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# Section 1. Biology

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## REPRODUCTIVE SIGNIFICANCE AND SURVIVAL OF DIFFERENT COHORTS IN THE MUSKRAT (ONDATRA ZIBETHICUS L.) POPULATION IN THE SOUTH ARAL REGION

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**Abstract**

The article discusses the issues of studying the survival and reproductive value of the muskrat population in the conditions of the Southern Aral Sea region. The specific survival rates of males and females remain generally similar to the pattern of changes in survival rates calculated for the generation as a whole.

New indicators of muskrat population parameters have been obtained, characterizing the demographic characteristics of different cohorts.

**Keywords:** *the Aral region, survival rate, reproductive processes, cohorts, environmental factors, muskrat population*

### Introduction

One of the pressing problems of modern ecology remains the study of the organization and dynamics of communities of terrestrial vertebrates. In the recent past, the lower reaches of the Amu Darya were the main base for muskrat farming in Uzbekistan; almost 90–95% of the skins were harvested here (Reimov R., 1985; Reimov R., Nuratdinov T., Shiryaev V., 1989).

All over the world, the problems of sustainability and stability of biological systems are being positively addressed. The decline in biodiversity, its full existence, undoubtedly leads to profound consequences for the evolution of species. A decrease in population dynamics, an increase in their habitability, and the struggle for surviving during the formation of their diversity lead to the fact that natural ecosystems from rodent communities are traditionally widely used as standard objects. In zoological and ecological studies, problems of anthropogenic changes in landscapes, among other things, affect the population composition and population dynamics of representatives of small fauna (Mambetullaeva S. M. 1994; Reimov R., Nuratdinov T., Shiryaev V., 1989).

Muskrat (Ondatra Zibethica) is one of the species from the order of rodents (Rodentia).

At home in North America, in Karakalpakstan, the muskrat has successfully acclimatized. Distributed from the lower reaches of the Amu Darya River to the Aral Sea. It was brought to the Amu Darya delta in 1944 (355 heads) from the Altai Territory (Mambetullaeva S. M. 1994; Reimov R., Nuratdinov T., Shiryaev V., 1989).

It was brought to Karakalpakstan to enrich the fauna and currently lives along the banks of streams and canals, where it digs holes for housing where there is an access to the water. The muskrat's dense and beautiful fur does not get wet and allows it to swim even in winter. On lakes with gently sloping shores, the muskrat builds permanent huts of semi-aquatic plants mixed with silt. A solid house frozen in winter with access to water is inaccessible to any predator (Moiseev V. A., Ametov M. B., 1986).

## Material and methods

The material for the research was data from commercial muskrat samples (n = 34 individuals, including 16 males) in the period from 2021 to 2023. in the Dautkul, Zhaltyrbas and Karateren reservoirs, where the unique population of the species were small for 20 years due to the drying out of many reservoirs due to regulation of the flow of the Amu Darya River. Samples were collected annually from mid-September to mid-October (Moiseev V. A., Ametov M. B., 1986). Each trap line consisted of 100 traps, set annually along the same 2 km route. The method is based on the simultaneous usage of information on the number and age of animals in commercial samples taken over several years. Summarizing tables make it possible to reduce the influence of capture selectivity on the sampling structure, since the muskrat, like a number of other rodent species, is predominantly caught in adult individuals (Olenev G. V., Grigorkina E. B., 2014).

The method allows us to obtain an estimation of the absolute number and structure of the population of animals that originally lived in a given territory and were captured not only in the first year of their life, but also over several consecutive years. To do this, to the number of animals caught in the year of capture, the number of animals of 2 and 3 years of age, caught in the next three years, but belonging to a certain cohort by birth date, is added.

The method makes it possible to estimate the total number of animals that lived in the study area, but were not caught in the first year of capturing, and to significantly clarify the population structure (Muskrat. Morphology, sistematics, ecology. 1993).

The term "cohort" was used in its usual understanding as a set of individuals born in one short period of time (Olenev G. V., Grigorkina E. B., 2014). In other words, the term "cohort" can be defined as "a set of individuals born during a period of mass emergence of broods." During the breeding season, three such aggregates have been identified in the muskrat (Larin B. A., 1966). In the river delta Amu Darya, the first wave of intensive reproduction occurs from the last ten days of April – up to May inclusive (the first cohort); the second wave is June-July (second cohort), the third wave is August-September (third cohort). The term "generation" was the totality of individuals born in the current breeding season (the sum of cohorts of a given year of birth).

