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HYDRODYNAMIC PROPERTIES OF COMPLEX SOLUTIONS OF GLYCYRRHIZIN ACID WITH MONOAMMONIUM SALT OF MONOIZOPROPYLIDEN LAGOXILINE

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Annotation: *This article provides information on obtaining a water-soluble supramolecular complex compound of monoisopropylidene lagoxiline (MIPL) and glycyrrhizic acid (GK) and studying the nature of the forces affecting the viscosity of aqueous solutions and the structuring of aqueous solutions of the complex compound.*

Key words: *Monoisopropylidene lagoxilin, lagoxilin, monoammonium salt of glycyrrhizic acid, hydrophobic, hydrophilic, thermodynamic*

INTRODUCTION.

Today, a lot of research is being carried out on the world scale to extract medicinal substances from plants, to determine their chemical structure and biological activity, and to put them into practice. Natural compounds isolated from plants have high biological activity, and terpenoids and their derivatives occupy a special place among such compounds. Plants of the Lagochilus species have great practical value for their medicinal properties. They have been included among the effective hemostatic medicinal plants in the East since ancient times, and have been used as an effective medicine in various bleeding processes. In this article, we mainly discuss the nature of forces affecting the viscosity of aqueous solutions of monoizopropyliden lagoxylin (MIPL) and glycyrrhizic acid (GKMAT) supramolecular complex compounds, and the structuring of aqueous solutions of complex compounds due to "hydrophobic-hydrophobic" interactions and hydrogen bonds. Synthesis and preparation of glycyrrhizinic acid (GKMAT) as well as monoizopropyliden lagoxylin (MIPL) and glycyrrhizinic acid (GKMAT) supramolecular complex compounds, given in the literature, aregiven brief information.

THEORETICAL PART

When determining the viscosity of aqueous solutions of cupramolecular complexes, it was carried out on the Ubellode viscometer, which allows them to be diluted directly in the viscometer. To determine the relative viscosity, solvent (t_0) and

0.2%; 0.1%; 0.05%; The flow time (t_i) of solutions at a concentration of 0.025% was measured. The calculation was made according to the following formula (1):

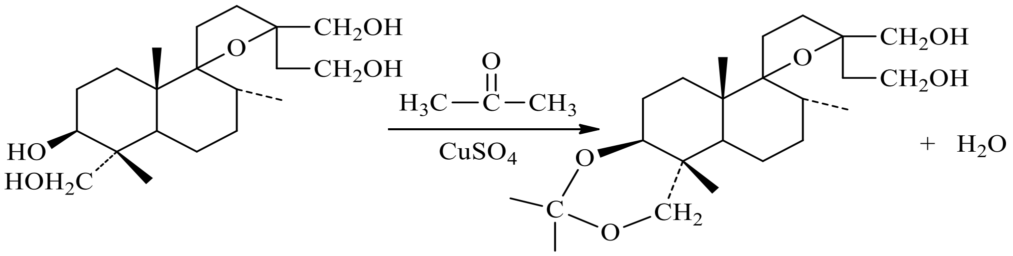
$$\eta_{relative} = \frac{t_i}{t_0}; \quad \eta_{comparison} = \frac{t_i - t_0}{t_0}; \quad \eta_{quoted} = \frac{\eta_{comparison}}{C}$$

Formula 1. The formula for the concentration dependence of the given viscosity $(\eta_{сол}/C)$ *of an aqueous solution of the complex.*

In order to isolate the diterpenoid Lagohilus inebrians Bge from the plant Lagohilus inebrians Bge, the plant was crushed and sprayed with a 20% solution of alkali (sodium hydroxide), and after drying, it was extracted in a dichloroethane solvent. After the dichloroethane solution was filtered and concentrated, it was cooled in a refrigerator and crystals of the technical lagochilin diterpenoid were isolated.

To obtain lagoxiline from monoizopropylidene on the basis of lagoxiline, lagoxiline was dissolved in acetone, anhydrous copper sulfate and concentrated sulfuric acid were added dropwise, and it was brought to rN=3-4. The mixture was placed in a separatory funnel and extracted with cyclohexane 4 times in 100 ml. The cyclohexane solutions were combined and washed with 5% sodium bicarbonate solution. It was neutralized and the solvent was distilled off, and the residue was washed with ether-benzene 15:1 on a silica gel column. recrystallized from ether, filtered, washed with absolute ether, and dried to give pure monoizopropyliden lagoxyline.

