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PURIFICATION BY FILTRATION OF SATURATED ETHANOLAMINES FROM HEAT-RESISTANT SALTS AND DEGRADATION PRODUCTS

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Abstract
The article presents the results of studies on purification of aqueous solutions of spent saturated ethanolamines from heat-resistant salts of destruction products by the filtration method. The work studied the process of purification of ethanolamines by three-stage filtration (sand, coal - bentonite and anionite (Tulsion (USA) A-23 and AB-17-8 RF strongly basic anionites of the gel type)) was recognized as the most effective method, and their properties were analyzed.

Keywords: ethanolamine, natural gas, hydrogen sulfide, carbon dioxide, absorbent, diethanolamine (DEA) and methyldiethanolamine (MDEA), destruction, anions, sand, activated carbon, mechanical compounds, heat resistant salts.

Introduction

Today, a 30% aqueous solution of MDEA is used in the gas purification process of Shortan GKM JSC, DEA and "Mubarak GQIZ" LLC. During the purification of natural gas from toxic compounds in these solutions, the absorption process takes place at high pressure and the desorption process takes place in these solutions at high temperature, and as a result of this process continuing continuously, cases were observed that the absorption solution became saturated and became useless in the gas purification process. It has been determined from the analysis that the temperature and pressure continuously change during the absorption gas cleaning process, the formation of various compounds (oxygen, metal oxides and hydroxides) in the solution causes the destruction and polymerization of ethanolamines, and the accumulation of resinous substances in the solution. In addition to resinous substances, the absorbent solution contains nitrogen, sulfur compounds, formic acid, glycolates, acetates, bicins, oxalate, nitrate, sulfate, chloride salts, iron sulfide from hard rocks, iron oxides and hydroxides, and other impurities [1].

Analyzing the samples of ethanolamine working solutions used in the absorption process and the main parameters of the technological mode, the ways of destabilizing the working mode of

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absorption devices in the purification of natural gas from H_2S, SO_2, COS, CS_2 and other harmful compounds, the reasons for the foaming of the ethanolamine solution under the influence of chemical compounds and its exit from the system were determined, and in the future, they were determined ways to improve the technological process of absorption-desorption purification of natural gas were chosen in order to prevent or reduce it.

Based on the results of the conducted studies, it was determined that the most effective method of cleaning the used ethanolamines from harmful compounds is the combined method based on the ionization method.

It is required to clean the used-saturated ethanolamine solution from mechanical impurities, TBT, salts, DDM and other organic compounds. For this, first of all, 100 cm^3 of quartz river sand with a particle size of 0.34-1.0 mm prepared in advance is passed at a temperature of 65-75 ^oC and a speed of 1.5-2.1 l/h.

Results and Discussion

Purification of absorbent solutions in the combined ion exchange method, the solution was purified in the laboratory device in the following order (Fig. 1).