## Results and discussion

The dynamics of seasonal changes in the size of the muskrat's genital organs shows that in the conditions of the reservoirs of the Southern Aral Sea region, muskrats do not breed all year round.

Activation of reproductive processes begins at the end of winter, with spring warming and partial release of ice from water bodies, i.e. The muskrat is one of the animals that reproduces only during the warm season. In September, the animal's sexual activity dies down. The active breeding period is April-August. In the conditions of the lower reaches of the Amu Darya, muskrat reproduction lasts 6–6.5 months. The size of the broods can be judged by the average number of embryos per female (Reimov R., 1985; Reimov R., Nuratdinov T., Shiryaev V., 1989). The litter size also varies slightly over the years – 7.1–7.8. In the Amu Darya delta, on average there are 9.8 embryos in the first litter, 8.8 in the second, and 7.7 in the third. The minimum litter size is observed in young females of the first litter of the current year –5.5 (Berestennikov D. S., 1979; Bulakhov V. L., Kurennaya M. I., 1976).

In November-December, the muskrat intensively prepares for wintering; the animals are active during the day, building new dwellings and restoring abandoned huts. In winter, when water bodies are covered with ice, muskrats are more active during the day. In the spring, with the first emergence of the muskrat from under the ice, signs of the spring year are observed. At this time, the activity of the muskrat generally increases, pairing and dispersal begins. In summer, the muskrat leads a more active lifestyle than in winter (Reimov R., 1985; Reimov R., Nuratdinov T., Shiryaev V., 1989). However, the decisive factor that determines the intensity of reproduction and population dynamics is the state of reservoirs – water availability throughout the year, the availability of food and places for the construction of dwellings (Komarov A.V., 1990). The natural conditions of the Amu Darya delta have changed dramatically, anthropogenic desertification is occurring, numerous lakes and swampy habitats are drying up, and degradation of reed and cattail thickets is observed.

The noticeable predominance of males in the spring is explained by their activity during the breeding season. The sex ratio in newborns of the early litter is almost equal (49.0% females and 51.0% males) (Ivanter E.V., 1975).

In the Amu Darya delta, due to collector, drainage and waste waters, small lake systems (Lake Akchakul, etc.) and some small reservoirs, the total usable area of which is about 20 thousand hectares, are most regularly supplied with water.

In the life of a muskrat during the year, several stages can be distinguished that are most important for characterizing the reproductive process of the species: rutting and settlement; creation of family settlements and protection of sites; autumn migrations and wintering (Reimov R., 1985; Reimov R., Nuratdinov T., Shiryaev V., 1989). The coefficient of variability in the proportion of females among age groups ranges from 60% to 89% (Bulakhov V.L., Kurennaya M.I. 1976; Bolshakov V.N., Danilov N.N., 1979).

Currently, much attention is paid to assessing the role of various factors in shaping the dynamics of animal populations. It is of particular interest to conduct these assessments on animals living in two environments (aquatic and aquatic). The assessment of the main factors of the population dynamics of Ondatra Zibethica was carried out in two periods: at the beginning and at the end of the breeding season. The highest coefficient of variation falls on the share of young animals (1–8 months). The coefficient of variation among breeding females was also highest for the age of 1–8 months.

Most species of small mammals are characterized by high fertility and short life expectancy (a little more than a year), which depending on the influence of various factors, determines changes in population numbers. Rodents can bear up to three or more litters per breeding season. However, high mortality means that a small part of the population survives the winter. More successful survival of small mammals is facilitated by the mechanism of delayed growth and maturation in the third cohort, which increases its life expectancy. Different authors call this mechanism alternative developmental pathways or different types of ontogenesis (Tsvetkova A.A. 2010; Muskrat. Morphology, sistematics, ecology. 1993).

The maximum specific survival rate is typical for the age interval $0^{+}$ $1^{+}$. During the period from the starting point of capturing until the following autumn, 43.5% of muskrats survive.