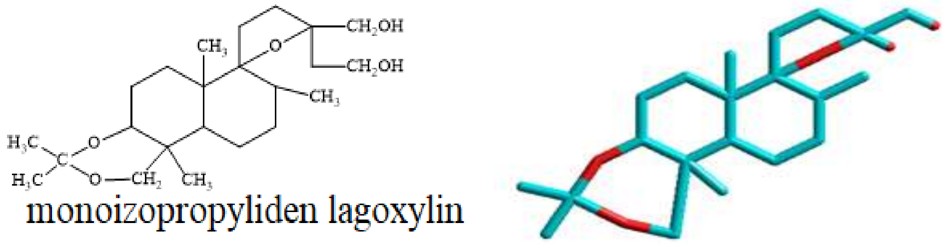
The synthesis of 3,18-O-monoizopropylidene lagoxyline when lagoxiline reacts with acetone in the presence of anhydrous copper sulfate is presented in scheme 2 [1-2]:



Lagoxiline 3,18-O-monoizopropylidenelagoxiline

Scheme 2.Scheme of the synthesis of 3,18-O-monoizopropylidenelagoxyline

Monoizopropyladen lagoxyline, chemical structure formula is shown in Figure 1



monoizopropyliden lagoxylin

Figure 1. Chemical structure of monoisopropylen lagoxiline

The IR spectrum of monoizopropyliden lagoxylin is presented in Figure 2

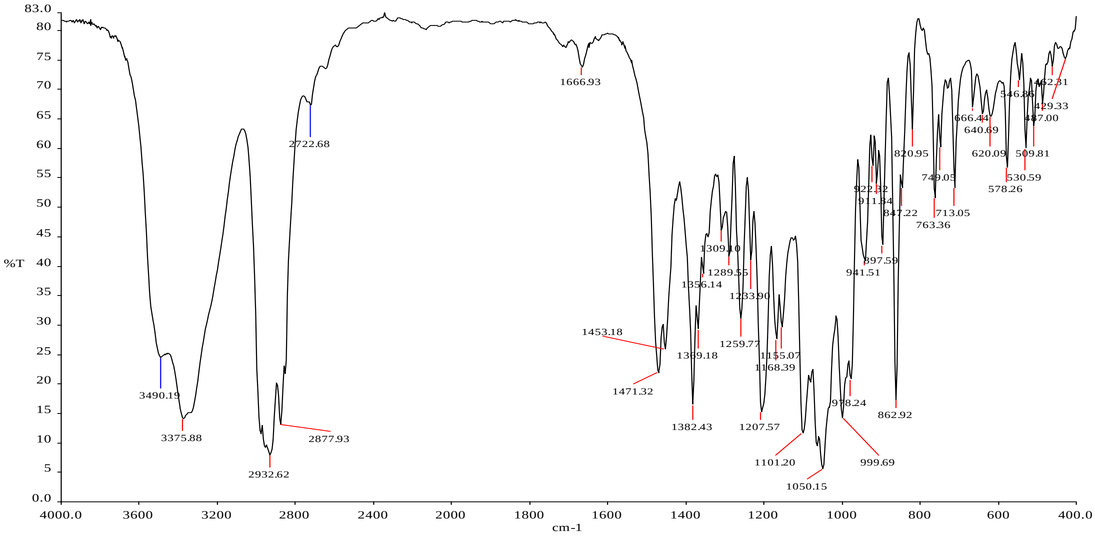


Figure 2. IR spectrum of monoizopropyliden lagoxylin

In the IR-spectrum of monoizopropylidene lagoxiline, the valence vibration frequencies of the OH groups in the molecule were observed in the intensive state at 3490, 3375 cm^{-1}, the valence vibration frequencies of the CH_3, CH_2 groups appeared at 2932, 2878 cm^{-1}, CH_3 at 1471, 1453, 1382, 1369 cm^{-1} , CH_2, CH groups, the symmetric valence vibrations of the epoxide ring at 1207 cm^{-1}, the frequencies characteristic of their asymmetric vibrations at 941 cm^{-1}, and the deformation vibrations of this ring at 863 cm$_{-1}$ were observed. The frequencies of valence vibrations of C-O-C, C-OH bonds in the molecule at 1101-1050 cm^{-1} observed in the intense state are presented in Table 2.7 [1-2]:

Some physico-chemical parameters of Lagoxilin diterpennoide and Monoizopropylidenelagoxilins were studied (Table 1).

Table 1

Some physico-chemical parameters of Lagoxilin diterpennoide and Monoizopropylidenelagoxilins

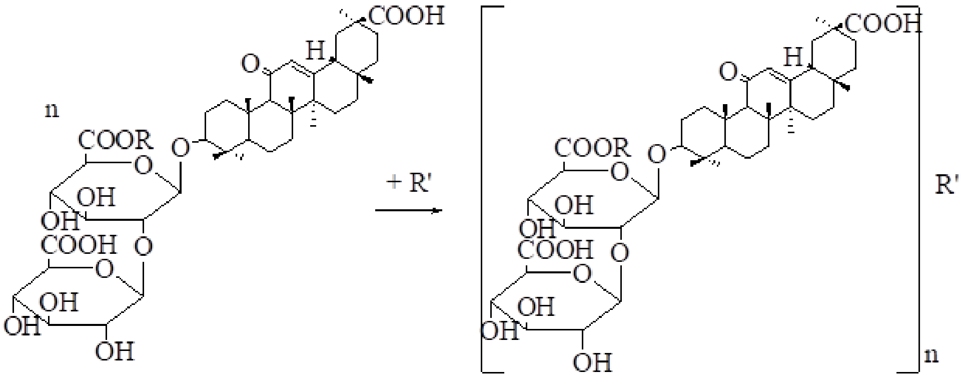
№	Substances	Gross formula and Mol.wei.	T.$_{liquid.}$ ^{0}C	R$_f^*$ (system)	[α] $_D$ 0,5% water:ethanol 1:1	IR spectrum см$^{-1}$
1.	LG White crystal	$C_{20}H_{36}O_5$ 356	167-168	0,15 (I)	-	1053(-O-); 2938,(CH_3) 3336,(OH)
3	MIPl White crystal	$C_{23}H_{40}O_5$ 396	150-151	0,72 (**II**)	+ 12	1666(-O-);2932,(CH_3) 3490,(OH)