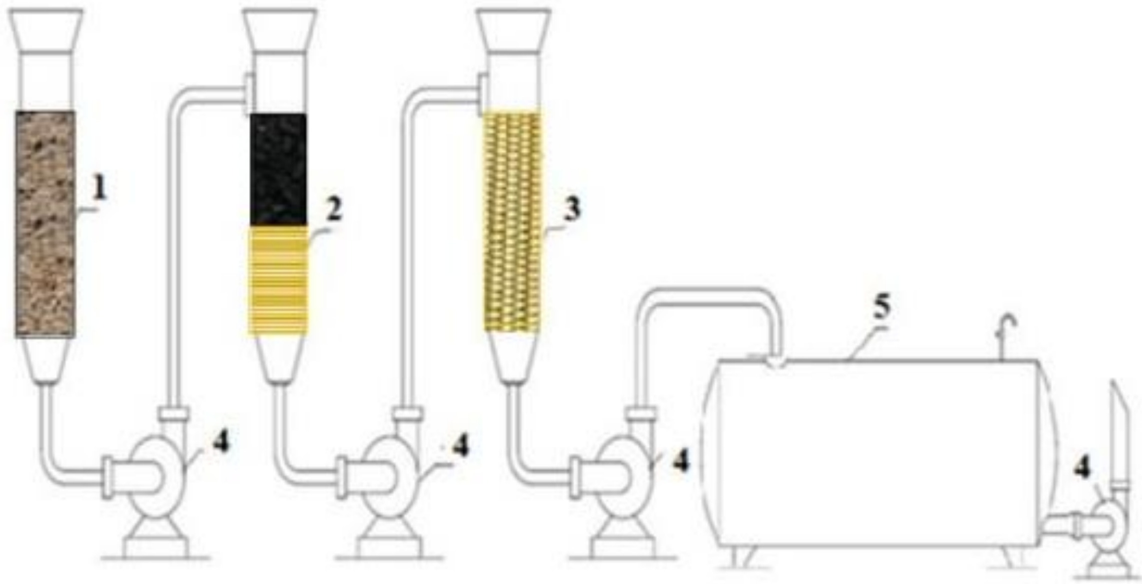

Fig. 1. Purification of the used DEA solution by filtration

1- Sand filter, 2- AG-3 type activated charcoal and bentonite filter, 3- A-23 or AV-17-8 anionite filter, 4- Pumps, 5- Container for storing purified technical DEA solution.

In the first filtration, pre-treated and prepared quartz with a particle size of 0.34-1.0 mm is passed through 100 cm^3 of river sand at a temperature of 65-75 ^oC and a speed of 1.5-2.1 l/h. After purification with river sand, ethanolamine mechanical impurities in the solution decreased to 0.007% in DEA and 0.005% in MDEA. The amount of harmful compounds in the ethanolamine solution is almost unchanged, the amount of SO_2, Cl- and SO_4^{2-}, the total concentration of tarry substances is up to 1.83 g/l in DEA and 1.91 in MDEA, the concentration of NO3 is 233 mg/l in DEA and MDEA It was found that the total sulfur concentration was partially reduced to 689.41 mg/l in DEA content and 691.39 mg/l in MDEA content. Purification of mechanical impurities in solution up to 59-63% was achieved.

In column 1, the absorbent solution purified from mechanical impurities was passed through column (2) where activated carbon of type AG-3 and Navbahor bentonite, separated from each other by filter paper, were placed in a previously prepared diethanolamine solution to remove tarry substances and organic compounds in the solution [2-4].

In the second stage of filtration, AG-3 type activated carbon and specially treated Navbahor bentonite were used. The ethanolamine solution was passed through a 100 cm^3 layer of coal and bentonite at a rate of 1.5-2.1 l/h at a temperature of 60-70 ^oC. During such filtration, it was found that formic acid, bound ethanolamine compounds, tarry compounds, sulfur compounds and other organic compounds were absorbed and they were present in the solution [5].

If the carbon layer placed in the column (2) cleans the solution from organic compounds, heavy oxygen substances such as bitcins, bound ethanolamine compounds and tarry substances, the bentonite layer in the filter will partially absorb the salts along with the tarry substances contained in the solution. From the obtained analysis results, we witnessed that when DEA working solution is filtered in bentonite, bentonite absorbs polarized molecules contained in absorbent working solution. This means that cleaning TBTs dissolved in solution using a bentonite layer is more effective than other methods [6,7].

A sample of technical DEA solution purified with a 30% solution of used-saturated DEA was analyzed by chromatography-mass spectroscopy.

The results of the chromatography-mass spectroscopic analysis in Figure 2 show that in the saturated absorbent solution, the products of thermal destruction of amines, organic compounds, TBT, inorganic salts and other amines polymerized under the influence of temperature and pressure were more than the norm. This indicates deterioration of the physico-chemical and working properties of the absorbent solution.

