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PURIFICATION OF SATURATED DIETHANOLAMINE USING ACTIVATED CARBON

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Annotation: The article presents the results of a study to determine the physicochemical adsorption properties and some technological parameters for the production of activated carbons based on bones. In the course of the study, the physicochemical and adsorption characteristics of the new activated carbon obtained by us from walnut seeds were determined. For the purpose of import substitution, its characteristics were compared with the well-known industrial activated carbon of the AG-3 brand.

Key words: AG-3, activated carbon, pyrolysis, condensate, methanol.

Introduction: In gas treatment industries, alkanolamines such as methyldiethanolamine (MDEA) and diethanolamine (DEA) are widely used for the removal of acidic components like CO₂ and H₂S from natural gas streams. During long-term operation, these amine solutions degrade due to thermal stress, oxidative decomposition, and contamination by hydrocarbons, corrosion inhibitors, surfactants, and suspended solids. Such degradation not only reduces the absorption efficiency but also leads to severe operational issues like foaming, corrosion, and fouling of equipment. Among the key factors influencing foaming are the accumulation of thermostable salts, high-molecular-weight hydrocarbons, and mechanical impurities, all of which compromise the performance and recyclability of amine solutions.

Activated carbon adsorption remains one of the most effective methods for regenerating spent amine solutions. Commercial activated carbons such as AG-3 have demonstrated reliable efficiency in removing organic and inorganic impurities. However, due to economic and logistical challenges, especially in regions with limited access to imported materials, there is a growing interest in developing activated carbon from locally available raw materials.

This study investigates the purification of saturated diethanolamine (DEA) and methyldiethanolamine (MDEA) solutions using activated carbon derived from fruit seed shells (referred to as AU-KO), and compares its performance with industrial-grade AG-3 carbon. Key indicators such as solution concentration, density, pH, foam height, foam life, mechanical impurities, and thermostable salt content were analyzed to assess purification efficiency. The results demonstrate the viability of AU-KO as a local alternative with comparable or superior adsorption capacity, supporting its potential role in cost-effective and sustainable amine purification systems.

Literature review: The purification and regeneration of spent alkanolamine solutions, particularly diethanolamine (DEA) and methyldiethanolamine (MDEA), have been extensively studied due to their critical role in gas sweetening processes. Over time, amine solutions accumulate degradation products such as heat-stable salts, high-boiling hydrocarbons, surfactants, and corrosion by-products, which reduce absorption efficiency and lead to operational issues including excessive foaming, corrosion, and amine losses [1–3].

Various methods have been proposed for removing these contaminants, including ion exchange, distillation, chemical neutralization, and adsorption. Among these, adsorption using activated carbon is considered the most effective and economically feasible technique for removing a broad spectrum of organic and inorganic impurities [4,5]. Commercially available activated carbons, such as AG-3 and NH-30, have been widely used for this purpose, offering high surface area, microporosity, and thermal stability suitable for amine system regeneration [6].

However, the increasing cost and limited availability of imported activated carbons have led to the exploration of alternative sources, particularly agricultural waste materials for activated carbon production. Studies have shown that fruit shells, nut husks, coconut shells, and other lignocellulosic biomass can serve as efficient precursors for producing activated carbon with high adsorption capacity [7–9]. Thermal and steam activation processes have been optimized to tailor the pore structure and surface chemistry of these carbons to target specific contaminants in amine systems [10].

Recent research highlights the importance of matching pore size distribution and surface functionality of the carbon to the nature of the contaminants—hydrophobic hydrocarbons, hydrophilic salts, or solid particulates [11]. In this context, activated carbon derived from fruit seed shells (AU-KO) has shown promising results, with adsorption characteristics comparable to AG-3 carbon, especially in terms of reducing foam height, minimizing thermostable salt content, and improving the working concentration of MDEA solutions [12].

Despite this progress, there remains a knowledge gap in systematic comparisons of locally produced activated carbons with industrial-grade carbons under identical conditions in gas purification systems. This study aims to address this gap by evaluating the adsorption efficiency of AU-KO carbon and its applicability as an import-substituting material in amine solution regeneration.

Methodology: To obtain activated carbons, the carbonization process was carried out at a temperature of 400-800°C, which was controlled by a thermocouple located in the middle of the reactor. The temperature rise rate was 7-10°C per minute.

Upon reaching the required temperature of the experiment, the sample was kept in the reactor for 1-2 h and then cooled to room temperature. The evolved gaseous pyrolysis products were evacuated from the reactor through a gas outlet tube and sent to a cooled condenser for condensation of water and resin vapors.