It is already known in the literature that monoizopropyliden lagoxylin is not well soluble in water, therefore its biological activity, especially hemostatic activity, is limited. GKMAT was isolated from the root of Glycyrrhiza glabra (liquorice) plant based on the method previously known in the literature to obtain the water-soluble supramolecular complex compound of Lagoxilin and some physico-chemical parameters were studied.

In order to obtain supramolecular complexes of monoizopropyliden lagoxyline with GKMAT, GKMAT was dissolved in 50% acetone, monoizopropylidene lagoxyline was added under stirring, and the reaction mixture was stirred at room temperature for 10-12 hours. Acetone was removed and the aqueous fraction was lyophilized. Preparation of the GKMAT supramolecular complex with tetraacetyl lagoxylin is presented in Scheme 4.

Scheme 4

The scheme for obtaining the supramolecular complex of monoizopropyliden lagoxylin with GKMAT



where: R=H; NH_4; К. n=2; 4; 9.

R^1=MIPl,

A water-soluble supramolecular complex of monoizopropyliden lagoxylin with GKMAT was obtained. In the study of the structure of supramolecular compounds, physical methods based on the interaction of organic molecules with electromagnetic light, in particular, in the IR spectrum (vibration of atoms in the molecule ($\lambda=10^{-4}-10^{-2}$ см) spectra) are widely used [3-6].

Information about the intermolecular interactions between organic compounds and which functional groups are involved in these interactions can be obtained based on the comparison of the IR-spectra of the starting substances and the final products. Based on this, the process of evaluating the formation of a supramolecular complex with Monoizopropyliden lagoxylin with GKMAT was carried out on the basis of the IR-spectroscopy method and other physicochemical analyzes.

Supramolecular complex compounds of 1:2, 1:4, 1:9 molecular ratios of GK and GKMAT with monoizopropyliden lagoxylin were obtained. Supramolecular complex compounds of 1:2, 1:4, 1:9 molecular ratios of GK obtained with monoizopropyliden lagoxyline are white amorphous powders, well soluble in water. Supramolecular complex compounds of GKMAT obtained with monoizopropyliden lagoxylin in molecular ratios of 1:2, 1:4, 1:9 are pale yellow amorphous powders, well soluble in water. The angle of deflection of the plane of the polarized light beam $[\alpha]_D$ was determined. They are optically active compounds, substances that bend the plane of polarized light. The obtained supramolecular complex compounds were characterized by some physicochemical and spectral parameters, which are listed in Table 2. [3-6].

Table 2

Some physicochemical parameters of supramolecular complexes of GK, GKMAT and GKMKT salt with monoizopropylidenlagoxylin

3	Substances fat	T. $_{liquid}$.C^0 divorce.	R_f^*	α]$_D$ 0,5 % EtOH(50 %)	IR , см$^{-1}$	ffect
1	ГК:МИПЛ: 2:1 (2076)	199-201	0,12 (I) 0.94 (IV)	60	1043,,(COC);1726 (CO) 2936,(CH$_3$);3407 (OH)	4,4
2	ГК:МИПЛ: 4:1 (3756)	203-205	0,11 (II) 0.94 (IV)	52	1043,(COC);1632 (CO) 2946, (CH$_3$);3406 (OH)	0,0
3	ГК:МИПЛ 9:1 (7956)	207-209	0.10 (II) 0.93(IV)*	54	1043, (COC);1734 (CO) 2948, (CH$_3$);3405 (OH)	8,9
4	ГКМАТ: МИПЛ 2:1 (2189)	181-184	0,58 (VI), 0,40 (I), 0,10 (II)	20	1042, (COC);1659 (CO) 2941, (CH$_3$);3405 (OH)	0,9
5	ГКМАТ: МИПЛ 4:1 (3972)	186-188	0,55 (VI) 0,37 (I)	16	1042, (COC);1632 (CO) 2945, (CH$_3$);3422 (OH)	6,1
6	ГКМАТ: МИПЛ 9:1 (8442)	190-192	0.36 (I) 0.1 (II)	18	1042, (COC);1630 (CO) 2947 (CH$_3$);3418 (OH)	4
7	ГКМКТ: МИПЛ 2:1 (2152)	201-203	0.81(V)	22	1043, (COC);1658 (CO) 2945, (CH$_3$);3367 (OH)	0,6
8	ГКМКТ: МИПЛ 4:1 (3908)	205-207	0.80(V)	24	1043, (COC);1660 (CO) 2948, (CH$_3$);3368 (OH)	2,6
9	ГКМКТ: МИПЛ 9:1 (8298)	210-212	0.79(V)	22	1659, (COC);2947 (CO) 3368 (CH$_3$);3366 (OH)	3,8