In the next age interval, the specific survival rate sharply decreases; only 26% of one-year-old animals survive to age $2^{+}$. An even sharper decrease in specific survival rate is observed in the last age interval – only 4.2% of two-year-old animals survive to age $3^{+}$ (Melnikov Yu. I., Dunaev V.V., 1990; Larin B.A., 1966).

The specific survival rates of males and females remain generally similar to the pattern of changes in survival rates calculated for the generation as a whole. Females are characterized by a slightly higher specific survival rate in all age classes. This leads to a slight predominance of females in the adult ($1^{++}$) part of the population. The higher survival rate of females may be associated with selective (increased) catching of males due to the characteristics of their behavior – guarding the area, going out first to feed, etc. The highest specific survival rate in the first year of life (0.62) was found in the first cohort, the lowest – the second (0.36), the most numerous (Table 1). The survival rate of the third cohort (0.44) took an intermediate position.

**Table 1.** *Specific survival rate (px) of muskrats belonging to different cohorts*

| Age class | 1 cohorts | | 2 cohorts | | 3 cohorts | |
|---|---|---|---|---|---|---|
| | ***Sx*** | ***px*** | ***Sx*** | ***px*** | ***Sx*** | ***px*** |
| 0+ | 12 | 0.62 | 8 | 0.36 | 9 | 0.44 |
| 1+ | 16 | 0.07 | 11 | 0.21 | 8 | 0.42 |
| 2+ | – | – | 5 | 0.22 | 8 | 0.24 |
| 3+ | – | – | 0.s3 | – | 1 | – |

***Note:*** *Sx – number of age classes $1^+$; $2^+$; $3^+$; px – survival rate per unit of time, in this case – per year*

The following year, the specific survival rate of animals belonging to the first cohort decreases, and in the age interval $1^+ – 2^+$ it is the lowest. Only about 9% of $1^+$ animals survive to age $2^+$, or about 6% of the original autumn cohort size. In the second cohort, 22% of animals $1^+$ survive to age $2^+$, or about 8% of the initial autumn number. The specific survival rate of the third cohort is quite high both in the first year of life and in the second. Almost half of the animals $1^+$ or 21.8% of the initial autumn number survive to age $2^+$ in this cohort. Such a sharp change in specific survival rates in different cohorts as age increases lead to a significant redistribution of the share of different cohorts in the generation as it ages (Table 2).

**Table 2.** *Ratio of cohort sizes as generation age increases,%*

| Age class | Cohort size,% of the total generation size | | |
|---|---|---|---|
| | **1 cohort** | **2 cohort** | **3 cohort** |
| $0^+$ | 19.5 | 43.0 | 37.5 |
| $1^+$ | 28.4 | 36.6 | 35 |
| $2^+$ | 8.5 | 26.7 | 64.8 |
| $3^+$ | 0 | 20.8 | 70.2 |

The large reproductive contribution of females of the third cohort to the total number of descendants of the generation is ensured by their higher survival rate. The high specific survival rate of the third cohort in the first year of life can be partly explained by the relatively weak fishing pressure on it (Bulakhov V. L., Kurennaya M. I. 1976; Bolshakov V. N., Danilov N. N., 1979). It is much more difficult to find an explanation for the high specific survival rate of representatives of the third cohort in adulthood, when these muskrats practically do not differ in size from representatives of the other cohorts. A priori, it can be assumed that at least the commercial component of mortality in these animals should have been the same as in representatives of other cohorts (Bolshakov V. N., Danilov N. N., 1979; Komarov A. V. 1990; Reimov R., 1985).

Thus, as a result of the undertaken research, new, previously unknown, values of muskrat population parameters were obtained, characterizing the demographic characteristics of different cohorts. It was revealed that there are no statistical differences in the fertility of animals of different cohorts. Among the three cohorts formed in a generation, the maximum average specific survival rate is typical for representatives of the third cohort. This leads to the fact that in the second or third year of life, the share of the third cohort in the total number of the generation turns out to be the largest.

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# SUPRAMOLECULAR COMPLEXES OF LAGOHYRZINE WITH MONOAMMONIUM SALT OF GLYCYRRHIZIC ACID AND THEIR BIOLOGICAL ACTIVITY

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## Abstract

This article presents the synthesis of supramolecular complexes of lagohyrzine in various molar ratios with the monoammonium salt glycyrrhizic acid. Their IR spectra are discussed, as well as some physico-chemical properties and hemostatic activity of the obtained complexes.

