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#### ALKALIC CLEAVAGE OF 1,2-AMINOESTERS AND RELATED COMPOUNDS

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Abstract: The work investigated the reactions of alkaline cleavage of quaternary salts of 1,2-aminoesters and related compounds containing various hydrocarbon groups at the quaternary nitrogen atom aiming to synthesize organic compounds containing O, N heteroatoms and a double C=C bond. As a result of research, it was established that alkylation of the OH group in quaternary compounds 1, 2 - amino alcohols and their related compounds occurs under mild conditions in a system of 50% aqueous alkali and benzene at  $0-20^{0}C$ . It has been shown that under extremely mild conditions, reactions of quaternary derivatives of 1, 2-amino alcohols with alkyl halides under the same conditions lead to almost quantitative formation of the corresponding quaternary amino esters.

It has been established that these amino ester salts are easily decomposed at  $100-120^{0}$ C. In this case, the formation of new and destruction of old bonds is determined by the natural groups located at the quaternary nitrogen atom. It has been established that quaternary salts of 1,2-amino esters containing an ethyl group at the nitrogen atom easily lose the ethyl group in the form of ethylene without affecting the 2-alkoxyethyl group and form the corresponding 1,2-amino esters.

It has been established that the patterns typical for the reaction of quaternary salts of 1,2-amino alcohols under the same conditions are characteristic of salts of 1,3- and 1,4-amino alcohols. Since the alkaline cleavage of the resulting quaternary salts of 1,3 and 1,4-amino esters, which are formed by the interaction of the corresponding quaternary salts of 1,3- and 1,4-amino alcohols with organohalogen compounds, occurs similarly.

It has been established that salts of 1,2-amino esters formed by the interaction of organohalogen compounds with quaternary salts of 1,2-aminoalcohols containing an allylic group at the nitrogen atom upon alkaline cleavage easily eliminate the allylic group in the form of allylic alcohol and form the corresponding 1,2-amino esters.

Interesting results were obtained when studying the alkaline cleavage of the corresponding quaternary salts of 1,2-amino esters formed by the reaction of organohalogen compounds with (2-hydroxyethyl) trimethylammonium hydroxide or chloride. It has been shown that in the presence of methyl groups at the nitrogen atom, the 2-alkoxyethyl group of the quaternary salt of 1,2-amino esters undergoes cleavage, similarly to the salts of cyclic 1,2-aminoesters. As a result, vinyl ethers are formed.

Key words: quaternary salts, 1,2-amino alcohols, 1,2-aminoesters, vinyl ethers, quaternary salts of 1,2-amino alcohols, quaternary salts of 1,2-aminoesters, 2-trialkylammonioethoxides, aqueous solutions of alkalis.

**UDC** 547.435. 1`21`32`33

#### Introduction

We have discovered a new direction of quaternary salts reaction of 1,2-amino- alcohols with concentrated aqueous solutions of alkalis, which consists in the formation of bipolar 2-trialkylammonioethoxides at 0-20 °C. It has been established that the reaction is subject to kinetic control and includes the intermediate formation of the OH form of trialkyl(2-hydroxyethyl) ammonium and subsequent deprotonation of hydroxyl in 2-hydroxyethyl group with OH<sup>-</sup> ions [1]. We have found that alkylation of the OH group in quaternary compounds of 1,2-amino alcohols and

related compounds occurs exclusively under mild conditions at 0-20°C in a system of 50% alkali and benzene [2]. The formation of 2-trialkylammonioethoxides from quaternary derivatives of 1,2-aminoalcohols, as well as their interaction with electrophiles, is to some extent reminiscent of the O-alkylation reaction of the hydroxyl group in alcohols with alkyl halides, occurring under phase-transfer catalysis conditions [2,3,4-10], but differs significantly in the reaction mechanism. It has been established that this generates 2-trialkylammonioethoxides, which easily interact with electrophiles to form the corresponding quaternary salts of 1,2-aminoesters without the use of foreign catalysts [2].

Exceptionally mild conditions, availability and a wide range of starting compounds in the reactions of quaternary derivatives of 1,2-aminoalcohols with organic halogen compounds, leading to the almost quantitative formation of quaternary salts of 1,2-aminoesters, made it expedient to study the chemical transformations of the latter and related compounds for the purpose of the synthesis of organic compounds containing heteroatoms O, N and a double C=C bond. In this regard, it is of interest to study the reaction of alkaline cleavage of quaternary salts of 1,2-aminoesters and related compounds containing various hydrocarbon groups at the quaternary nitrogen atom.

The alkalic cleavage of quaternary salts of 1,2-aminoesters formed in the reaction of quaternary salts of 1,2-aminoalcohols with organic compounds was studied mainly using a method that excludes the isolation of the quaternary salt of 1,2-aminoester from the reaction medium. In this case, the reaction leading to the quaternary salt of 1,2-aminoester was carried out with an excess of 50-55% aqueous potassium or sodium hydroxide in benzene at 10-20°C for 3-4 hours. Then the alkaline cleavage of the resulting salt was carried out by heating.

It turned out that the quaternary salts of 1,2-aminoesters are easily dissociated at 100-120°C. In this case, the nature of the formation of new and destruction of old bonds is determined by the nature of the groups located at the quaternary nitrogen atom.

It has been established that quaternary salts of 1,2-aminoesters containing an ethyl group at the nitrogen atom easily lose the ethyl group in the form of ethylene without affecting the 2-alkoxyethyl group and form the corresponding 1,2-amino-esters (1-7) with a yield of 72-86%

(1) 
$$R^1 = R^2 = R^3 = C_2H_5$$
. (2)  $R^1 = R^2 = C_2H_5$ ,  $R^3 = C_3H_7$ . (3)  $R^1 = R^2 = C_2H_5$ ,  $R^3 = C_4H_9$ . (4)  $R^1 = R^2 = C_2H_5$ ,  $R^3 = C_5H_{11}$ . (5)  $R^1 = R^2 = C_2H_5$ ,  $R^3 = CH_2CH = CH_2$ . (6)  $R^1 = R^2 = C_2H_5$ ,  $R^3 = CH_2C_6H_5$ . (7)  $R^1 = R^2 = C_2H_5$ ,  $R^3 = CH_2C_6H_5$ .

It has been established that the patterns typical for the reaction of quaternary salts of 1,2-aminoalcohols, under the same conditions, are characteristic of quaternary salts of 1,3- and 1,4-aminoalcohols and related compounds. Since the alkaline cleavage of quaternary salts of 1,3- and 1,4-aminoesters occurs similarly, which are formed by the interaction of the corresponding quaternary salts of 1,3- and 1,4-aminoalcohols with benzyl chloride. These reactions are also interesting bath aqueous solution of alkali because in this case we have examples indicating the generality of the properties of quaternary salts of amino alcohols to form bipolar 3-trialkylammonio-propoxides and 4-trialkylammonio-butocoids from the corresponding quaternary salts of 1,3- and 1,4-aminoalcohols in concentrated

$$\bigoplus_{(C_2H_5)_3N(CH_2)_nOH} X \stackrel{\bigodot}{\longrightarrow} \underbrace{(C_6H_5CH_2CI, NaOH)}_{(C_6H_6 \cdot H_2O)} \xrightarrow{(C_2H_5)_3N(CH_2)_nOCH_2C_6H_5} X \stackrel{\bigodot}{\longrightarrow} \underbrace{(C_2H_5)_3N(CH_2)_nOCH_2C_6H_5}_{NaOH} X \stackrel{\bigodot}{\longrightarrow} \underbrace{(C_2H_5)_2N(CH_2)_nOCH_2C_6H_5}_{(9,10)} \times \underbrace{(C_2H_5)_3N(CH_2)_nOCH_2C_6H_5}_{(9,10)} \times \underbrace{(C_2H_5)_3N(CH_2)_nOCH_$$

It has been established that quaternary salts of 1,2-aminoesters formed by the interaction of organohalogen compounds with quaternary salts of 1,2-amnoalcohols containing an allylic group at the nitrogen atom upon alkaline cleavage easily eliminate the allylic group in the form of allylic alcohol and form the corresponding 1,2-aminoesters with high outputs

$$(H_{3}C)_{2}HN \xrightarrow{RX, KOH, 10-20 ° C} \xrightarrow{(H_{3}C)_{2}NN} \xrightarrow{(H_{3}C)_{2}N} \xrightarrow{(H_{3}C)_{2}HN} \xrightarrow{X} \xrightarrow{CH_{2}CH_{2}OH}$$

$$(11) R=CH_{2}CH=CH_{2}, (12) R=CH_{2}CH_{2}CH_{2}OH \xrightarrow{(I1-13)} \xrightarrow{(I4)} \xrightarrow{(I4)} \xrightarrow{(I4)} \xrightarrow{CH_{2}CH=CH_{2}} \xrightarrow{RX, KOH, 10-20 ° C} \xrightarrow{(P_{4}CH=CH_{2})} \xrightarrow{CH_{2}CH=CH_{2}} \xrightarrow{CH_{2}CH_{2}OH} \xrightarrow{(I00-120 ° C)} \xrightarrow{(I00-120 ° C)} \xrightarrow{N-CH_{2}CH_{2}OR} \xrightarrow{N-CH_{2}CH_{2}OH} \xrightarrow{(I5-20)} \xrightarrow{(I4)} \xrightarrow{(I4)}$$

(15) R=C<sub>2</sub>H<sub>5</sub>. (16) R=C<sub>3</sub>H<sub>7</sub>. (17) R=C<sub>4</sub>H<sub>9</sub>. (18) R=C<sub>5</sub>H<sub>11</sub>. (19) R=CH<sub>2</sub>CH=CH<sub>2</sub>. (20) R=CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>.