I. Ethyl acetate, **II.** Ethyl acetate-acetone 5:1, **III.** Chloroform-acetone 9:1, **IV.** Chloroform-acetone 5:1, **V.** - Ethyl acetate- ethanol 6:2, **VI.** Chloroform-ethanol 5:1, **VII**. Butanol-Acetic acid-Water 3:1:1, **VIII**. Ether-Hexane 4:1 **IX**. Benzene:acetone 5:1.

In the IR spectrum of supramolecular complex compounds of GKMAT obtained with monoizopropyliden lagoxylin in molecular ratios of 1:2, 1:4, 1:9, valence vibrations of the carbonyl group of GK and acetyl lagoxylins in the region between 1740-1725 cm^{-1} and hydrogen bonds in the region between 3400-3250 cm^{-1} it was observed that the valence vibrations of the hydroxyl groups involved in the formation are manifested in the form of a broad shoulder, and the deformation vibrations of the methyl groups are manifested in the region between 2935-2925 cm^{-1} The IR spectrum of the supramolecular complex compound of MIPL:GKMKT in a ratio of 1:2 is presented in Fig. 3.

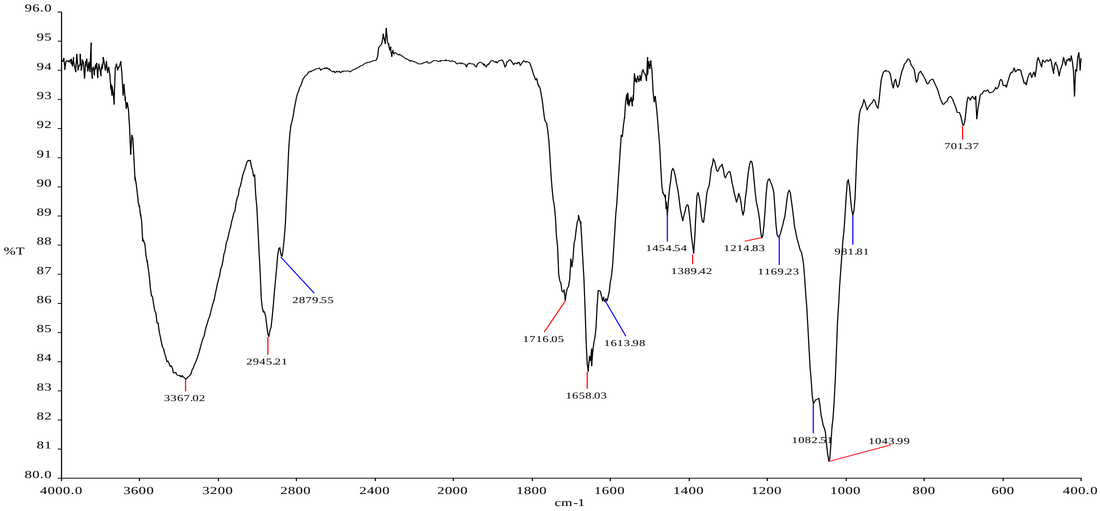


Figure 3. IR spectrum of the supramolecular complex of GKMKT with monoizopropyliden lagoxyline in a ratio of 1:2

Figure 3 Physical methods based on the interaction of organic molecules with electromagnetic light in the study of the structure of all supramolecular complex compounds obtained, in particular, their IR- (vibrational spectrum of atoms in the molecule, $\lambda=10^{-4}-10^{-2}$ cm) spectrum and UV- (outer step) Electron spectrum based on changes in the energy state of electrons, $l=10^{-6}-10^{-4}$ cm) spectra were widely used. With the help of these methods, it is possible to draw conclusions about new interactions and bonds based on the differences in the spectrum of the starting substances and supramolecular complexes. In the analysis of the structure of supramolecular complexes, their UV and IR spectrum data were used.

In the UV spectrum, the intense absorption maximum value corresponding to the π-π^* transition of C=O conjugated with the double bond in the C ring of GK and its salts is observed at a wavelength of 250-251 nm in the near UV region in the water:ethanol (1:1) system. Since monoizopropylidene lagoxyline does not have a chromophore group, it does not show absorption in the UV spectral region. [3-6.].(Fig. 4).