**Keywords:** *Lagochilus, diterpenoids, 9,13-epoxylabdan, lagohirzine, lagodene, hemostasis monoammonium salt of glycyrrhizic acid*

## Introduction

Plants of the genus Lagochilus have been known for their medicinal effect for a long time and are among the most famous medicinal plants of the East, as hemostatic agents are valuable. Lagochilus-based drugs are successfully used to stop various bleeding, but they have some disadvantages: insolubility in water and oral administration, as a result of which the necessary effect develops slowly (Flora SSSR. M.,– L.: AN SSSR. 1954; Abramov M. M., Yaparova S. A., 1963). To eliminate these disadvantages, a number of acetyl and isopropylidene derivatives of lagochiline have been obtained. It has been shown that the hemostatic activity depends in a certain way on the number of free hydroxyl groups of lagochilin and its derivatives (Zaynutdinov U. N., Islamov R., Dalimov D. N., Abduraxmanov T. R., Matchanov A. D., Vipova N. L., 2002). One of these lagohilin derivatives is the diterpenoid lactone lagohirzine, on the basis of which the hemostatic drug Lagoden was created. Lagohirzine in its free form is found in three species of plants of the genus Lagochilus (L. hard-haired, L. bristly and L. gypsum). But its content in these plants is 0.2–0.3% and therefore it was synthesized on the basis of (Zaynutdinov U. N., Islamov R., Dalimov D. N., Abduraxmanov T. R., Matchanov A. D., Vipova N. L., 2002).

Lagohilin in the presence of anhydrous copper sulfate with acetone forms 3.18-O-isopropylidenlagohilin, the latter, upon oxidation with potassium permanganate, turns into isopropylidenlagohyrzine, which after acid hydrolysis forms lagohyrzine. The reduction of lagohyrzine with lithium alumohydride leads to the formation of tetraol, corresponding to lagohilin in physico-chemical parameters. Consequently, lagohirzine contains the skeleton of lagohilin in its molecule. To increase the effectiveness of the hemostatic action of lagochilin itself (A.S.1293990 SSSR. Sposob polucheniya lagoxirzina. Islamov R., Zaynutdinov U. N., Aslanov X. A., Sadikov A. S., Danilchuk D. N., Yankovskiy B. A., Zaxarov V. P.) and its derivatives, their molecular complexes with water-soluble polymers are obtained. In addition to polymers, a number of low molecular weight compounds have complexing properties. For example, gossypol forms a number of molecular complexes with molecules of organic solvents (Dalimov D. N., Zaynutdinov U. N., Musaev U. N., Matchanov A. D. Muxamadiev M. G., Yuldashev X. A., 2001; Ibragimov B. T., Nazarov G. B., Talipov S. A., 1988; Ibragimov B. T., Talipov S. A., Zory P. M., 1994). But a more effective complexing agent is 18β-H-glycyrrhizic acid (GA), a triterpene glycoside isolated from licorice root (Ibragimov B. T., Talipov S. A., 1996).

In connection with the above, it was interesting for us to obtain supramolecular complexes with the monoammonium salt of glycyrrhizic acid (MASGA) lagohyrzine in various ratios. The choice of lagohirzine molecules as objects of research is due to the fact that lagohirzine is an intermediate product of the preparation of Lagodene and it is poorly soluble in water, and therefore has relatively weak hemostatic activity.

The drug "Lagoden" has a good hemostatic property. It has been shown that Lagoden surpasses all known drugs of this purpose in its hemostatic activity. However, the method for producing Lagodene is multi-stage and requires expensive solvents. In the end, the yield of the product is very low, relative to lagohilin is 12–14% (Zaynutdinov U. N., Islamov R., Dalimov D. N., Abduraxmanov T. R., Matchanov A. D., Vipova N. L., 2002). Therefore, the creation of new hemostatic drugs based on local, affordable plant raw materials at lower cost is relevant.

**The purpose of this scientific work** is the modification of lagohilin and the diterpenoid lactone lagohirzine by molecular association and the production of water-soluble derivatives with MASGA, as well as the study of their hemostatic activity.