(21) R=C<sub>2</sub>H<sub>5</sub>. (22) R=C<sub>3</sub>H<sub>7</sub>. (23) R=C<sub>4</sub>H<sub>9</sub>. (24) R=C<sub>5</sub>H<sub>11</sub>. (25) R=CH<sub>2</sub>CH=CH<sub>2</sub>. (26) R=CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub> The ease of elimination of the allyl group from the ammonium cation of the quaternary salt of a 1,2-aminoester can be explained by the fact that with nucleophilic substitution of the ammonium group with an OH- ion, the highest point of the energy barrier or the transition state between reactants and products is more easily achieved due to stabilization, caused by delocalization of electrons of the double bond of the allylic group

Based on the reaction described above, we were able to synthesize a long-chain diamino ester, a linear analog of diazacrown compounds, starting from more accessible starting compounds. It turned out that the quaternary salt of 1,2-amino- alcohol is 1-allyl-1-(2-hydroxyethyl) piperidinium bromide in a system of 50% KOH and benzene, reacting with 1,5-dibromo-3-oxapentane at 20 °C forms almost quantitatively a diquaternary ammonium salt (27), which, upon alkaline cleavage, also easily loses allyl groups and turns into the corresponding diamine (28) with three ether groups.

$$2 \bigoplus_{\text{CH}_2\text{CH}=\text{CH}_2}^{\text{CH}_2\text{CH}=\text{CH}_2} \xrightarrow{\text{BrCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{Br}, \text{ KOH, } 10\text{-}20 \text{ °C}} \xrightarrow{\text{C}} \xrightarrow{\text{CH}_2\text{CH}_2\text{OH}} \xrightarrow{\text{C}}$$

No less interesting results were obtained when studying the alkaline cleavage of the corresponding quaternary salts of 1,2-aminoesters formed by the reaction of organohalogen compounds with (2-hydroxy-ethyl) trimethylammonium hydroxide or chloride. It has been established that in the presence of methyl groups at the nitrogen atom, the 2-alkoxyethyl group of the quaternary salt of the 1,2-amino- ester undergoes cleavage, similarly to the salts of cyclic 1,2-aminoesters. As a result, vinyl ethers are formed, some analogues of which are of great importance in the chemical industry.

$$\bigoplus_{(\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OH}} \ominus \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OH}} \ominus \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OH}} \ominus \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{CH}_2\operatorname{CH}_2\operatorname{OR}} \times \bigcirc \\ (\operatorname{CH}_3)_3\operatorname{NCH}_2\operatorname{$$

(29)  $R=CH_3$ , (30)  $R=C_2H_5$ , (31)  $R=C_3H_7$ , (32)  $R=i-C_3H_7$ , (33)  $R=C_4H_9$ , (34)  $R=i-C_4H_9$ , (35)  $R=C_5H_{11}$ , (36)  $R=i-C_5H_{11}$ , (37)  $R=CH_2C_6H_5$ .

The structure of the synthesized compounds (1-38) was established by physicochemical methods of analysis, including counter syntheses, GLC and H-NMR spectroscopy. By counter synthesis starting from acetylene and the corresponding alcohol using the Favorsky reaction according to the method described in [12], the structures of vinyl ethers (29-35) were established.

H-NMR spectral data for some 1,2-aminoesters are given in Table 1. In the H-NMR spectra of 1,2-aminoesters, the signals of the protons of the methylene groups in the N-CH<sub>2</sub>CH<sub>2</sub>O fragment appear in the corresponding regions at 2.28-2.56 ppm. (N-CH<sub>2</sub>) and 3.31-3.36 ppm. (CH<sub>2</sub>O) respectively. However, instead of the expected corresponding clear triplets, multiplets are observed, which are caused by overlaps in the signals of the protons of the methylene groups, directly associated with the corresponding heteroatoms, but located in the alkyl radicals R<sup>1</sup>, R<sup>2</sup> and R<sup>3</sup>. The signal of the methylene protons of the allyl group directly bonded to the oxygen atom appears at 3.76-3.86 ppm. In weaker fields at 4.6 and 5.175 ppm. signals of the protons of the benzyloxy and benzhydryloxy groups appear. The signal of the proton of the methylene group of the benzyl radical bound to the nitrogen atom appears in a higher field at 3.48 ppm than the signal of the CH<sub>2</sub> group in the benzyloxy radical (4.6 ppm). Signals of protons located in alkyl radicals and one carbon atom removed from heteroatoms appear in the corresponding regions of the spectrum. In this case, the signals of the protons of the terminal methyl groups and the C-CH<sub>2</sub>-C group appear at 0.89-1.01 and 1.24-1.52 ppm. in the form of a triplet and multiplet, respectively. Sigma protons of aromatic rings and olefinic protons of the allyl group appear in appropriate places (see Table 1).

It is noted that the identification of 1,2-amino esters by H-NMR spectra is facilitated in the presence of trifluoroacetic acid. For example, in the H-NMR spectrum of 1-(2-ethoxyethyl) piperidine, the signals from the protons of two methylene groups directly bonded to the oxygen atom and the signals from the six protons of three methylene groups directly bonded to the nitrogen atom appear as multiplets at 3.39 and 2 .34 ppm respectively (Fig. 15a). However, in the presence of trifluoroacetic acid (Fig. 15b), as a result of the formation of an oxonium and ammonium center in the molecule, the signal of the protons of the  $\alpha$ -methylene group located in the 2-ethoxyethyl group is detected in the form of a clear triplet at 3.0 ppm. At the same time, a clear triplet appears at 2.5 ppm. protons of two methylene groups directly bonded to nitrogen and located in the heterocycle. Multiplet in the region

of 1.38 ppm. corresponds to six protons of three methylene groups located in a heterocycle distant from the nitrogen atom. Triplet at 1.16 ppm corresponds to the signal of the protons of the methyl group located in the ethyl radical.

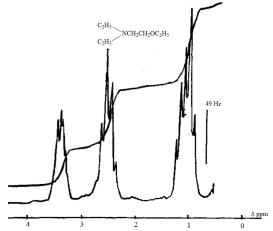


Fig.1. H-NMR spectrum of N,N-diethyl-2-ethoxyethylamine (1)

**Table 1.** H-NMR spectrum data of compounds (1, 2, 4-7, 9-11, 13, 15, 19, 20, 28)

No. of	No of pic.	Groups Structure of compound	CCH3	$CCH_2C$	$NCH_2C$	CCH2O	OCH <sub>2</sub> C=C	$NCH_2A_I$	OCH2Ar	OCH(AL)2	AL.	$H_I$	H <sup>2</sup> C=C H <sup>1</sup>	H <sup>3</sup>
	,	S.C.C. S.C. S.C. S.C. S.C. S.C. S.C. S.				Che	mical sl	nift (Δ),	m.d., (	δ-scale	and sign	al format		
1	1	(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> OC <sub>2</sub> H <sub>5</sub>	0,89	-	2,46	3,34	-	-	-	-	-	-	-	-
2	2	(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> OC <sub>3</sub> H <sub>7</sub>	0,89	1,52	2,56	3,31	-	-	-	-	-	-	-	-
4	3	(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> OC <sub>5</sub> H <sub>11</sub>	0,88	1,31	2,45	3,31	-	-	-	-	-	-	-	-
5	4	(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> OCH <sub>2</sub> CH=CH <sub>2</sub>	0,99	-	2,46	3,36	3,86	-	-	-	-	5,56-6,09	5,06	5,26
6	5	(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> OCH <sub>2</sub> C <sub>6</sub> H <sub>5</sub>	0,90	-	2,45	3,36	-	-	4,60	-	7,15	-	-	-
7	6	$C_6H_5CH_2(C_2H_5)NCH_2CH_2OC_2H_5$	1,01	-	2,51	3,31	-	3,48	-	-	7,13	-	-	-
9	7	(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> OCH <sub>2</sub> C <sub>6</sub> H <sub>5</sub>	0,91	1,41	2,44	3,40	-	-	4,60	-	7,15	-	-	-
10	8	(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> OCH <sub>2</sub>	0,90	1,31	2,45	3,38	-	-	4,61	-	7,14	-	-	-
11	9	C <sub>6</sub> H <sub>5</sub>	-	-	2,28	3,36	3,76	-	-	-	-	5,61-6,00	4,80	5,19
		(CH <sub>3</sub> ) <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> OCH <sub>2</sub> CH=CH <sub>2</sub>			2,1									
					NCH <sub>3</sub>									
13	10		-	-	2,4	3,38	-	-	-	5,18	7,1	-	-	-
		(CH <sub>3</sub> ) <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> OCH(C <sub>6</sub> H <sub>5</sub> ) <sub>2</sub>			2,05									
					NCH <sub>3</sub>									
15	11		1,06	1,38	2,34	3,39	-	-	-	-	-	-	-	-
19	12	(CH <sub>2</sub> ) <sub>5</sub> NCH <sub>2</sub> CH <sub>2</sub> OC <sub>2</sub> H <sub>5</sub>	-	1,24	2,28	3,36	3,84	-	-	-	-	5,51-6,11	5,03	5,24
20	13	(CH <sub>2</sub> ) <sub>5</sub> NCH <sub>2</sub> CH <sub>2</sub> OCH <sub>2</sub> CH=CH <sub>2</sub>	-	1,35	2,26	3,38	-	-	4,31	-	7,1	-	-	-
		(CH <sub>2</sub> ) <sub>5</sub> NCH <sub>2</sub> CH <sub>2</sub> OCH <sub>2</sub> C <sub>6</sub> H <sub>5</sub>			2,37									
28	14			1,39	2,31	3,44	-	-	-	-	-	-	-	-
		(CH <sub>2</sub> ) <sub>5</sub> NCH <sub>2</sub> (CH <sub>2</sub> OCH <sub>2</sub> ) <sub>3</sub> CH <sub>2</sub> N(			2,42									
		CH <sub>2</sub> ) <sub>5</sub>												

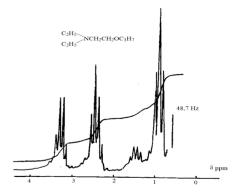


Fig.2. H-NMR spectrum of N,N-diethyl-2-propoxyethilamine (2)

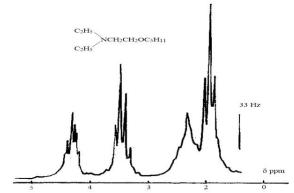


Fig.3. H-NMR spectrum of N,N-diethyl-2-penthyloxyethilamine (4)

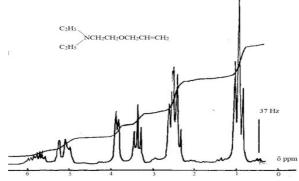


Fig.4. H-NMR spectrum of N,N-diethyl-2-allyloxyethilamine (5)

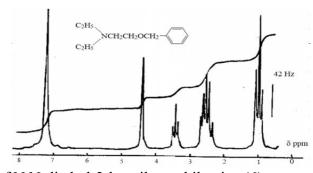


Fig.5. H-NMR spectrum of N,N-diethyl-2-benziloxyethilamine (6)

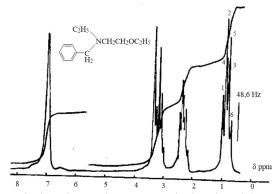


Fig.6. H-NMR spectrum of N-ethyl-N-benzyl-2-etoxyethylamine (7)

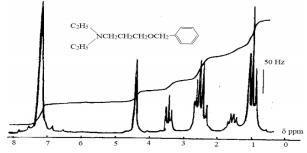


Fig.7. H-NMR spectrum of N,N-diethyl-3-benzyloxypropylamine (9)

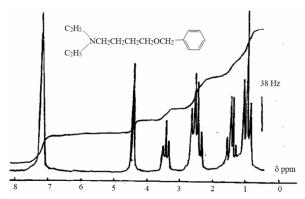


Fig. 8. H-NMR spectrum of N,N-diethyl-4-benzyloxybutylamine (10)

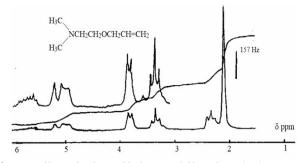


Fig.9. H-NMR spectrum of N,N-dimethyl-2-allyloxyethilamine (11)

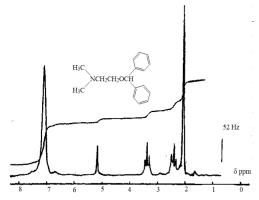


Fig. 10. H-NMR spectrum of N,N-dimethyl-2-benzhydryloxyethilamine (13)