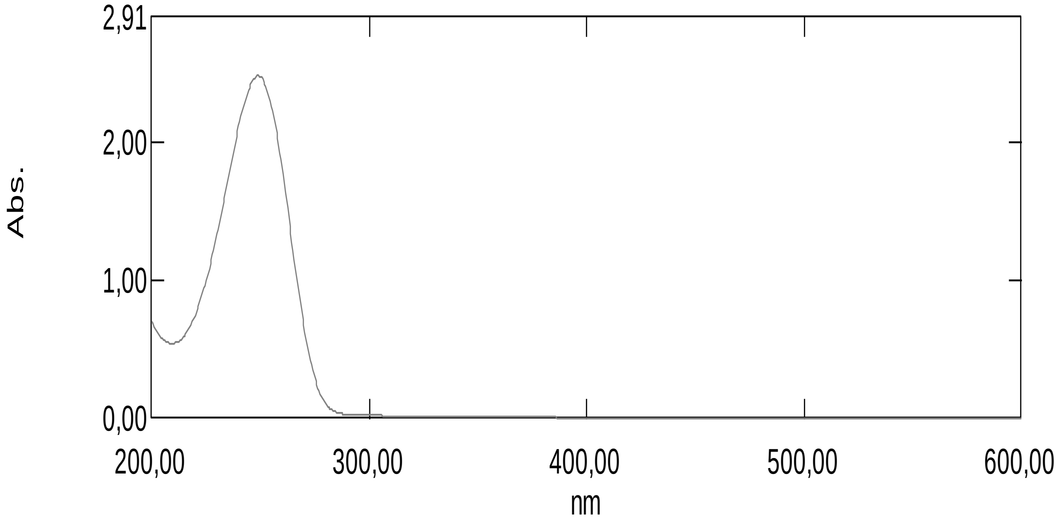


Figure 4. UV spectrum of the supramolecular complex of GK with monoizopropyliden lagoxyline in a ratio of 1:2

In the IR spectrum of compounds synthesized with monoizopropyliden lagoxylin based on GK and its salts, in the field of vibrations of functional groups, the valence vibrations of the carbonyl group of the carboxyl groups, which do not participate in the formation of hydrogen bonds of GK, show 1734-1726 cm^{-1}. The valence vibrations of the carbonyl group in the carboxyl groups involved in the formation of hydrogen bonds of GK and its mono salts are observed in the range of 1660-1632 cm^{-1}. It is observed that the vibrational frequencies of the carbonyl groups in the starting materials shift to a lower frequency range of 72-67 cm^{-1}, which in turn indicates their participation in the formation of hydrogen bonds. In the high-frequency range of the IR spectrum, it is observed that the valence vibrations of the hydroxyl groups in positions 15, 16 of monoizopropyliden lagoxylin appear in the relatively low frequency range of 3420-3410 cm^{-1}. It is observed that the melting point of synthesized complex compounds also goes away with decomposition. Furthermore, complex compounds were characterized by thin layer chromatography, such as $/\alpha/_D$.[3-6].

Based on the change of the basic vibrational frequencies of the functional groups in the IR spectra of the initial substances, it is possible to speculate about the presence of interactions between molecules in the formation of molecular complexes. In particular, the valence vibration frequencies of the OH group in the GKMKT molecule were shown at 3390 cm^{-1}, and in the complex at 3367 cm^{-1}. The difference in valence vibration frequencies of OH groups by 23 cm^{-1} indicates the formation of hydrogen bonds in the complex. In addition, the fact that this area has a broad shoulder indicates the presence of ion-dipole interactions between molecules. In the 1039 cm^{-1} bands, the valence vibration frequencies of the C-O-C and C-OH bonds in the GKMKT molecule were observed in an intensive state, and the vibration frequencies of these bonds in the complex were observed in the 1082, 1044 cm^{-1} bands, due to the formation of hydrogen bonds between these groups during the formation of the complex. indicates. In addition, the valence vibration frequency of the epoxy ring in the monoizopropylidene lagoxyline complex was observed in the region of 1214 cm^{-1} [3-6].

When comparing the spectra of the initial substances (MIPL, GKMAT) and the spectra of complex compounds (MIPL-GKMAT), it is possible to observe a number of changes that occurred, and if we assume that it occurs due to "hydrophobic-hydrophobic" interaction and hydrogen bonds, it indicates the formation of a supramolecular complex compound [3-6].

In the study of the structure of complex substances, including supramolecular compounds, physical methods based on the interaction of organic molecules with electromagnetic light are widely used, in particular, their UV- (electron spectrum based on the change in the energy state of electrons in the outer level, $\lambda=10^{-6}$ – 10 $^{-4}$ см) [3-6].

In the UV-spectrum, the intense absorption maximum value corresponding to the π-π^* transition of π-electrons of C =O conjugated with the double bond in the C ring of GKMKT was observed in the near-UV range of 250-253 nm in the water:ethanol (1:1) system. It is shown in Figure 5 below.