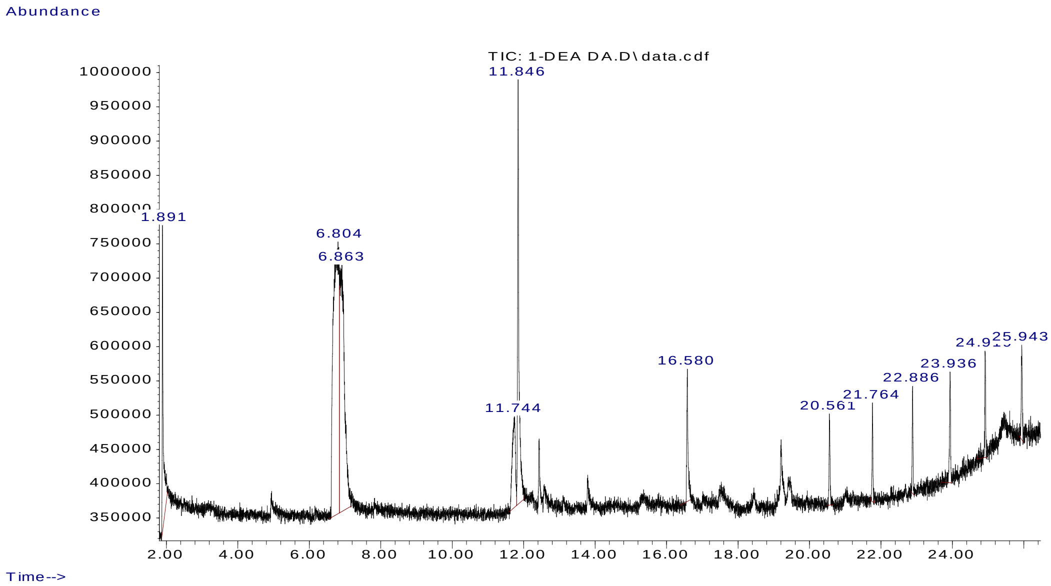

Fig. 2. Spent-Saturated DEA Solution Chromatomass Spectroscopy Analysis

After the three-step purification process, the second sample obtained from technical DEA was also analyzed by chromatography-mass spectroscopy.

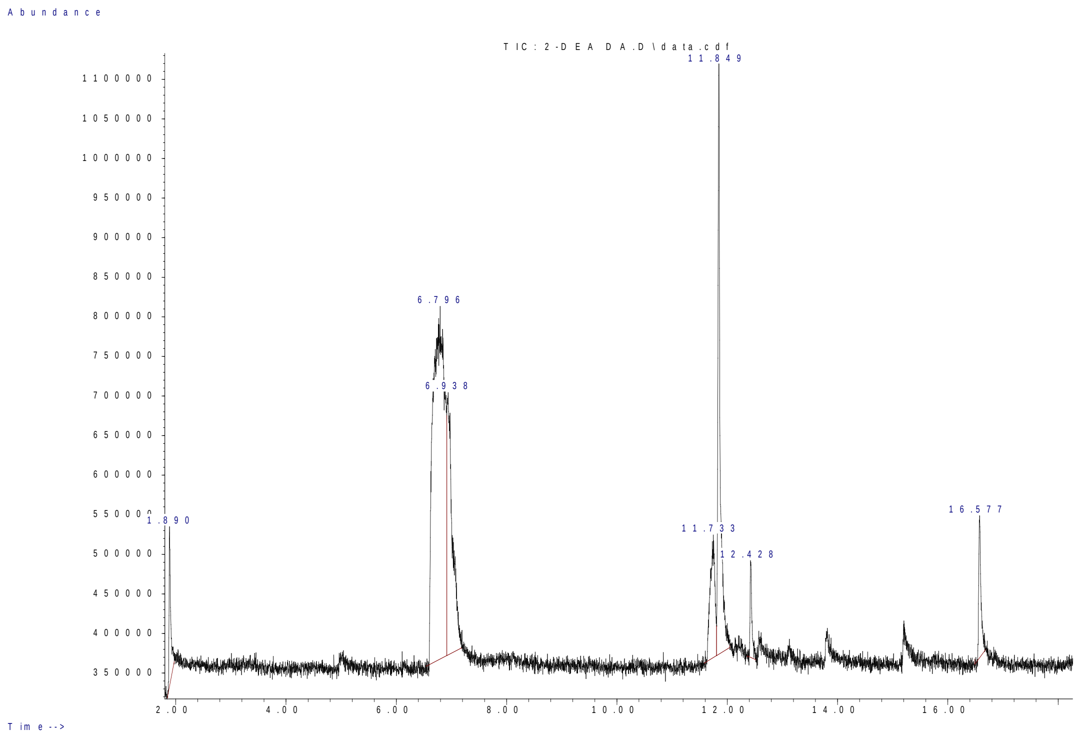

Fig. 3. Chromatomass-spectroscopy analysis of working DEA solution purified by combined method.

The pure DEA obtained for the sample, used in the gas purification process and purified by the combined method was investigated by infrared (IR) and Raman-spectroscopy and gas chromatographic methods.

It is known that the IR-spectroscopy method can provide complete information about functional groups in organic compounds. IR analysis method was carried out on Fure-IR-spectrometer device.