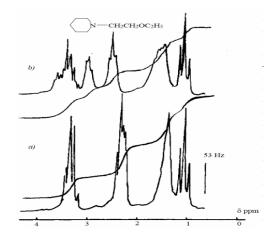


Fig.11. H-NMR spectrum of 1-(2-ethoxyethyl)piperidine (15) in CCl<sub>4</sub> solution and in the presence of CF<sub>3</sub>COOH (6)

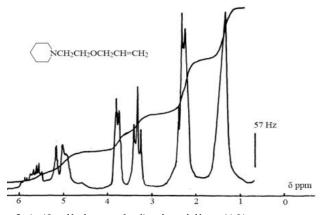


Fig.12. H-NMR spectrum of 1-(2-allyloxyethyl) piperidine (19)

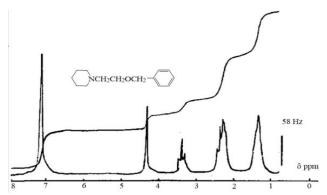


Fig.13. H-NMR spectrum of 1-(2-benzyloxyethyl) piperidine (20)

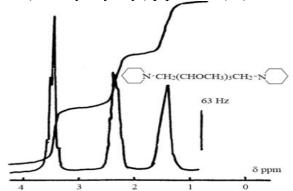


Fig. 14. H-NMR spectrum of 1,11-dipiperidino-3,6,9-trioxaundecane (28)

#### **Methods of experiment**

#### 1. Preparation of 1,2-, 1,3- and 1,4-aminoesters and related compounds.

1.1. Preparation of N,N-diethyl-2-ethoxyethylamine (1). In a three-neck flask equipped with a reflux condenser and a stirrer, dissolve 16.8 g (0.3 mol) of potassium hydroxide in 14-15 ml of water (or 12 g of sodium hydroxide in 12 ml of water) at 25-30°C. Then 50 ml of benzene, 18.15 g (0.1 mol) of chloride or 22.6 g (0.1 mol) of (2-hydroxyethyl)triethylam-monium bromide and 10.9 g (0.1 mol) of ethyl bromide. The reaction mixture is intensively stirred at 25-30°C for 3-4 hours. At the end of mixing, separate the reflux condenser from the reaction flask and equip it with a nozzle equipped with a thermometer and a downward condenser. The gas outlet tube of the downward cooler is connected to a Tishchenko flask and a water-jet pump. The operation of the water jet pump is regulated so that a slightly reduced pressure is maintained in the system. In the Tishchenko flask, an aqueous solution of potassium bromide and bromine is used as a working solution to absorb the formed ethylene. Then benzene is distilled off from the reaction mixture and, without changing the receiver, the temperature in the flask is gradually raised to 120°C and maintained for 20-30 minutes. At 97-100 °C, part of the reaction product is distilled off with water. When finished, cool the flask and add 20 ml of water to the residue. The resulting solution is extracted with distillate. Separating the organic part, drying over sodium sulfate. Then benzene is distilled off and N,N-diethyl-2ethoxyethylamine is isolated from the residue by distillation. Yield 12 g (83%).

The dibromoethane formed in Tishchenko's flask is separated, washed with water, dried over calcium chloride and distilled. Kip. 132°C, 1.5380, 2.1783. Yield 80% of theoretical. Compounds (2-4,7) are prepared similarly (see Table 2).

1.2. Preparation of N,N-diethyl-2-allyloxyethylamine (5). To a mixture of 16.8 g (0.3 mol) of potassium hydroxide in 14-15 ml of water at 15-20°C with vigorous stirring, add 50 ml of benzene,

18.15 g (0.1 mol) chloride or 22.6 g (0.1 mol) (2-hydroxyethyl)-triethylammonium bromide and 7.65 g (0.1 mol) allyl- chloride or 12.1 g (0.1 mol) allylbromide. Stirring of the reaction mixture is continued for 3 hours, and the temperature is adjusted using a water bath so that it does not rise above 30°C. The reaction mixture is then processed as described in Method 1.1. and obtain 11.35 g (72.3%) of compound (5).

Compounds (6,9,10) are prepared similarly (see Table 2).

1.3. Preparation of N,N-dimethyl-2-allyloxyethylamine (11). Using the installation described above, 16.55 g of benzene is added to a mixture of 16.8 g (0.3 mol) of potassium hydroxide, 16.8 g (0.8 mol) of water and 25 ml (2.8 mol) of benzene at 15-20°C (0.1 mol) allyl(2-hydroxysyl)dimethylammonium chloride. The reaction mixture is vigorously stirred and 7.65 g (0.1 mol) of allylchloride is added dropwise, the flow rate of which is adjusted so that the temperature of the medium does not rise above 20°C. After adding allylchloride, the reflux condenser in the installation is replaced with a downward one and the temperature of the reaction mixture is raised to 95-100°C and at the same time benzene is distilled off. Heating is continued for 50-60 minutes, then the flask is cooled, and 20 ml of water is added to the mixture. The aqueous solution is extracted by distillation, washed with water, and dried over Na<sub>2</sub>SO<sub>4</sub>. Then benzene is distilled off and N,N-dimethyl-2-allyloxyethylamine (11) in the amount of 11.5 g (89.1%) is isolated from the residue by distillation.

We have established that, under the conditions of Method 1.3, N,N-dimethyl-2-allyloxyethylamine (11) can be synthesized starting from compounds of a similar structure, but with different halogen atoms in the composition. Thus, N,N-dimethyl-2-allyloxyethylamine (11) was obtained in yields of 92, 87, 90%, respectively, starting from allyl(2-hydroxyethyl)-dimethylammonium bromide and allyl bromide, allyl(2-hydroxyethyl)-dimethylammonium bromide and allyl chloride. Compounds (12,13) are prepared similarly (see Table 2).

1.4. Preparation of 1-(2-ethoxyethyl)piperidine (15). Using the installation described above, 17 g (0.304 mol) of potassium hydroxide is dissolved in 14 ml of water at 20-25 °C. Then 25 ml of benzene, 20.55 g (0.1 mol) of 1-allyl-1-(2-hydroxy-ethyl)piperidinium bromide, dissolved in 3 ml of water and 12 g (0.11 mol) ethyl bromide. Stirring the reaction mixture at the same temperature is continued for another 4 hours. Then the reflux condenser in the installation is replaced with a downward one and the temperature is raised to 100-110 °C, heated for 30-40 minutes and at the same time benzene is distilled off. The residue remaining in the flask is processed as in method 1.3 and obtain compound (15) in the amount of 14.0 g (89.2%). Compounds (16-26) are prepared similarly (see Table 2).

1.5. Preparation of 1,10-di(N-allylpiperidinio)-3,6,9-trioxaundecane bromide (27). In a three-neck cylindrical flask equipped with a reflux condenser, a stirrer and a bottom tap for draining the reaction mixture, dissolve 34 g (0.61 mol) of potassium hydroxide in 28 ml of water. Then, 50 ml of benzene, 20.5 g (0.1 mol) of 1-allyl-1-(2-hydroxyethyl)-piperidinium bromide, dissolved in 6 ml of water and 11, are successively added to the mixture at 20-25°C with vigorous stirring. 6 g (0.05 mol) 1,5-dibromo-3-oxapentane. The reaction mixture is stirred at the same temperature for 8 hours. Then stop mixing. In this case, separation of the reaction mixture occurs within 25-30 minutes. After this, the precipitate of potassium bromide and an aqueous solution of excess potassium hydroxide are drained. 50 ml of water is added to the mixture remaining in the flask and stirred for 15-20 minutes, separated from benzene and acidified with hydrobromic acid until neutral. Then the water is distilled off under reduced pressure. 30 ml of propyl alcohol is added to the residue. The insoluble part is separated by filtration and propanol is distilled from the solution. Compound (27) is recrystallized from acetone. Yield 26.2 g (92%). T.pl. 105-107 °C. Found in %: C 50.30, H 8.32, N 4.76, Br 28.91. C<sub>24</sub>H<sub>46</sub>O<sub>3</sub>N<sub>2</sub>Br<sub>2</sub>. Calculated in %: C 50.53, H 8.07, N 4.91, Br 28.07.

1.6. Preparation of 1,10-dipiperidino-3.6.9-trioxaundecane (28). In a three-neck flask equipped with a reflux condenser and a stirrer, a mixture of 20 g of 40% aqueous potassium

hydroxide and 14.25~g~(0.025~mol)~1,10-di(N-allylpiperi-dino)-3,6,9-trioxaundecane dibromide (27) heated at 95-100 °C for 2-3 hours. At the end of the reaction, cool the flask and add 50 ml of water to the reaction mixture. The organic part is extracted with ether (2x20 ml), washed with water, and dried over Na<sub>2</sub>SO<sub>4</sub>. After drying, the ether is distilled off and compound (28) is isolated from the residue by distillation in the amount of 7.0 g (85.4%).

Table 2. Physical characteristics and analytical data of 1,2-amino alcohols and related

compounds

№	Name of compound	Yield	Boil.te	$n_{D}^{20}$	$d_4^{20}$	Eleme	ent analys	is, in
	Brutto formula	,	m., in		7	%		
		%	<sup>0</sup> C/mm			found./	calculate	d.
						С	Н	N
1	2	3	4	5	6	7	8	9
1	N,N – Diethyl-2-ethoxyethyl	83,0	54-	1,4191	0,8042	66,32	13,27	9,54
	amine C <sub>8</sub> H <sub>19</sub> NO		55/12			66,21	13,10	9,66
2	N,N – Diethyl-2-	82,4	68/14	1,4204	0,8020	67,53	13,32	8,76
	propoxyethylamine C <sub>9</sub> H <sub>21</sub> NO					67,32	13,21	8,81
3	N,N – Diethyl-2-	80,2	82-	1,4235	0,8072	69,42	13,36	8,21
	butoxyethylamine C <sub>10</sub> H <sub>23</sub> NO		83/14			69,36	13,29	8,09
4	N,N – Diethyl-2-	85,7	98/13,	1,4300	0,8119	70,45	13,51	7,62
	pentyloxyethylamine C <sub>11</sub> H <sub>25</sub> NO		5			70,59	13,37	7,49
5	N,N – Diethyl-2-	72,3	94/18	1,4333	0,8356	68,64	12,27	8,81
	allyloxyethylamine C <sub>9</sub> H <sub>19</sub> NO					68,79	12,10	8,92
6	N,N – Diethyl-2-	79,1	116-	1,4986	0,9452	75,12	10,27	6,59
	benzyloxyethylamine C <sub>13</sub> H <sub>23</sub> NO		118/4			75,36	10,14	6,76
7	N-(2-Ethoxyethyl)-N-	86,0	94-	1,4918	0,9329	75,56	10,40	6,58
	ethylbenzylamine C <sub>13</sub> H <sub>21</sub> NO		96/1,5			75,36	10,14	6,76
9	N,N - Diethyl-3-	83,3	127-	1,5087	0,9691	75,87	10,64	6,04
	benzyloxypropylamine		128/10			76,02	10,41	6,33
	$C_{14}H_{23}NO$							
10	N,N - Diethyl-4-	80,0	139-	1,5136	0,9640	76,37	10,83	5,69
	benzyloxybutylamine C <sub>15</sub> H <sub>25</sub> NO		141/10			76,60	10,64	5,96
11	N,N – Dimethyl-2-	89,1	73-100	1,4274	0,8369	65,36	11,47	10,7
	allyloxyethylamine C <sub>7</sub> H <sub>15</sub> NO					65,12	11,62	3
								10,8
								5
12	N,N – Dimethyl-2-	87,0	102-	1,5122	0,9726	73,56	9,36	7,63
	benzyloxyethylamine C <sub>11</sub> H <sub>17</sub> NO		103/4			73,74	9,50	7,82
13	N,N – Dimethyl-2-	85,2	155-	1,5462	1,0198	80,23	8,31	5,54
	benzihydryloxyethylamine		156/5			80,00	8,24	5,49
	$C_{17}H_{21}NO$							
14	1-(2-Ethoxyethyl)piperidine	89,2	84-	1,4499	0,8940	68,47	12,37	8,63
	C <sub>9</sub> H <sub>19</sub> NO		86/16			68,79	12,10	8,92
16	1-(2-Propoxyethyl)piperidine	86,8	96-	1,4507	0,8890	70,37	12,41	8,29
	$C_{10}H_{21}NO$		98/20			70,18	12,28	8,19
17	1-(2-Butoxyethyl)piperidine	87,1	107-	1,4496	0,8815	71,64	12,51	7,28
	$C_{11}H_{23}NO$	<u> </u>	108/14			71,35	12,43	7,57