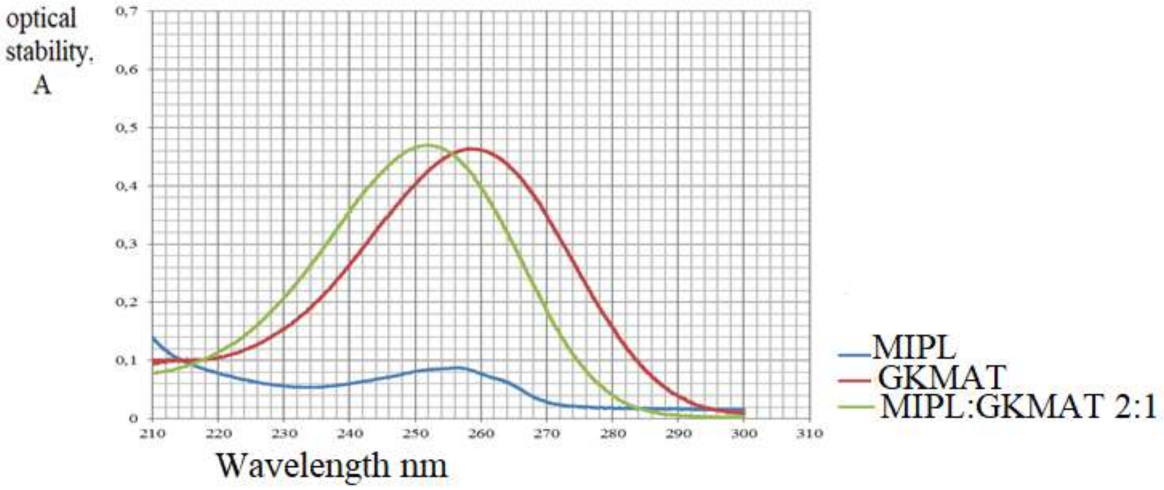


Figure 5. Supramolecular complex (MIPL-GKMKT) combination of MIPL with GKMKT in ratio 2:1 UV-spectrum Cm=510^{-5}mol/l, 50% etonol solvent

Discussion of the obtained results.

The resulting supramolecular complex compound is a pale yellow, amorphous powder. 0.1% aqueous solutions of GKMAT complex compounds have the property of forming a gel. [3-6].

Studying the nature of the micellar structures formed in the GKMAT solution and the forces that ensure the stability of the micelles in this process allows us to know the effects of which the complexes are formed. It is known that the structure of micelles in SFM solution is influenced by factors such as solvent and system temperature, presence of electrolyte, nature of SFM and its concentration. As a result of the influence of these factors, micelles of different shapes can be formed in the solution, and the most thermodynamically stable form prevails among them under selected conditions. In the literature, it is shown that the micelle formation critical concentration (MCC) of GKMAT solution starts from 0.2% ($2.125 \cdot 10^{-3}$ mol/l) [3-6].

When determining the viscosity of aqueous solutions of cupramolecular complexes, it was carried out on the Ubellode viscometer, which allows them to be diluted directly in the viscometer. To determine the relative viscosity, solvent (**t0**) and 0.2% - 0.1%; 0.05%; The flow time (t_i) of solutions at a concentration of 0.025% was measured. The calculation was made according to the following formula (1):

$$\eta_{\text{relative}} = \frac{t_i}{t_0} \;; \quad \eta_{\text{comparison}} = \frac{t_i - t_0}{t_0} \;; \quad \eta_{\text{quoted}} = \frac{\eta_{\text{comparison}}}{C}$$

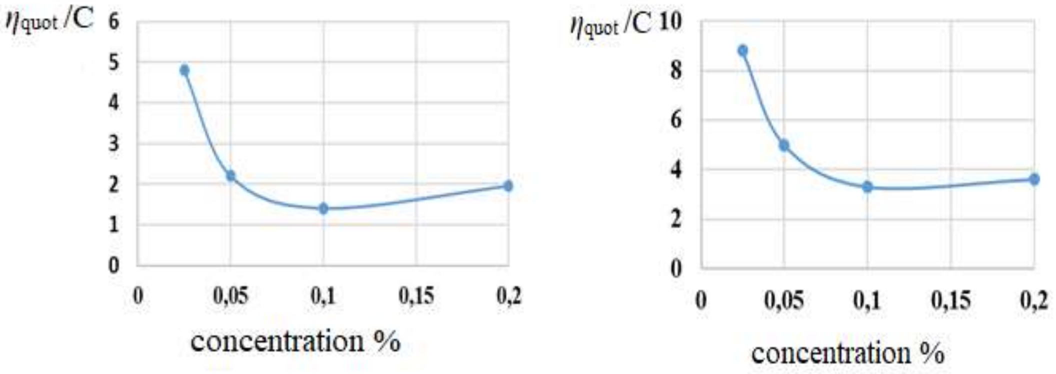
Formula 1. The formula for the concentration dependence of the given viscosity ($\eta_{\text{сол}}/C$) of an aqueous solution of the complex.

