was operated at ambient temperature and was not exposed to CO_2.
-DGA	CO_2	At a temperature of approximately 210 °C, the reaction is 90% reversible.	N,N'-bis-(hydroxyethoxyethyl) liquid	

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Secondary:				
-DEA	CO_2	τ=8 ch, t=125 °C, C_{DEA}=20%	N,N'-bis-(gidrokietil)-piperazin, N,N',N''-tris-(gidroksietil)-etilendiamin, N-(gidroksietil)-imidazolidon, N-MDEA, N,N'-bis-(gidroksietil)-glitsin	
		t=175 °C, C_{DEA}→2%		
-DIPA			3-(2-oksipropil)-5-metiloksazolidon	
Tertiary:				
-MDEA		τ=144 ch., t=180 °C, C_{MDEA}=4,28 mol/l	2-(Dimethylamino)ethanol, ethylene glycol, 1,4-dimethylpiperazine, methanol, N-(hydroxyethyl)-methylpiperazine, TEA (triethanolamine), trimethylamine, ethylene oxide	

The main method of reducing amine losses caused by thermochemical decomposition is sorption purification (using activated carbon, zeolites, etc.) or filtration, which removes degradation products from the system.

Adding small amounts of aqueous soda solution or organic solvents to the amines accelerates the primary hydrolysis of the oxazolidone-2 degradation products, which in turn reduces the concentration of decomposed compounds in the solution.

It is also important to maintain the regeneration temperature of the saturated amine – it should not exceed 130 °C.

Mechanical losses occur during mixture circulation, within the storage system, and through leakages in non-tight equipment. These losses depend on operational discipline and should not exceed 10% of the total amine losses.

Other amine losses occur due to chemical interactions with gas components (COS, CS_2, RSH, etc.) and oxygen in the air.

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During regeneration, DEA reacts with COS, and the products desorb easily along with amine release. The concentration of CS_2 in natural gas is usually insignificant (up to 20 mg/m^3), and when DEA is heated, more than 80% of the CS_2 reaction products are regenerated.

MDEA does not react with COS or CS_2. Similarly, mercaptans, disulfides, and thiophenes do not react with amines, and therefore no absorption products are formed.

Ethanolamines are not prone to oxidative degradation. In the presence of oxygen, hydrogen sulfide reacts with amines to form thiosulfates, dithiocarbamic acid salts, thiourea, and other compounds, which cannot be decomposed by heating.

To prevent oxidative degradation and amine losses, it is necessary to avoid contact with air, for example by maintaining an inert gas “blanket” in amine storage tanks.

Losses of amines through reactions with gas mixtures cannot be precisely calculated, since many different substances may participate in these processes. Such losses are typically estimated to be around 5–10 mg/m^3 of feed gas.

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