18	1-(2-Pentyloxyethyl)piperidine	86,8	91-	1,4514	0,8788	72,60	12,78	7,31
	$C_{12}H_{25}NO$		92/2,5			72,36	12,56	7,04
19	1-(2-Allyloxyethyl)piperidine	83,7	99-	1,4670	0,9130	71,30	11,52	8,43
	$C_{10}H_{19}NO$		100/15			71,01	11,24	8,28
20	1-(2-Benzyloxyethyl)piperidine	87,4	132-	1,5170	0,9940	76,58	9,41	6,16
	$C_{14}H_{21}NO$		133/3			76,71	9,59	6,39
21	1(2Ethoxyethyl)hexahydroazepi	87,1	107-	1,4554	0,8960	70,01	12,53	8,27
	ne C <sub>10</sub> H <sub>21</sub> NO		109/15		Í	70,18	12,28	8,19
22	1-(2-	86,2	120-	1,4558	0,8910	71,07	12,64	7,29
	Propoxyethyl)hexahydroazepine		121/16		Í	71,35	12,43	7,58
	$C_{11}H_{23}NO$							
23	1-(2-	83,4	135-	1,4622	0,8970	72,49	12,71	6,89
	Butoxyethyl)hexahydroazepine		137/15			72,36	12,56	7,04
	$C_{12}H_{25}NO$							
24	1-(2-	89,7	118-	1,4667	0,8997	73,51	12,47	6,29
	Pentyloxyethyl)hexahydroazepin		120/2		Í	73,24	12,68	6,57
	e C <sub>13</sub> H <sub>27</sub> NO							
25	1(2Allyloxyethyl)hexahydroazep	88,1	120-	1,4719	0,9158	72,40	11,25	7,82
	ine C <sub>11</sub> H <sub>21</sub> NO		122/15			72,13	11,48	7,65
26	1(2Benzyloxyethyl)hexahydroaz	92,1	147-	1,5226	0,9976	77,46	9,64	6,27
	epine C <sub>15</sub> H <sub>23</sub> NO		149/3			77,25	9,87	6,01
28	1,10-Dipiperidino-3,6,9-	85,4	184-	1,4790	0,9926	65,63	10,72	8,63
	trioxaundecane C <sub>18</sub> H <sub>36</sub> N <sub>2</sub> O <sub>2</sub>		186/3			65,85	10,98	8,54

#### 2. Preparation of simple vinyl ethers

- 2.1. Preparation of vinyl butyl ether (33) using (2-hydrocoethyl)- trimethylammonium chloride. In a flask equipped with a stirrer and a separating funnel, a mixture of 12 g (0.3 mol) sodium hydroxide, 12 g (0.67 mol) water, 13.95 g (0.1 mol) (2-hydroxyethyl) trimethylammoni-um chloride is gradually added 13.7 g (0.1 mol) butyl bromide at 5-10 °C with vigorous stirring of the reaction mixture. After adding butylbromide, the reaction mixture is stirred for another 1.5-2 hours. Then, heating the flask, raise the temperature of the reaction mixture to 110-115°C. The azeotropic mixture collected in the receiver is washed with water, dried over K<sub>2</sub>CO<sub>3</sub> and distilled over K<sub>2</sub>CO<sub>3</sub>. Vinylbutyl ether (33) is obtained in a yield of 9.2 g (92%). Compounds (29-32, 34-37) are prepared similarly (see Table 3). When using methyl bromide, cool the separatory funnel with ice.
- 2.2. Preparation of vinylbutyl ester (33) using (2-hydroxyethyl) trimethylam-monium (choline) hydroxide. In the above-described installation, 12 g (0.207 mol) of potassium hydroxide are dissolved in 24.1 g of a 50% aqueous solution of (2-hydroxy-ethyl) trimethylammonium hydroxide at 5-10°C. Then, butyl bromide is gradually added to the mixture at the same temperature with vigorous stirring. After adding butyl bromide, the reaction mixture is stirred for another 1.5-2 hours and processed as described in method 2.1. and obtain vinylbutyl ether (33) in the amount of 9.0 g (90%).

Compounds (29-32, 34-38) were prepared similarly.

2.4. Preparation of vinyl butyl ether (33) starting from butyl alcohol and acetylene<sup>288</sup> [12](counter synthesis). In an autoclave with a volume of at least 1000 ml made of alloy steel with a stirrer, tested at 350 atm. and not containing silver and copper parts, place 74 g (0.1 mol) of butyl alcohol and 8 g of finely ground potassium hydroxide. Then the autoclave is closed, the air is carefully replaced with acetylene, and acetylene is supplied to a pressure of 16 atm. and slowly heat to 140-160 °C. As soon as the acetylene pressure drops and becomes constant, the autoclave is cooled and acetylene is supplied again to a pressure of 8-16 atm. and again heated to 140-160 °C. This

operation is repeated twice more. The autoclave is then cooled and the pressure is released. Potassium hydroxide and vinyl ether are separated from the reaction mixture. The ether is distilled off with water vapor. Then, after separating the water from the mixture, vinyl butyl ether is dried over K<sub>2</sub>CO<sub>3</sub> and distilled on a 20-centimeter Vigreux column. Yield 74%.

Compounds (29-32, 34-37) were prepared similarly. Identification of vinyl esters is carried out by GLC analysis.

**Table 3.** Physicochemical characteristics of vinyl esters obtained using (2- hydroxyethyl) trimethylammonium chloride and hydroxide and N-(2-hydroxyethyl) pyridinium chloride

№	Names of	Initial	Yiele	d, in %	Boil.	$n_{D}^{20}$	$d_4^{20}$	Elemen	ntal
$N_{\underline{0}}$	compounds, gross	halide		Hydroxi	temp.,			analysi	is, in
	formula		e (2-	de (2-	in °C			%	
			-	hydroxye				найд./і	вычис
			yethyl)	thyl)-					T.T.
				trimethyl				С	Н
			ylamm onia	ammonia					
1	2	3	4	5	7	8	9	10	11
29	Vinylmethyl ether	CH <sub>3</sub> Br	87	89	6	1,3730	0,7725		10,20
	C <sub>3</sub> H <sub>6</sub> O							62.07	10.34
30	Vinylethyl ether	C <sub>2</sub> H <sub>5</sub> Br	88	84	36	1,3768	0,7589	66,43	11,32
	C <sub>4</sub> H <sub>8</sub> O							66,67	11,11
31	Vinylpropyl ether	C <sub>3</sub> H <sub>7</sub> Br	89	86	65	1,3920	0,7670	69,54	11,47
	$C_5H_{10}O$							69,77	11,63
32	Vinylisopropyl ether	<i>i</i> -C <sub>3</sub> H <sub>7</sub> Br	78	76	47	1,3842	0,7596	69,90	11,37
	$C_5H_{10}O$							69,77	11,63
33	Vinylbutyl ether	C <sub>4</sub> H <sub>9</sub> Br	92	90	94	1,4020	0,7886	72,27	12,18
	$C_6H_{12}O$							72,00	12,00
34	Vinylizobutyl ether	<i>i</i> -C <sub>4</sub> H <sub>9</sub> Br	87	89	83	1,3981	0,7820	72,20	12,27
	$C_6H_{12}O$							72,00	12,00
35	Vinylpentyl ether	C <sub>5</sub> H <sub>11</sub> Br	94	91	118	1,4113	0,7837	73,41	12,51
	C <sub>7</sub> H <sub>14</sub> O							73,68	12,28
36	Vinylizopentyl ether	<i>i</i> -C <sub>5</sub> H <sub>11</sub> Br	90	92	113	1,4072	0,7826	73,93	12,01
	C <sub>7</sub> H <sub>14</sub> O							73,68	12,28
37	Vinylbenzyl ether	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> Cl	86	87	47-	1,5185	0,9862	80,37	7,61
	$C_9H_{10}O$				48/15			80,59	7,46
					MM				

#### **Conclusions and discussions**

So, the possibility of alkylation of the OH group in quaternary compounds of 1,2-aminoalcohols at 0-20°C in a system of 50% aqueous alkali and benzene without the addition of a catalyst under extremely mild conditions has been demonstrated. It has been established that the patterns typical for the reaction of quaternary salts of 1,2-aminoalcohols, under the same conditions, are characteristic of quaternary salts of bis-, tris(2-hydroxyethyl)amines, 1,3- and 1,4-amino alcohols and related compounds.

The alkaline cleavage of quaternary compounds of 1,2-aminoesters was studied for the first time. Salts with an ethyl group at the nitrogen atom form 1,2-, 1,3- and 1,4-aminoester and ethylene, and with an allyl group - 1,2-aminoester and allylic alcohol. Tertiary amine and vinyl ether are formed when the nitrogen atom has methyl groups.