In order to find out the nature of the micellar structures formed in the solution of the GKMAT:MIPL (2:1) complex and the nature of their stabilizing forces, the effect of various agents on the viscosity of its solutions: intermolecular hydrogen bond breakers (urea), hydrophobic interaction shielding (xylose) and electrolyte (KCl) agents was studied .

During the studies, when the viscosity of the aqueous solution of the complex ($\eta_{\text{сол}}/C$) was studied as a function of concentration, a break was observed in the curve representing this relationship at the point where the concentration of the complex was equal to 0.2%, and since this situation indicates the beginning of a structural change in the solution at this concentration, the hydrodynamic properties of complex solutions studied at concentrations of 0.2% and less.

Molecular complexes of GKMAT with acetyl derivatives ("guest" compounds) of GKMAT differ from each other in terms of hydrophilic-hydrophobic balance and, of course, have different effects on the hydrodynamic properties of GKMAT solution.

As mentioned above, in order to study the effect of the intermolecular hydrogen bond breaking agent on the hydrodynamic properties of the GKMAT:MIPL (2:1) complex, the process was carried out in a 1 M aqueous solution of urea. According to the results of viscometric measurements (Fig. 7, a), the stability of the rearrangement of these complexes can be due not only to hydrogen bonds, but also to other mutual hydrophobic and hydrophilic effects and electrostatic attraction forces.



a b

Figure 7. Curves of concentration dependence of viscosities of aqueous solutions of GKMAT:MIPL (2:1) complex in 0.1 M urea (a) and 0.01 M xylose (b) media

Under the influence of urea, it is expected that the GC clusters in the form of Xn (n=1..6) formed at the expense of cooperative hydrogen bonds and the viscosity values of supramolecular complexes will decrease. However, this decrease was evident only in the complex concentration range of 0.1–0.2%. When the concentration is less than 0.1%, the viscosity of the solution increases dramatically. This allows us to conclude that the supramolecular complex manifests itself as a polyelectrolyte. In fact, only one COOH group in GKMAT has a salt form and the other two carboxyl groups are protonated. As the degree of dilution of the solution increases, the degree of dissociation of the remaining carboxyl groups increases. As a result, the dissociated carboxyl groups begin to push each other under the influence of the "neighbor" effect, as a result of which the supramolecular complex begins to reorganize and move from a compact form to a spread form. This, in turn, leads to an increase in the viscosity of the solution.

Compounds containing a carboxyl (COOH) group are weak electrolytes, and therefore their aqueous solutions obey Ostwald's dilution law ($К_{diss} = \frac{\alpha^2}{1-\alpha}С_{М}$), α<<1 when $К_{diss} = \alpha^2 С_{М}$).) The dissociation of glycyrrhizic acid can be represented schematically as (1):

$GK - (COOH)_3 \leftrightarrow GK - (COO^-)_3 + 3H^+$

$$K_{dis} = \frac{[GК{-}(COO^-)_3][H^+]^3}{[GК{-}(COOH)_3]} \qquad (1)$$

Schematic representation of GK dissociation

As mentioned above, one carboxyl group in GKMAT is completely dissociated as it is in salt form. Then the process can be expressed in the following (2.) form:

$GKMAT - (COOH)_2 \leftrightarrow GK - (COO^-)_2 + 2H^+$

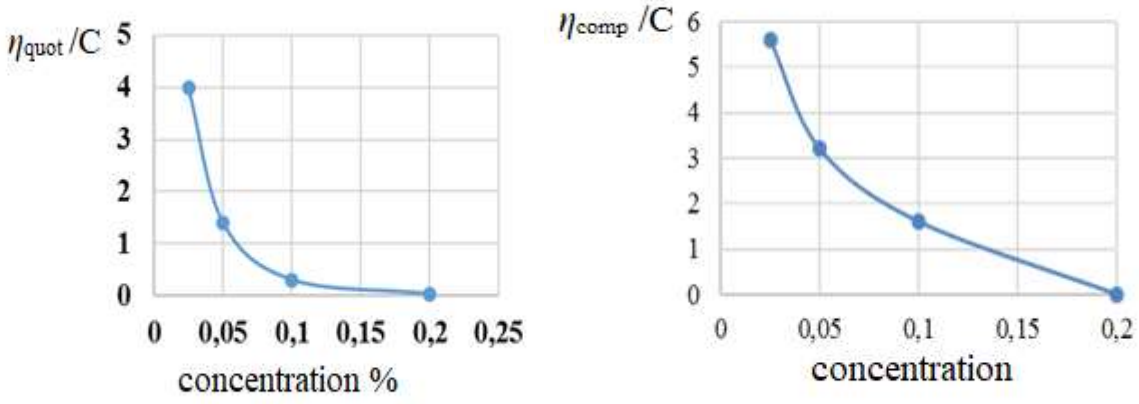
$$К_{dis} = \frac{[GК{-}(COO^-)_2][H^+]^2}{[GК{-}(COOH)_2]} \qquad (2).$$

Schematic representation of the dissociation of GKMAT

As the concentration of the complex in the solution decreases, the degree of dissociation (α) increases.