Cooled carbonizates were discharged from the reactor and their absorption properties were determined using standard methods. The activation of the carbonizate was carried out in the same reactor. For activation, carbonized granules are loaded into a tubular reactor, which was purged with a stream of nitrogen gas for 15 minutes to remove oxygen from the reaction zone. The upper flange of the reactor is equipped with a branch pipe for the inlet of superheated water vapor, and the lower one has a branch

pipe for withdrawing the steam-gas mixture. The superheated steam required for activation is obtained in the steam generator. The flow rate of water vapor for activation is controlled by the amount of water entering the steam generator by changing the rate of its flow in the capillary depending on the pressure above the water created in the dosing tank using nitrogen. The activation temperature is controlled by heating the reactor and the steam generator. The sample was heated to the final activation temperature, which was in the range 800-950 ° C. When the set temperature was reached, water vapor was fed into the reactor from the generator for 1-2 hours. The volume of activation gases containing hydrogen, carbon oxides, and methane after separation from unreacted water vapor is measured by a gas meter, and the chemical composition is measured by chromatography using a carbon column and thermal conductivity detector.

After heat treatment, the resulting activated carbon was left to cool to room temperature without air access.

The quality of the obtained target products was controlled by measuring their bulk density, ash content, and adsorption activity with respect to benzene.

Results: The technical characteristics of the obtained activated carbon from the nut seed shell (hereinafter AU-KO) have been determined. Some characteristics of AU-KO activated carbon are compared with the characteristics of active industrial activated carbons of the AG-3 and NH-30 brands are shown in Table 1.

Table 1

Comparative technical characteristics of activated carbon coal AG-3, NH-30 and AU-KO

No	Indicators	Ah-3 (Russia)	NH-30 (China)	Presented Sample (AU-KO)
1	Fractional composition, %			
	5 mm	0,4	2	8
	3.6 mm	3	10	16
	from 2.8 to 3.6 mm	86	70	64
	from 1.5 to 2.8 mm	10	15	5
	from 1.0 to 1.5 mm	0,6	3	7
2	Bulk density, g/dm ³	480	520	455
3	Iodine activity,%	88	98	86,4
4	Activity for benzene, % or g / dm ³	31	34	33
		148	176	150,2
5	Total pore volume, cm ³ /g	0,860	0,692	0,720
6	Moisture content,%	6	5	6
7	Solness,%	14	4,2	7
8.	Abrasion strength,% min.	75	78	76

Comparison of the results of experimental research data with the literature showed that the obtained activated carbon AU-KO in terms of adsorption activity and other physicochemical parameters is at the level of known activated carbons AG-3 and NH-30, which are one of the highest quality coals of world industrial production. Thus, the results of the conducted studies demonstrate the expediency of processing fruit seeds in the Republic to obtain carbon adsorbents for various purposes.

The composition of the purified alkanolamine solution must meet the following requirements for reuse in the amine purification of natural gas: amine degradation products - no more than 10% by weight,

liquid high-molecular hydrocarbons - no more than 0.05% vol., Heat-resistant salts - 1% by weight. mechanical impurities - 500 mg/l. All these parameters affect the foaming of the amine solution.

To carry out experiments to determine the effect of organic substances on the foaming of alkanolamines, the following reagents were selected: as a solution of alkanolamine, a 25% (wt.) DEA solution was used, and as organic substances, pentane, octane, nonane, undecane, cyclopentane, cyclohexane, fractions 120 ° C and 150 ° C condensate, methanol, 10% residue from the distillation of technical methanol, as well as a pure solution of DEA. At the entrance to the experiment, the height (H) of the foam of a 25% DEA solution was determined with the addition of the above organic substances at various concentrations. The results are shown in Table 2.

Table 2.

Results of determination Purpose of foam 25% DEA solution with various additives

№	Sample names	Mass fraction of additives, %	Height of foam H, mm
1.	DEA + Pentan	0,1	0
		0,5	0
		1,0	3
2.	DEA + Octan	0,1	1
		0,5	4
		1,0	11
3.	DEA + Nonan	0,1	8
		0,5	22
		1,0	24
4.	DEA + Undekan	0,1	21
		0,5	32
		1,0	37
5.	DEA + cyclopentan	0,1	1
		0,5	3
		1,0	0
6.	DEA + Cyclohexane	0,1	-
		0,5	0
		1,0	5
7.	DEA + FR. 120 ° C condensate	0,1	4
		0,5	8
		1,0	11
8.	DEA + FR. 150 ° C condensate	0,1	8
		0,5	12
		1,0	17
9.	DEA + Methanol	0,1	0
		0,5	0
		1,0	0
10.	DEA + 10% residue from producing technical methanol	0,1	0
		0,5	1
		1,0	3
11.	DEA + Clean DEA solution	0,1	0
		0,5	0

	1,0	0
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Based on the above research results in Table 1, a graph is plotted of the dependence of the foam height (H) of a 25% DEA solution on the mass fraction of various additives (Fig. 1).