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## 1,2-AMİNOESTERLƏRİN VƏ BAĞLANTILI BİRLƏŞMƏLƏRİN ALKALİ KLİVİCA

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Xülasə: İşdə müxtəlif hidrokarbon qrupları ilə zəngin 1,2-aminoesterlərin və əlaqəli birləşmələrin kuaternar duzlarının alkali parçalanma reaksiyaları tədqiq edilmişdir. Bu reaksiyalar, O, N

heteroatomları və C=C dublet bağını ehtiva edən orqanik birləşmələrin sintezini hədəfləyir. Tədqiqatlar nəticəsində, kuaternar birləşmələr olan 1,2-aminoalkol və əlaqəli birləşmələrin OH qrupunun alkilasyonunun 50% sulu alkali və benzol sistemində 0-200°C şərtlərində yumşaq şəraitdə baş verdiyi müəyyən edilmişdir. Həmçinin, aşkar edilmişdir ki, kuaternar 1,2-aminoalkol duzlarının alkil halogenidlərlə reaksiyası, həmin şərtlər altında, müvafiq kuaternar aminoesterlərin demək olar ki, kəmiyyət baxımından əmələ gəlməsinə gətirib çıxarır.

Məlum olmuşdur ki, bu aminoester duzları 100-120°C-də asanlıqla dekompozisiyaya uğrayır. Bu zaman, yeni əlaqələrin yaranması və köhnə əlaqələrin pozulması, kuaternar azot atomunda yerləşən təbii qruplarla müəyyən edilir. 1,2-aminoesterlərin kuaternar duzlarının etil qrupunu asanlıqla etilen şəklində itirdiyi və bu zaman 2-alkoksietil qrupuna toxunulmadığı və müvafiq 1,2-aminoesterlərin əmələ gəldiyi müəyyən edilmişdir.

Həmçinin aşkar edilmişdir ki, 1,2-aminoalkolların kuaternar duzlarının reaksiyası üçün tipik olan qaydalar, 1,3- və 1,4-aminoalkolların duzları üçün də xarakterikdir. Çünki müvafiq kuaternar duzlarının 1,3- və 1,4-aminoesterlərinin alkali parçalanması da eyni şəkildə baş verir. Həmçinin, 1,2-aminoesterlər duzlarının alkali parçalanmasının araşdırılmasında maraqlı nəticələr əldə edilmişdir. Bu duzlar, müvafiq 1,2-aminoalkolların kuaternar duzlarının orqanohalogenlərlə reaksiyası ilə əmələ gəlmişdir və bu reaksiyalarda allylik qrupunun allylik spirt şəklində asanlıqla çıxması və müvafiq 1,2-aminoesterlərin əmələ gəlməsi müşahidə edilmişdir. Çox maraqlı nəticələr əldə edilmişdir, çünki 2-hidroksietil trimetilamonium hidroksid və ya xlorid ilə orqano-halogenlərlə reaksiyadan yaranmış müvafiq 1,2-aminoesterlər kuaternar duzlarının alkali parçalanması tədqiq edilmişdir. Bu zaman metil qruplarının azot atomunda olması ilə 1,2-aminoesterlərin kuaternar duzlarının 2-alkoksietil qrupunun parçalanması, 1,2-aminoesterlərin siklik duzları ilə eyni şəkildə baş verir. Bu nəticədə vinil eterləri əmələ gəlir.

**Açar sözlər:** kuaternar duzlar, 1,2-aminoalkollar, 1,2-aminoesterlər, vinil eterləri, 1,2-aminoalkolların kuaternar duzları, 1,2-aminoesterlərin kuaternar duzları, 2-trialkylammonioetoksidlər, alkali məhlullar.

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#### DIRECTIONS FOR THE DEVELOPMENT OF GREEN ENERGY IN AZERBAIJAN

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The article is dedicated to meeting the socio-economic needs of the country's population by increasing the share of environmentally friendly and renewable energy sources in Azerbaijan's modern energy supply. It is known that Azerbaijan has a significant amount of fuel and energy resources. However, in recent years, the Republic of Azerbaijan, like all countries in the world, has been attracting new energy sources to its energy balance. However, in recent years, the Republic of Azerbaijan, like all countries in the world, has been attracting new energy sources to its energy balance. In the context of declining natural resources and increasing demand in the world, it is important to implement major measures to expand the "green" economy in order to achieve sustainable development. The article sets out a number of tasks related to increasing the application of environmentally friendly technologies, promoting waste recycling and restoration of contaminated areas, and expanding the application of environmentally friendly "green" technologies. It was concluded that Azerbaijan's favorable geographical location and climatic conditions allow for the widespread use of environmentally friendly alternative energy sources.

Keywords: Karabakh, Karabakh economic region, "green energy", "green energy" opportunities of Karabakh, "green energy" infrastructure, directions of development of "green energy", expansion of the application of "green" technologies.

#### Introduction

Increasing the use of "green energy" is coming to the forefront in the world as one of the means of solving environmental problems in the 21st century. Green energy refers to the use of alternative energy sources (solar, water, wind, etc.) in various fields of human activity. Recently, the topic of ecology and resource conservation has been widely discussed and reflected in the works of various scholars. The term "green energy" is becoming widespread in the media, as well as in the scientific community. This topic has become increasingly relevant with its inclusion in international and national programs and conferences. It is believed that sustainable development is a concept put forward on how to build the future world in the current conditions so as not to disrupt the development of the new (future) generation. The world is increasingly focusing on climate and environmental risks, and on the modification of tools and infrastructure for sustainable development. In this concept, the use of green economy is a tool that encompasses suitable methods and means by which the intended economic result can be achieved [2].

Azerbaijan is a country rich in renewable energy resources. It is noted that the country has a potential of 135 GW in land areas and 157 GW in maritime areas. In addition, wind energy potential has been estimated at 3,000 MW, solar energy potential at 23,000 MW, and bioenergy potential at 380 MW. These figures show that Azerbaijan has high potential to benefit from renewable energy resources. In the country's economy, where green energy is at the forefront, a large number of private and public investment activities are primarily aimed at increasing the efficient use of natural resources; reducing negative factors that directly affect the decline of

biodiversity; and reducing the emission of harmful substances into the atmosphere, which ultimately leads to a reduction in global pollution of all nature. As civilization develops and the world's population grows rapidly, and production capacities increase, there is a demand for more fuel resources, and these are considered one of the most complex problems of the modern era. Thus, the main sources of fuel resources used today are exhaustible sources, and these resources are also unevenly distributed around the world. Many countries around the world lack access to important energy resources, and the struggle for energy is becoming increasingly global [2].

It should be noted that Azerbaijan has already gone down in history as one of the rare countries that has restored and revived territories liberated from occupation through its financial and economic power. It is believed that the main goal of organizing the "Green Energy Zone" in Karabakh is to create renewable energy reserves and, in general, to take targeted measures to reduce carbon dioxide emissions in our country. It should be noted that the decree of the head of state dated May 3, 2021, envisages measures to create a "green energy" zone in the liberated territories. Energy projects implemented in a complex and systematic manner in these areas will soon be of strategic importance in creating an exemplary and efficient "green energy" space in Azerbaijan. These factors will also provide a significant boost to the development of our country's non-oil sector. Professor V.A. Gasimli and a group of researchers are of the opinion that: "The essence of the "green energy" approach adopted in Azerbaijan is that, on the one hand, energy will increasingly be obtained from renewable sources, and on the other hand, the energy savings we make are more efficient than producing the same amount of energy. As part of the "Green Growth" concept, a policy is being implemented in Azerbaijan to transform the Karabakh and East Zangezur regions into "green energy" zones [3].

Another important issue is the historic work that Azerbaijan has begun to transition to a new socio-economic development model based on the principles of "green energy" and "green economy". These factors not only strengthen the economic security of our country, but also create additional potential for significantly increasing energy security, which is an important component of it.

It should be noted that one of the main directions in the National Priorities for Socio-Economic Development is the formation of a "Country with a Clean Environment and Green Growth" by 2030. Taking this into account, 2024 was declared the "Year of Solidarity for a Green World" by the Decree of President Ilham Aliyev. Another historic and important issue is the holding of the next Session of the Conference of the Parties (COP-29) of the UN Framework Convention on Climate Change in Azerbaijan in November 2024. By hosting such an internationally prestigious event, Azerbaijan has also significantly increased its international image and competitiveness.

The construction of modern villages and cities based on "smart" technologies in the Karabakh economic region, the intensive implementation of the First State Program on the Great Return, and the creation of a "green energy" zone are having a high impact on the development of the region. Similar development processes are shaping a new advanced and modern model of regional development in our country [6].



Table 1: Energy production; Source: Energetika Nazirliyi

According to operational data for January 2025, electricity production in the republic last month was 2,493.4 million kWh. During the month, electricity production at thermal power plants amounted to 2,224.9 million kWh, at hydroelectric power plants 218.3 million kWh, and from other sources 50.2 million kWh. During this period, 2.9 million kWh of electricity was produced at wind power plants, 26.4 million kWh at solar power plants, and 20.9 million kWh at the Solid Waste Incineration Plant. Compared to the same period last year, an increase of 96.4 million kWh was recorded in electricity production from renewable energy sources, including hydroelectric power plants. During the month, electricity production by "Azerenergy" OJSC amounted to 2,232.6 million kWh (2,021.6 million kWh at thermal power plants, 211.0 million kWh at hydroelectric power plants), 32.0 million kWh at the Nakhchivan Autonomous Republic State Energy Service (22.4 million kWh at hydroelectric power plants), 2.0 million kWh at hydroelectric power plants, 3.2 million kWh at hydroelectric power plants), 2.0 million kWh at wind power plants by "Azerishig" OJSC, and 226.8 million kWh at independent power plants. During this period, electricity exports amounted to 157.4 million kWh, while imports amounted to 11.2 million kWh[7].

I would like to inform you that environmental problems are becoming acute worldwide. Climate change is making it difficult to secure essential food supplies. Land plots are being abandoned or flooded due to drought. In addition, to ensure sustainable development, it is necessary to conserve resources and take into account the needs of future generations. This requires the development of "green energy" sectors and the establishment of green economy principles. In such a situation, Azerbaijan, while sharing its oil and gas resources primarily with the countries of the European Union, attaches strategic importance to the development of renewable energy sources. The renewable energy potential in Azerbaijan is 135 GW onshore and 157 GW offshore. Also, the wind energy potential is 3,000 MW, and the solar energy potential is 23,000 MW. Currently, 1.7 thousand of the 8.3 thousand MW of electricity produced in our country is due to the capacity of hydroelectric power plants. This means a 20% share. The expansion of the network of hydroelectric power plants in the liberated territories will create conditions for increasing the share of "green energy" [4].

It should be noted that a strong electricity network was formed in the Karabakh region during the Soviet Union, and 10 electricity networks operated here. The hated enemy completely plundered this infrastructure during the occupation. In the current reality, the region's strong energy resources and the fact that it attracts attention with its great potential give reason to say that a new energy infrastructure and network based on more modern technologies will soon be created in Karabakh. If we look at the resource possibilities for this, we will see that there is a prospect of creating a network of hydroelectric power stations at the base of the rivers Tartar, Bazarchay, Hakari, etc. Work in this direction has already been improved and some previously operating hydroelectric power stations have been restored. Thus, the restored 20 MW hydroelectric power station was put into operation in Sugovushan ("Sugovushan-1" and "Sugovushan-2"), etc. In addition to the above, we believe that in the near future, more intensive development of green energy infrastructure will be ensured in the Karabakh economic region [5].