Theoretically, nonlinear compounds have 3N–6 vibrations, and most of these vibrations are observed by IR-spectroscopy, and some by Raman-spectroscopy. Therefore, the study of the studied compounds using IR and Raman spectroscopy methods gives a positive result. In IR-spectroscopy methods, functional groups with a bond dipole moment different from zero give intense absorption, while in Raman spectroscopy, on the contrary, functional groups with a bond dipole moment close to zero give intense absorption. Raman spectra of the DEA sample were analyzed on the Renishaw InViaRaman spectrometer (laser wavelength 785 nm) at the Institute of Ion-Plasma and Laser Technologies of the Academy of Sciences of the Republic of Uzbekistan, and IR on the Carry-IQ-Fure spectrometer of the same institute.

For DEA, there are ($3 \cdot 21^{-6}$) 50 vibrations, most of which are observed in the IR spectrum. However, due to the fact that the molecule is symmetrical (the presence of similar groups), the vibrations corresponding to the groups of substances in the studied solution give absorption peaks at one frequency. Symmetric valence vibrations of the methylene group in the molecule of the substance combine with peaks caused by asymmetric vibrations.

We see that the main absorption lines in the Raman spectrum of DEA are observed at 2950, 2878, 2799, 1457, 1296, 1129, 1045, 943, 872, 752, 532, 468 and 343 cm^{-1} (Fig. 4).

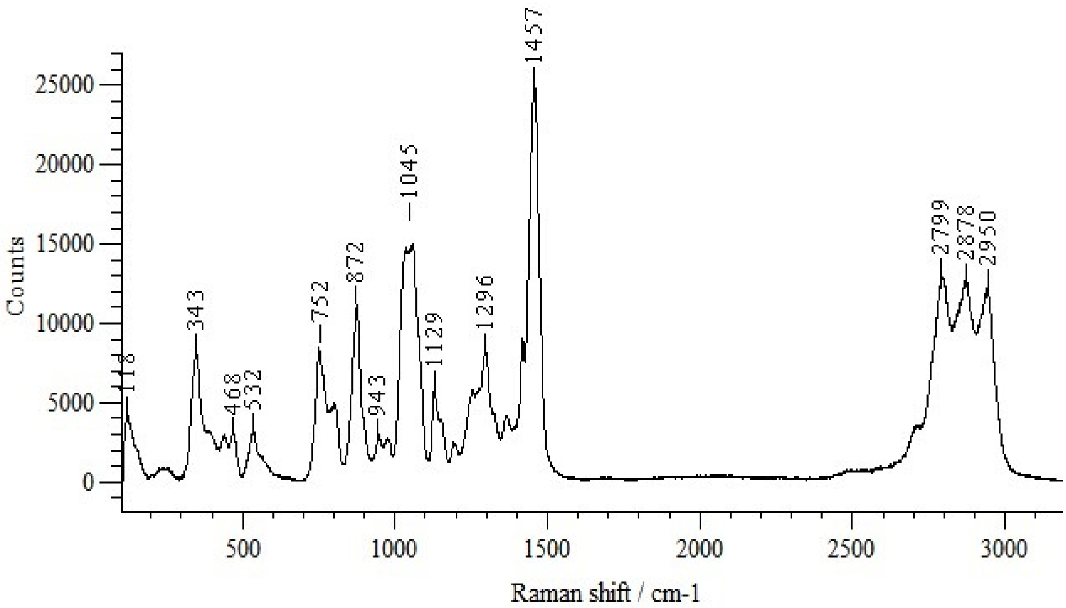

Fig. 4. Raman spectrum of neat DEA

The main peaks in the DEA IR spectrum are the peaks caused by valence and deformation vibrations of methylene groups and OH, C–N and C–O vibrations. For example, the intense peak in the high frequency region (3359 cm^{-1}) was caused by valence vibrations of OH groups. C–O and C–N absorption lines were found to occur at 1034 and 1201 cm^{-1}, respectively.

Conclusion

Thus, it was found that the most effective method of removing toxic compounds from the saturated ethanolamine solution used in the gas purification process and using them again in the gas purification process is the three-stage filtration method. The used ethanolamine solution was passed through sand, bentonite, activated carbon, and anionite filters in order to purify it. Temperature and environment suitable for each filtration process were studied and analyzed, and optimal conditions were selected. Using this method, 60-70% of the saturated ethanolamine solution was purified, technical ethanolamine was obtained, and it was possible to re-use natural gas for purification of toxic compounds.

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