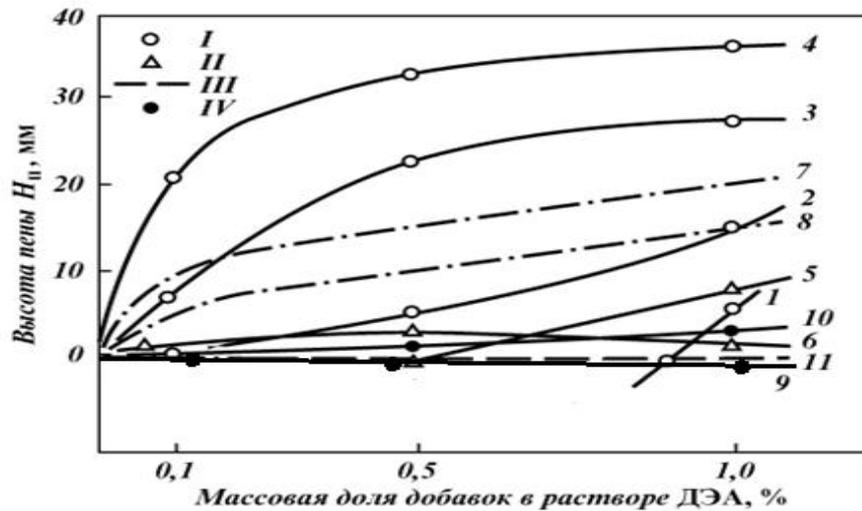


Figure: 1. Influence of various additives on the foaming ability of a 25% DEA solution:

I - paraffins; II - naphthenes; III - condensate fractions; IV - alcohols; 1 - C_5H_{12} ; 2 - C_8H_{18} ; 3 - C_9H_{20} ; 4 - $C_{11}H_{24}$; 5 - C_6H_{12} ; 6 - C_5H_{10} ; 7 - condensate, fraction $150^{\circ}C$; 8 - condensate, fraction $120^{\circ}C$; 9 - methanol; 10 - the remainder (10%) from the distillation of technical methanol; 11 - pure DEA solution.

The results of the study showed that the expansion rate of the foam depends on the nature and properties of impurities in the gas coming from the field and can cause foaming of amine solutions. The greatest foaming is caused by hydrocarbons having a boiling point above $100^{\circ}C$ (condensate, oil), surfactants, and some corrosion inhibitors. From the results of the experiment, it was revealed that hydrophobic substances such as paraffinic, naphthenic, aromatic hydrocarbons and condensate fractions increase the height and life of the foam, due to the formation of a strong protective film at the interface, while hydrophilic alcohols and amino alcohols reduce them or do not form foaming of the solution.

To determine the effect of inorganic substances on the foaming of alkanolamines, the following salts were chosen: $MgCO_3$, $CaCO_3$, KCl , K_2SO_4 , and MEA solution. The results of experimental studies to determine the effect of salts $MgCO_3$, $CaCO_3$, KCl , K_2SO_4 on the foaming ability of the MEA solution are shown in Figure 2.

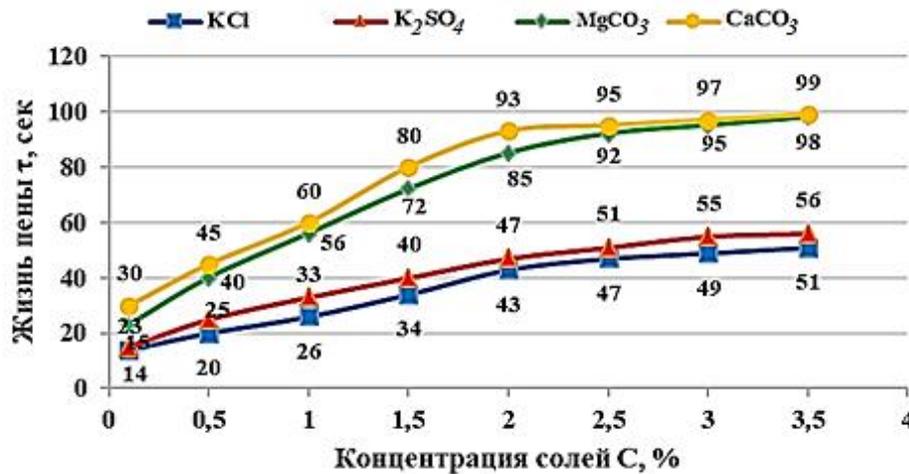


Fig. 2. The dependence of the life expectancy of the foam τ (c) on the concentration of salts with (%) in the MEA solution

To determine the amount of mechanical impurities in the MDEA solution, three samples were taken: the initial one, purified with AG-3 activated carbon and purified with AU-KO activated carbon. The results obtained from the determination of mechanical impurities in the MDEA solution are shown in Table 3.