#### Result

Azerbaijan has significant potential for green energy production due to its geographical and climatic advantages. The country is developing various projects aimed at increasing the use of green energy and reducing carbon emissions in line with its sustainable development goals. The planned establishment of the "Green Energy Zone" in the Karabakh and East Zangezur regions is an important part of these efforts. Among Azerbaijan's renewable energy sources, solar, wind, and hydroelectric energy hold a significant position. The country's total renewable energy potential is estimated at 135 GW on land and 157 GW in offshore areas. Additionally, the wind energy potential is calculated at 3,000 MW, solar energy potential at 23,000 MW, and bioenergy potential at 380 MW. The transition to green energy not only provides environmental benefits but also enhances Azerbaijan's energy independence and supports its economic development. The designation of 2024 as the "Year of Solidarity for a Green World" and the hosting of COP-29 in Azerbaijan in November 2024 are significant steps in strengthening the country's international environmental commitments.

In this context, increasing investments in renewable energy, strengthening infrastructure, and promoting green technologies will enable Azerbaijan to advance on the path of sustainable development. In the long term, the effective implementation of green energy policies will reduce the country's dependence on fossil fuels and bring significant gains in environmental sustainability.

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## AZƏRBAYCANDA YAŞIL ENERJİNİN INKIŞAFI ÜZRƏ İSTİQAMƏTLƏR

#### Sevda Qasımova, Gülnar Məmmədova

## Azərbaycan Memarlıq və İnşaat Universiteti

Məqalə Azərbaycanın müasir enerji təchizatında ekoloji cəhətdən təmiz və bərpa olunan enerji mənbələrinin payının artırılması yolu ilə ölkə əhalisinin sosial-iqtisadi tələblərinin ödənilməsi məsələsinə həsr olunub. Məlumdur ki, Azərbaycan mühüm miqdarda yanacaq və enerji ehtiyatlarına malikdir. Lakin son illərdə Azərbaycan Respublikası bütün dünya ölkələri kimi, enerji balansına yeni enerji mənbələri daxil etməyə başlayıb. Təbiət ehtiyatlarının azalması və dünya üzrə artan tələbat kontekstində, dayanıqlı inkişafı əldə etmək üçün "yaşıl" iqtisadiyyatın genişləndirilməsi məqsədilə mühüm tədbirlərin həyata keçirilməsi vacibdir. Məqalədə ekoloji cəhətdən təmiz texnologiyaların tətbiqinin artırılması, tullantıların təkrar emalının təşviqi və çirklənmiş ərazilərin bərpası, həmçinin ekoloji cəhətdən təmiz "yaşıl" texnologiyaların tətbiqinin genişləndirilməsi ilə bağlı bir sıra vəzifələr qoyulmuşdur. Nəticədə, Azərbaycanın əlverişli coğrafi mövqeyi və iqlim şəraiti ekoloji cəhətdən təmiz alternativ enerji mənbələrinin geniş istifadə olunmasına imkan verir.

Açar sözlər: Qarabağ, Qarabağ iqtisadi rayonu, "yaşıl enerji", Qarabağ "yaşıl enerji" imkanları, "yaşıl enerji" infrastrukturu, "yaşıl enerji"nin inkişaf istiqamətləri, "yaşıl" texnologiyaların tətbiqinin genişləndirilməsi.

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## XƏZƏR DƏNİZİ REGİONUNDA MƏSKUNLAŞMIŞ BİOFİLTRLƏR VƏ BUNLARIN EKOSİSTEMDƏKİ ƏSAS ROLU

## L.İ.Əsədli t.e.n, dos.N.A.Vəliyev

## Azərbaycan Memarlıq və İnşaat Universiteti

Xəzər dənizi dünyanın ən böyük qapalı su hövzəsi olmaqla yanaşı, zəngin bioloji müxtəlifliyə malik bir ekosistemdir. Bu ekosistemdə müxtəlif təbii filtrasiya sistemləri, o cümlədən biofiltrlər, dəniz suyunun özünü təmizləməsi və çirkləndiricilərin biodeqradasiyası prosesində mühüm rol oynayır. Antropogen təsirlər, xüsusilə də neft və neft məhsulları ilə çirklənmə, Xəzərin ekoloji sabitliyini pozsa da, biofiltrlər dəniz mühitinin özünübərpa qabiliyyətinin əsas təbii komponentlərindən biri kimi çıxış edir. Bu məqalədə Xəzər dənizinin biofiltr orqanizmləri, onların təsnifatı, fəaliyyət mexanizmləri və ekosistemdəki rolu elmi əsaslarla təhlil edilir, habelə onların mühafizəsi və bərpası araşdırılır.

Açar sözlər: Xəzər dənizi, biofiltr, ekosistem, neft çirklənməsi, mikroorqanizmlər, fitobentos, biodeqradasiya, ekoloji sabitlik

#### **GİRİS**

Xəzər dənizi yalnız dünyanın ən böyük qapalı su hövzəsi kimi deyil, həm də özünəməxsus geoloji, hidrobioloji və ekoloji xüsusiyyətləri ilə seçilən bənzərsiz bir dənizdir. Onun unikal coğrafi quruluşu və iqlim xüsusiyyətləri dənizdə zəngin və spesifik bioloji müxtəlifliyin formalaşmasına səbəb olmuşdur. Bu dəniz hövzəsində rast gəlinən bir çox flora və fauna növləri endemik xarakter daşıyır, yəni yalnız Xəzərə məxsusdur. Belə bənzərsiz bioloji müxtəliflik və ekosistem zənginliyi, dənizin regional və qlobal əhəmiyyətini artırır.

Xəzər dənizi həmçinin təbii filtrasiya sistemlərinin, xüsusilə də biofiltrlərin fəaliyyət göstərdiyi nadir dəniz mühitlərindən biridir. Biofiltrlər müxtəlif orqanizmlərdən — bakteriyalar, yosunlar, molyusklar və digər filtrasiya edən canlılardan ibarət olub, su mühitindəki üzvi və qeyriüzvi çirkləndiricilərin parçalanmasında və ya udulmasında iştirak edirlər. Onlar dənizin özünü təmizləmə qabiliyyətini formalaşdıran əsas bioloji mexanizmlərdən biri hesab olunur. Beləliklə, biofiltrlər yalnız suyun keyfiyyətini yaxşılaşdırmaqla kifayətlənmir, həm də ümumi ekosistem dayanıqlığının qorunmasında mühüm funksiyalar yerinə yetirirlər.Lakin son onilliklərdə Xəzər dənizində müşahidə olunan intensiv antropogen təzyiqlər, xüsusilə də neft və qaz sənayesinin genişlənməsi, urbanizasiya, sənaye tullantılarının və məişət sularının birbaşa dənizə axıdılması, dəniz ekosisteminin tarazlığını ciddi şəkildə pozmuşdur. Neft və neft məhsulları ilə çirklənmə həm kimyəvi, həm də bioloji parametrlərin dəyişməsinə səbəb olaraq su orqanizmlərinin yaşamaq və çoxalmaq qabiliyyətini azaldır. Bu da öz növbəsində biofiltr orqanizmlərin fəaliyyətinə və populyasiyasının davamlılığına birbaşa mənfi təsir göstərir.

Bununla yanaşı, aparılmış tədqiqatlar göstərir ki, müəyyən şəraitdə biofiltr orqanizmlər çirklənməyə qarşı adaptasiya qabiliyyətinə malikdirlər və hətta çirklənmənin ilkin mərhələlərində mühitdə öz fəaliyyətlərini gücləndirə bilirlər. Məsələn, bəzi mikroorqanizmlər — Pseudomonas, Bacillus, Rhodococcus kimi bakteriya növləri — polisiklik aromatik karbohidrogenləri və digər zərərli maddələri parçalamaq üçün spesifik fermentlər istehsal edir. Eyni zamanda, yosunlar və su bitkiləri, məsələn Ulva, Cladophora, Chara və Zostera kimi növlər, su sütununda olan qida maddələrini (ammonium, nitrat, fosfat və s.) udaraq həm trofik səviyyəni balanslaşdırır, həm də eutrofikasiya riskini azaldır.

Biofiltrlərin fəaliyyət mexanizmləri yalnız filtrasiya və sorbsiya ilə məhdudlaşmır. Onlar həm də biokimyəvi çevrilmələr yolu ilə çirkləndiriciləri daha az toksik formaya salaraq ətraf

mühitə uyğunlaşdırırlar. Məsələn, neft komponentlərinin parçalanması zamanı bəzi bakteriyalar oksidləşmə-fermentasiya mexanizmlərindən istifadə edərək bu maddələri karbon dioksid və suya qədər parçalaya bilirlər. Bu isə təbii ekoloji proseslərin dayanıqlığını qoruyaraq, antropogen təzyiqlərin mənfi təsirlərini minimuma endirə bilər.

Bu kontekstdə, Xəzər dənizində biofiltr orqanizmlərin ekologiyası, onların taksonomik müxtəlifliyi, fəaliyyət səmərəliliyi və çirklənməyə qarşı adaptiv mexanizmlərinin araşdırılması son dərəcə aktuallaşmışdır. Həmçinin, onların qorunması və bərpa olunması istiqamətində təbii mühafizə tədbirlərinin və biotexnoloji yanaşmaların tətbiqi ekosistemin uzunmüddətli dayanıqlığını təmin etmək baxımından əhəmiyyətlidir.

Bu məqalədə Xəzər dənizinin biofiltr orqanizmləri müxtəlif istiqamətlər üzrə elmi əsaslarla təhlil olunur. Əsas məqsəd — bu orqanizmlərin növ tərkibini, ekosistemdəki funksional rolunu, antropogen təsirlərə qarşı göstərdikləri bioloji reaksiyaları və onların gələcəkdə dəniz mühitinin bərpasında istifadəsinin perspektivlərini təqdim etməkdir. Bununla yanaşı, biomüxtəlifliyin qorunması, dəniz ekosistemlərinin bərpası və dayanıqlı idarə olunması baxımından biofiltrlərin strateji əhəmiyyətinə diqqət yetiriləcəkdir.

#### Biofiltlərin növləri

Biofiltrlər adətən aşağıdakı əsas kateqoriyalara ayrılır:

Mikroorqanizm əsaslı biofiltrlər: bakteriya, göbələk və protozoalar neft və digər üzvi çirkləndiriciləri parçalayaraq onların toksikliyini azaldır.

Fitobiofiltrlər: yosunlar və su bitkiləri, o cümlədən fitoplankton və makrofitlər qida maddələrinin və ağır metalların udulmasında iştirak edirlər.

Zoobiofiltrlər: filtrasiya ilə qidalanan heyvanlar (məsələn, molyusklar və bəzi xərçəngkimilər) suda üzən hissəcikləri və bakteriyaları udaraq həm qidalanır, həm də suyu təmizləyir.