The same situation can be observed in the presence of an agent affecting the hydrophobic effect, that is, when conducting viscometric measurements in the medium of 0.01 M of xylose, the curve of the system viscosity as a function of concentration was similar to that in the presence of urea (Fig. 7, b). This also indicates that the hydrodynamic properties of the supramolecular complex are more affected by the conformational changes caused by the electrostatic repulsion of the deprotonated carboxyl groups. After the concentration exceeds 0.1%, the supramolecular complex behaves like non-electrolyte polymers, that is, the viscosity of the solution begins to be directly proportional to the concentration.

When studying the effect of ionic strength on the stability of the resulting supramolecular complex, an interesting phenomenon was observed. Generally, the hydrodynamic properties of polyelectrolytes change dramatically when the ionic strength of the system increases and they behave like non-electrolyte polymers.



a b

Figure 8. Graphs of concentration dependence of viscosities of GKMAT:MIPL complex aqueous solutions in 0.1n KCl (a) and 0.1n NaOH (pH=10.3; b) media.

This is because the ions added to the system block the functional groups of the ionogenic polymer, i.e. "screen" and weaken the "adjacent group" effect, and the conformational change is prevented. As a result, the viscosity of the system changes in direct proportion to the concentration. But the studied supramolecular complex is a strong electrolyte (KCl) (Fig. 8.a). behaving like a "polyelectrolyte" in its environment is explained by the robustness of the reorganization formed in the GKMAT and MIPL system.

The fact that the concentration dependence of the viscosity of supramolecular complex solutions of GKMAT:MIPL in 0.1 n NaOH medium decreases sharply with increasing concentration further strengthens our hypothesis about the effect of the above electrostatic forces (Fig. 8b). In an alkaline medium, carboxyl groups are neutralized and become completely deprotonated (– COONa).

When the hydrodynamic properties of aqueous solutions of GKMAT GKMAT:MIPL supramolecular complex are compared (Fig. 9.a), we can see that the relative viscosity of the complex solution is lower than that of GKMAT. This is explained by the formation of "host-guest" structures with a complex compact volume.

When the viscosity of a 0.1% aqueous solution of GKMAT-MIPL 2:1 was investigated as a function of temperature, it was observed that the viscosity of the complex reached a maximum when the temperature increased to 30^0C, and then decreased again (Fig. 9.b). It can be assumed that there are effects other than the usual effects in the formation of this complex, which are formed in a certain temperature range and then decrease again.

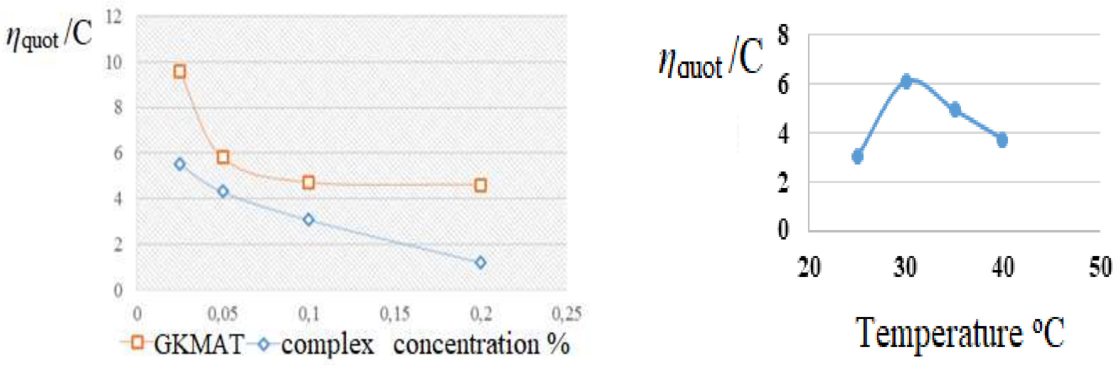


Figure 9. Graph of dependence of viscosity of 0.1% GKMAT-MIPL 2:1 aqueous solution on concentration (a) and temperature (b)

This can be likened to the "orientational effect" between molecules. In addition, the degree of dissociation of weak electrolytes also increases with increasing temperature. It can be assumed that due to the increased dissociation of carboxyl groups under the influence of heat, the fluidity decreases and the viscosity increases. When the temperature is higher than 300C, the viscosity decreases due to weakening of the "orientation effect" forces. In conclusion, it can be said that when the substance MIPL forms a supramolecular complex with GKMAT, its hydrophobicity decreases and its solubility in water increases.

CONCLUSIONS

1.GKMAT supramolecular complex compounds with monoisopropylidene lagoxiline were synthesized, the structure, physicochemical properties of the complexes were studied based on IR- and UV-spectroscopy methods.

2.The nature of the forces affecting the viscosity of aqueous solutions of GKMAT supramolecular complex compounds with monoisopropylidene lagoxiline and the structuring of aqueous solutions of complex compounds is explained by "hydrophobic-hydrophobic" interactions and hydrogen bonds.

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