Table 3

Results from determining mechanical impurities in solution MDEA

№	MDEA solution samples	The amount of fur. impurities, mg / l
1	The original	1068
2	Purified (AG-3)	488
3	Purified (AU-KO)	483

So, the best results of the analyzes carried out to determine the quality of the purified MDEA solution and other physicochemical indicators (density and pH of the solution) are shown in Table 4.

Table 4.

Physico-chemical characteristics of a purified MDEA solution

№	Parameters	MDEA solution samples		
		Source (regenerated)	Purified (activated carbon AG-3)	Purified (activated carbon AU-KO)
1	Concentration of amine,% of mass.	40	39	55
2	pH	10,80	10,40	10,40
3	Density, g/sm ³	1,092	1,085	1,122
4	The content of thermostable salts,% of the mass.	2,80	0,83	0,81

5	Content of mechanical impurities, mg/l	1068	488	483
6	Foaming: foam height, mm life foam, sec.	16 20	16 9	15 9

Discussion: From the above data, it follows that the working concentration of the MDEA solution increased after cleaning with AU-KO activated carbon when compared with the initial solution from 40 to 55%, and the indicator of the AG-3 purified coal solution slightly decreased, which means that the working concentration of the MDEA solution after cleaning with coals AG-3 and AU-KO is within the limits recommended by the design organization for operation, and the density corresponds to the concentration (1.085-1.122 g/cm³).

The content of thermostable salts in the initial regenerated solution of MDEA is 2.80 wt%, which exceeds the maximum permissible concentration by 1 wt%, and in the purified solution with AG-3 and AU-KO coals their concentration is 0.83 and 0.81 respectively, which does not exceed the maximum permissible concentration.

The amount of mechanical impurities in the original regenerated amine is high - 1068 mg/L, and in the purified samples with AG-3 and AU-KO coals is low - 488 and 483 mg/L when compared with the permissible concentration of 500 mg/L, since their high content in amine is one of the causes of foaming and can be a foam stabilizer.

The foam height of all samples of amine solutions was at a low level (15-16 mm). The foam destruction time (foam life) of the initial amine solution was at a high point of the average level (20 sec), while in the purified solutions with AG-3 and AU-KO coals it was at a low level (9 sec).

In the course of the studies carried out, it was revealed that thermostable salts and mechanical impurities accumulating during the operation of gas absorption cleaning units are the main causes of foaming of working solutions of alkanolamines. Analyzing the obtained results of experiments, it was found that activated carbon from local raw materials AU-KO is not inferior to the Russian analogue of activated carbon AG-3 in terms of the cleaning ability of spent alkanolamine solutions and other technical parameters. For the purpose of import substitution of the developed activated carbon, AU-KO can be recommended for adsorption purification of spent alkanolamines instead of AG-3 coal.

Conclusion: The conducted study demonstrates the effectiveness of activated carbon in purifying saturated MDEA solutions contaminated with various organic and inorganic impurities. Comparative evaluation between commercial AG-3 activated carbon and locally produced AU-KO carbon derived from fruit seed shells revealed that both adsorbents significantly improved the physicochemical parameters of the amine solutions.

Specifically, purification with AU-KO increased the working concentration of the MDEA solution from 40% to 55%, while reducing the content of thermostable salts from 2.80 wt% to 0.81 wt%, and mechanical impurities from 1068 mg/L to 483 mg/L. These values fall within the permissible operational ranges, indicating that AU-KO effectively restores the functionality of degraded amine solutions.

Foaming analysis showed that both AG-3 and AU-KO reduced foam height and foam life, with foam destruction time decreasing to 9 seconds—thereby minimizing the risks associated with foaming during gas treatment processes. Importantly, AU-KO demonstrated comparable adsorption efficiency to the commercial AG-3, making it a viable, low-cost, and sustainable alternative for the regeneration of spent alkanolamine solutions.

Overall, the use of AU-KO activated carbon represents a promising import-substitution strategy, supporting the development of local adsorbents for industrial gas purification systems. Its implementation can contribute to both economic and environmental benefits by valorizing agricultural waste and reducing dependency on imported materials.

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