Bu sistemlər təkcə çirklənmənin qarşısını almaqla kifayətlənmir, həm də ekosistemin bioloji tarazlığını qoruyur

#### Fitoplankton və makrofitlər:

Xəzər dənizinin sahilyanı zonalarında rast gəlinən Cladophora, Enteromorpha və Ulva kimi yaşıl yosun növləri, xüsusilə də dayaz sahələrdə biofiltr funksiyasını yerinə yetirirlər. Bu bitkilər su sütununda olan ammonyak, nitrat və fosfatları udaraq eutrofikasiya riskini azaldır

#### Molyusklar və digər filtrasiya edən fauna:

Xəzər dənizinin bentik zonasında geniş yayılan Dreissena polymorpha (zebra molyuskası) və Mytilaster lineatus kimi molyusk növləri suda üzən üzvi maddələri və mikroorqanizmləri süzərək qidalanır və eyni zamanda biofiltr rolunu yerinə yetirirlər

#### Mikroorganizm birləşmələri:

Neftlə çirklənmiş ərazilərdə Pseudomonas, Acinetobacter, Rhodococcus və Bacillus cinslərinə aid bakteriyalar neft məhsullarının biodeqradasiyasında əsas rol oynayır Biofiltrlərin neft və neft məhsulları ilə mübarizədə rolu:Xəzər dənizində neft sızmaları və hasilatı zamanı baş verən çirklənmələr əsasən polisiklik aromatik karbohidrogenlər (PAK-lar) və alifatik karbohidrogenlərlə bağlıdır. Bu birləşmələrin birbaşa parçalanması üçün təbii biofiltr sistemləri əsas rol oynayır.

#### Məsələn:

- Bacillus subtilis növü PAK-ları parçalayaraq daha az zərərli maddələrə çevirir.
- Ulva lactuca yosunu dəniz suyundakı ağır metalları və fenol birləşmələrini udaraq fitofiltrasiya prosesində iştirak edir.

Neft platformalarının fəaliyyəti, məişət tullantılarının birbaşa axıdılması və kənd təsərrüfatı axıntıları Xəzər dənizinin biofiltr sistemlərinə ciddi ziyan vurur. Xüsusilə də mikroorqanizm populyasiyalarında azalma və yosun çiçəklənməsinin artması nəticəsində biofiltr funksiyası zəifləyir. Araşdırmalara görə bəzi mikroorqanizm tiplərinin müxtəlifliyində 25-30%-lik azalma qeydə alınmışdır.

- Biotexnoloji yanaşmalar: Genetik modifikasiya olunmuş bakteriya ştammlarının tətbiqi ilə biofiltr effektivliyi artırıla bilər.
- Mühafizə zonalarının yaradılması: Biofiltr sistemlərinin yaşadığı zonaların antropogen təsirdən qorunması üçün xüsusi ekoloji bufer zonalar yaradılmalıdır.
- Biomonitorinq proqramları: Biofiltr komponentlərinin vəziyyəti üzrə daimi nəzarət və ekoloji risklərin erkən aşkarlanması məqsədilə biomonitorinq sistemləri tətbiq olunmalıdır.

#### **NƏTİCƏ**

Xəzər dənizinin biofiltr sistemləri onun ekoloji davamlılığında və özünübərpa qabiliyyətində əvəzsiz rol oynayır. Bu təbii mexanizmlər antropogen təsirlərlə mübarizədə və neft çirklənməsinin azaldılmasında mühüm bioloji alət kimi çıxış edir. Bu səbəbdən, onların qorunması və stimullaşdırılması ekoloji siyasətin prioritet istiqamətlərindən biri olmalıdır.

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## BIOFILTERS INHABITING THE CASPIAN SEA REGION AND THEIR KEY ROLE IN THE ECOSYSTEM

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The Caspian Sea, as the world's largest enclosed body of water, harbors an ecosystem rich in biological diversity. Within this ecosystem, various natural filtration systems, including biofilters, play a crucial role in the self-purification of seawater and the biodegradation of pollutants. Although anthropogenic influences—particularly oil and petroleum product pollution—disrupt the ecological stability of the Caspian Sea, biofilters act as one of the primary natural components supporting the sea's self-restoration capacity. This article provides a scientific analysis of biofilter organisms in the Caspian Sea, their classification, functional mechanisms, and ecological roles, as well as explores strategies for their conservation and restoration.

Keywords: Caspian Sea, biofilter, ecosystem, oil pollution, microorganisms, phytobenthos, biodegradation, ecological stability

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#### UTILIZATION OF MICROORGANISMS IN HYDROGEN PRODUCTION

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The urgent need for sustainable and clean energy sources has directed significant research attention toward biological hydrogen production via microorganisms. This article reviews the fundamental mechanisms by which various microorganisms—including photosynthetic bacteria, fermentative bacteria, and archaea—generate hydrogen under anaerobic or light-driven conditions. It highlights key metabolic pathways such as biophotolysis, photofermentation, and dark fermentation, alongside the role of enzymes like hydrogenases and nitrogenases. The integration of these microorganisms within optimized bioreactor systems is discussed as a critical factor for enhancing hydrogen yield and process scalability. While biological hydrogen production offers environmental advantages such as low energy input and utilization of renewable substrates, challenges including low conversion efficiencies, enzyme oxygen sensitivity, and gas collection remain. Advances in metabolic engineering, synthetic biology, and bioprocess optimization are identified as promising strategies to overcome current limitations and facilitate the commercial viability of microbial hydrogen technologies. This review underscores the potential of microbial hydrogen production as a key component in the transition toward a sustainable hydrogen economy.

Key word: Biohydrogen, photosynthetic bacteria, biophotolysis, renewable energy, synthetic biology, hydrogen, hydrogenase enzyme

#### Introduction

In the context of increasing global energy demand and growing environmental concerns, the development of sustainable and eco-friendly energy sources has become a top priority. Hydrogen gas is considered one of the most promising alternatives to fossil fuels due to its high energy yield and clean combustion, producing only water as a byproduct. Traditional hydrogen production techniques, such as steam methane reforming and electrolysis, are often energy-intensive and environmentally taxing. Consequently, biological hydrogen production, particularly through the use of microorganisms, has emerged as a viable and sustainable approach to clean energy generation.

Principles of Microbial Hydrogen Production

Microorganisms produce hydrogen as a metabolic byproduct, typically under anaerobic conditions. The key enzymes responsible for this process are hydrogenases and nitrogenases, which catalyze the reduction of protons to molecular hydrogen. Biological hydrogen production can occur via several pathways, depending on the type of microorganisms involved and the environmental conditions provided. These pathways include photofermentation, dark fermentation, and biophotolysis, among others.

#### 1. Photosynthetic Microorganisms

Certain photosynthetic bacteria, such as cyanobacteria and green sulfur bacteria, are capable of producing hydrogen through light-driven reactions. These organisms use solar energy to split water molecules, releasing oxygen and hydrogen in a process known as biophotolysis [4]. Cyanobacteria, for instance, possess both photosystem I and II, enabling them to perform oxygenic photosynthesis. Under specific stress conditions (such as nitrogen limitation), they redirect electron flow to hydrogenases, which facilitate hydrogen production. Despite the theoretical potential, practical implementation is challenged by low hydrogen yields and oxygen sensitivity of the hydrogenase enzymes.

#### 2. Fermentative Bacteria

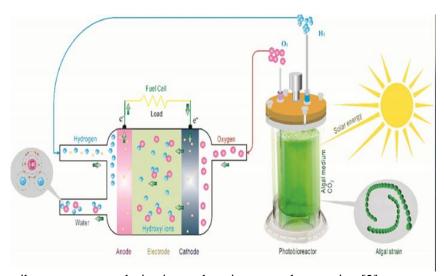
A diverse group of facultative and obligate anaerobic bacteria, including Clostridium spp., Enterobacter spp., and Escherichia coli, produce hydrogen through dark fermentation. In this process, carbohydrates such as glucose are metabolized anaerobically, resulting in the formation of organic acids (e.g., acetic and butyric acids) and hydrogen gas. Dark fermentation has the advantage of operating independently of light, allowing continuous production in controlled bioreactors. However, the process typically yields low hydrogen conversion efficiency, necessitating strategies such as co-culturing, pH control, and metabolic engineering to enhance productivity.

#### 3. Archaea and Methanogenic Microorganisms

Some archaea, particularly methanogens, can influence hydrogen availability in microbial consortia by consuming hydrogen in methanogenesis. However, certain conditions or engineered pathways can enable reverse methanogenesis, where methane is biologically converted back to hydrogen and carbon dioxide. Other extremophilic archaea, adapted to high temperatures or salinities, are being investigated for their robustness in hydrogen-producing systems, especially under industrial-scale conditions [5, 6].

#### **Bioreactor Systems and Process Optimization**

For effective hydrogen production at scale, specialized bioreactors are required to provide optimal conditions for microbial growth and metabolic activity. Key operational parameters include temperature, pH, substrate concentration, hydraulic retention time, and anaerobic conditions. Bioreactor designs such as continuous stirred tank reactors (CSTRs), upflow anaerobic sludge blanket (UASB) reactors, and photobioreactors have been developed and tested for various microbial hydrogen production systems. Furthermore, integrating waste biomass or industrial effluents as feedstock not only lowers production



costs but also contributes to waste valorization and environmental protection [2].

Pic 1. Hydrogen production from phototrophic microorganisms [7]

Pic 1. illustrates an integrated system that produces hydrogen fuel using solar energy, algae, and a fuel cell. On the right side, a photobioreactor contains a culture of algae that uses sunlight to perform photosynthesis. During this process, the algae consume carbon dioxide (CO<sub>2</sub>) and release oxygen (O<sub>2</sub>) into the system. The oxygen produced is directed to the fuel cell, shown on the left side of the diagram. Inside the fuel cell, water molecules are split at the anode to generate hydrogen ions (protons) and electrons. The hydrogen ions pass through the electrolyte to the cathode, while the electrons travel through an external circuit, creating an electrical current that can be used to power a load. At the cathode, oxygen from the

algae reacts with the hydrogen ions and electrons to produce water, completing the cycle. Meanwhile, hydrogen gas (H<sub>2</sub>) is collected as a clean and renewable fuel. This combined system effectively converts solar energy into usable hydrogen fuel by coupling biological photosynthesis with electrochemical energy conversion, offering a sustainable approach to hydrogen production [3].

Biological hydrogen production offers several significant advantages:

It relies on renewable resources (e.g., organic waste, sunlight).

- The processes occur at ambient temperatures and pressures, reducing energy input.
- It contributes to carbon-neutral energy cycles.
- It enables coupling with waste treatment, improving sustainability.

However, there are critical limitations to overcome:

- Low hydrogen yields compared to conventional methods.
- Oxygen sensitivity of key enzymes (particularly hydrogenases).
- Challenges in hydrogen gas collection, purification, and storage.
- Scale-up difficulties due to complex microbial interactions and bioreactor dynamics. Future Perspectives and Research Directions

To enhance the commercial feasibility of microbial hydrogen production, several research directions are being actively pursued. Metabolic engineering and synthetic biology offer tools to modify microbial pathways for higher hydrogen yields. For instance, knocking out competing pathways and overexpressing key enzymes can improve metabolic flux towards hydrogen. Moreover, genetically engineered strains with oxygen-tolerant hydrogenases could enable more robust systems. Omics technologies (genomics, proteomics, metabolomics) are also used to understand and manipulate microbial communities for optimized performance.

Integrating microbial hydrogen production with renewable energy systems (e.g., coupling photobioreactors with solar panels) and circular bioeconomy models (e.g., using agricultural waste) could lead to a scalable and economically viable hydrogen economy. International collaboration, government support, and interdisciplinary research will be critical to transforming this promising biotechnological solution into an industrial reality [1].

#### Conclusion

The utilization of microorganisms for hydrogen production represents a promising pathway toward clean and renewable energy. Although biological hydrogen production is still in the developmental phase, advances in microbiology, bioengineering, and process optimization are steadily addressing current limitations. As the global community strives for decarbonization and sustainable energy solutions, microbial hydrogen production could play a pivotal role in the future energy landscape.

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#### Hidrogen istehsalinda mikroorqanizmlərin istifadə edilməsi

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## Azərbaycan Memarlıq və İnşaat Universiteti

Davamlı və təmiz enerji mənbələrinə olan təcili ehtiyac, mikroorqanizmlər vasitəsilə bioloji hidrogen istehsalına əhəmiyyətli tədqiqat diqqətini yönəltdi. Bu məqalə müxtəlif mikroorqanizmlərin, o cümlədən fotosintetik bakteriyalar, fermentativ bakteriyalar və arxeyaların anaerob və ya işıqla idarə olunan şəraitdə hidrogen əmələ gətirdiyi əsas mexanizmləri nəzərdən keçirir. Hidrogenazlar və nitrogenazlar kimi fermentlərin rolu ilə yanaşı, biofotoliz, fotofermentasiya və qaranlıq fermentasiya kimi əsas metabolik yolları vurğulayır. Bu mikroorqanizmlərin optimallaşdırılmış bioreaktor sistemləri daxilində inteqrasiyası hidrogen məhsuldarlığını və prosesin miqyasını artırmaq üçün kritik amil kimi müzakirə edilir. Bioloji hidrogen istehsalı aşağı enerji girişi və bərpa olunan substratların istifadəsi kimi ekoloji üstünlüklər təklif etsə də, aşağı çevrilmə səmərəliliyi, ferment oksigen həssaslığı və qaz toplanması kimi problemlər qalmaqdadır. Metabolik mühəndislik, sintetik biologiya və bioproseslərin optimallaşdırılması sahəsində irəliləyişlər cari məhdudiyyətləri aradan qaldırmaq və mikrob hidrogen texnologiyalarının kommersiya imkanlarını asanlaşdırmaq üçün perspektivli strategiyalar kimi müəyyən edilir. Bu icmal davamlı hidrogen iqtisadiyyatına keçiddə əsas komponent kimi mikrob hidrogen istehsalının potensialını vurğulayır.

Açar sözlər: Biohidrogen, fotosintetik bakteriyalar, biofotoliz, bərpa olunan enerji, sintetik biologiya, hidrogen, hidrogenaza fermenti

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## INNOVATIVE HYDROGEN GAS PRODUCTION METHODS IN INDUSTRIAL APPLICATIONS

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Hydrogen gas is widely regarded as one of the most promising alternative energy carriers, with potential applications spanning from clean energy production to industrial processes. The production of hydrogen, once primarily based on fossil fuels, is rapidly evolving with the integration of renewable resources and innovative technologies. This article reviews the latest advancements in hydrogen gas production methods, exploring their relevance in industrial applications, and discusses the potential for a sustainable hydrogen economy.

Key word: Biohydrogen, photosynthetic bacteria, biophotolysis, renewable energy, synthetic biology, hydrogen, hydrogenase enzyme

#### Introduction

Hydrogen gas, with its high energy density and zero carbon emissions at the point of use, is considered a cornerstone for future clean energy solutions. Its versatile applications include use as a fuel for transportation, electricity generation, and industrial processes such as ammonia synthesis and petroleum refining. However, the production of hydrogen has traditionally been dependent on processes such as steam methane reforming (SMR) and coal gasification, both of which emit significant greenhouse gases. To address climate concerns, industries are focusing on cleaner and more sustainable methods of hydrogen production, aiming to achieve the goal of "green hydrogen."

This article delves into the latest innovative hydrogen production methods and their practical applications in industrial settings.

Electrolysis: Water to Hydrogen

Electrolysis is considered one of the most promising green hydrogen production methods, as it involves splitting water (H<sub>2</sub>O) into hydrogen (H<sub>2</sub>) and oxygen (O<sub>2</sub>) using electricity [2, 4]. When powered by renewable energy sources such as wind or solar, electrolysis produces hydrogen with no carbon emissions, often referred to as "green hydrogen." There are several types of electrolysis methods, including:

**Alkaline Electrolysis**: The most mature technology, using an alkaline solution (usually potassium hydroxide) to conduct electricity and produce hydrogen.

**Proton Exchange Membrane (PEM) Electrolysis**: More efficient and compact than alkaline electrolysis, PEM electrolyzers are ideal for applications requiring rapid response times and high purity hydrogen.

Anion Exchange Membrane (AEM) Electrolysis: A newer technology that promises to combine the advantages of both alkaline and PEM electrolysis, such as cost-effectiveness and high efficiency [6].

Despite its promise, electrolysis faces challenges such as high capital costs, particularly for PEM electrolyzers, and energy consumption. However, with advancements in electrolyzer efficiency and reductions in renewable energy costs, electrolysis is expected to become more competitive in industrial applications.

Thermochemical Water Splitting

Thermochemical processes involve using heat to drive the splitting of water into hydrogen and oxygen. These methods typically require high temperatures (above 1000°C) and are often coupled with concentrated solar energy or nuclear reactors. One of the most well-researched thermochemical cycles is the Sulfur-Iodine Cycle, which uses a series of chemical reactions to produce hydrogen efficiently.

The primary advantage of thermochemical water splitting is its potential to use concentrated solar energy, which can be a game-changer for hydrogen production in sunny regions. However, the technology is still in the early stages of development, and scaling it up for industrial use presents technical and cost challenges [5].

## **Biological Hydrogen Production**

Biological hydrogen production leverages microorganisms such as algae or bacteria that produce hydrogen as part of their metabolic processes. Dark fermentation and photofermentation are two biological processes being explored for hydrogen generation:

**Dark Fermentation**: This process uses anaerobic bacteria to break down organic matter, producing hydrogen as a by-product. It is considered a potential low-cost method for producing hydrogen from waste materials, such as agricultural waste or food scraps.

**Photo-fermentation**: Certain algae and bacteria can produce hydrogen using sunlight through a photosynthetic process. Although this method shows promise in terms of sustainability and low cost, it is currently limited by the slow rate of hydrogen production.

While still in the research phase, biological hydrogen production could provide a renewable and cost-effective method to produce hydrogen, especially when combined with waste-to-energy systems.

Biomass Gasification

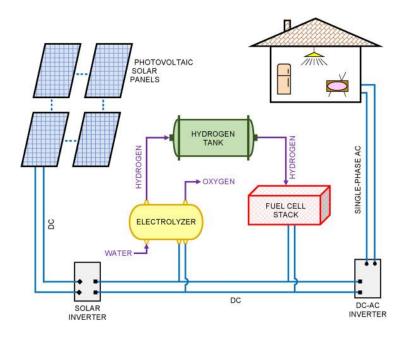
Biomass gasification is a thermochemical process that converts organic material (biomass) into hydrogen, carbon monoxide, and carbon dioxide. The hydrogen is extracted through subsequent processes such as water-gas shift reaction. This method offers the advantage of utilizing biomass feedstocks, which are often waste materials, such as wood chips, agricultural residues, and even municipal solid waste.

The key advantage of biomass gasification is its ability to use renewable feedstocks, making it a potentially sustainable source of hydrogen. However, the process requires complex technology and is not as efficient as direct electrolysis. Additionally, the use of biomass for hydrogen production must be carefully managed to avoid competition with food production or deforestation.

Integrated Solar-Hydrogen Systems

One of the most promising innovative methods is the integration of solar energy with hydrogen production. In regions with abundant sunlight, concentrated solar power (CSP) systems can be coupled with electrolysis or thermochemical cycles to produce hydrogen directly from solar energy. These integrated systems can achieve high efficiencies, especially in desert regions with a high solar irradiation index [1, 3].

Research in this area is focused on improving the efficiency and scalability of CSP systems and optimizing the integration with hydrogen production units. The development of hybrid systems that combine multiple renewable sources could be the future of sustainable hydrogen production, particularly in industrial applications that require large-scale hydrogen production.



Pic 1. Integrated Solar-Hydrogen Systems

Challenges and Future Directions

While these innovative methods of hydrogen production hold great promise, several challenges need to be addressed before they can be widely adopted in industrial applications:

**Cost**: Most green hydrogen production methods, such as electrolysis, remain expensive compared to traditional fossil-fuel-based methods.

**Scalability**: Many of these technologies are still in the research or pilot stages, and scaling them to meet industrial demand will require significant investment in infrastructure and technology development.

**Energy** Efficiency: The energy consumption of hydrogen production methods, especially thermochemical processes, can be quite high. Researchers are focusing on improving energy efficiency to make these methods more competitive.

The future of hydrogen production lies in continuous innovation and investment in new technologies. As renewable energy sources become more affordable and efficient, and as governments and industries push for a sustainable energy transition, hydrogen gas production could play a critical role in decarbonizing industries such as steel manufacturing, transportation, and energy storage.

#### Conclusion

Hydrogen gas production is undergoing a transformation as industries and researchers explore innovative methods that minimize environmental impact. Electrolysis, thermochemical processes, biological methods, and biomass gasification all offer pathways to sustainable hydrogen production. While challenges remain, the integration of these technologies into industrial applications could help pave the way for a clean hydrogen economy [2, 4].

The future of hydrogen production is bright, and with continued advancements, it will likely become a cornerstone of global efforts to reduce carbon emissions and transition to a sustainable, low-carbon future.

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## SƏNAYE TƏTBİQLƏRİNDƏ İNNOVATİV HİDROGEN QAZININ İSTEHSAL ÜSULLARI

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Hidrogen qazı təmiz enerji istehsalından sənaye proseslərinə qədər potensial tətbiqləri ilə geniş şəkildə ən perspektivli alternativ enerji daşıyıcılarından biri kimi qəbul edilir. Bir vaxtlar ilk növbədə qalıq yanacaqlara əsaslanan hidrogen istehsalı bərpa olunan mənbələr və innovativ texnologiyaların inteqrasiyası ilə sürətlə inkişaf edir. Bu məqalə hidrogen qazı hasilatı üsullarında ən son nailiyyətləri nəzərdən keçirir, onların sənaye tətbiqlərində aktuallığını araşdırır və davamlı hidrogen iqtisadiyyatı potensialını müzakirə edir.

Açar sözlər: Biohidrogen, fotosintetik bakteriyalar, biofotoliz, bərpa olunan enerji, sintetik biologiya, hidrogen, hidrogenaza fermenti

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