**Research objectives:** – isolation and purification of the diterpenoid lagohilin and synthesis of the diterpenoid lactone lagohirzine based on it.– isolation and purification of GA from the technical product of licorice root.– preparation of supramolecular complexes based on the diterpenoid lactone lagohyrzine and preparation of water-soluble derivatives with MASGA in various molar ratios.– study of the physico-chemical properties of the obtained supramolecular complexes.

## Materials and methods:

The identification of the obtained supramolecular complexes was carried out in several solvent systems using thin-layer chromatography on Silufole. All complex compounds dissolve well in water.

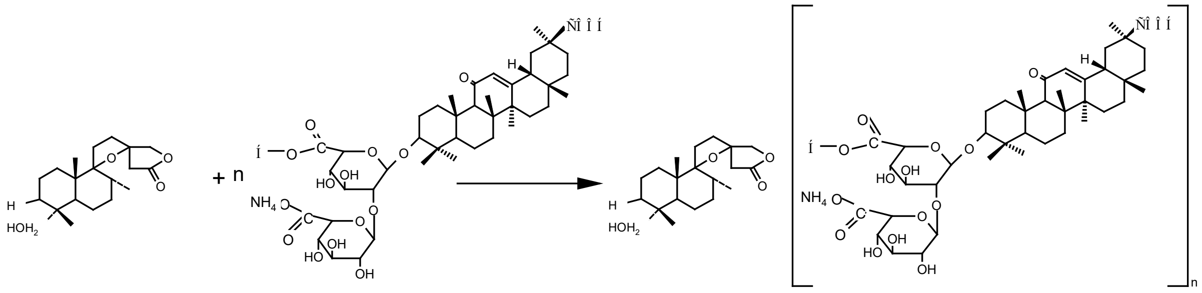
Therefore, the study of: "Supramolecular complexes of GA with drugs", and according to the analyzed phenomena – the mechanisms of micello and gelation of GA, the establishment of the basic physico-chemical properties of hydrogels of low molecular weight natural compounds in various aggregate states and the causes of the high biological activity of supramolecular complexes based on the monoammonium salt of GA with lagohilin is an important stage of this work.

## Results and discussion

The synthesis of lagohyrzine (LHZ) was carried out using a previously known technique (Zaynutdinov U. N., Islamov R., Dalimov D. N., Abduraxmanov T. R., Matchanov A. D., Vipova N. L., 2002). Molecular complexes obtained on lagohirzine with MASGA in various molar ratios of 1:1; 2:1 and 4:1. Purified MASGA (94–96%) was used for the synthesis of complexes.

The study of the solubility of the complexes showed that all the obtained complexes are highly soluble in water and insoluble in organic solvents, which significantly increases their bioavailability for the human body, which consists of 75–80% of water.

**Scheme 1.** *A general scheme for the preparation of molecular complexes of lagohilin and lagohirzine with GA and MASGA*



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To characterize the approximate structures of the obtained molecular complexes, comparative studies of the IR spectra of lagohirzine and MASGA with the spectra of their molecular complexes were carried out. (table 1).

According to the literature data, during complexation, the frequency of valence vibrations of carbonyl groups shifts to a high-frequency region relative to the initial one (Dalimov D. N., Isaev Yu. T., Sayitkulov A. M., 2001).

**Table 1.** *Some physico-chemical parameters of the molecular complexes of Lagohilin and lagohirzine with GA and MASGA*

| Complexes | Ratios | $R_f$ | | | IR spectrum, sm$^{-1}$ | Melting point, °C |
|---|---|---|---|---|---|---|
| | | I | II | III | | |
| MASGA | – | 0.10 | 0.06 | 0.04 | 3433, 1733, 1643 | 242–245 |
| LHZ | – | 0.48 | 0.43 | 0.38 | 3000–3600, 1783 | 141–142 |
| MASGA: LHZ | 1:1 | 0.15 | 0.54 | 0.92 | 3010–3580, 1795, 3415, 1745 | 182–184 |
| MASGA: LHZ | 2:1 | 0.42 | 0.65 | 0.86 | 3011–3580, 1797, 3415, 1745 | 190–192 |
| MASGA: LHZ | 4:1 | 0.85 | 0.94 | 0.82 | 3010–3580, 1798, 3415, 1745 | 198–200 |

*System: I – ethanol: chloroform (1:3), II – methanol: chloroform (1:3), III – methanol: acetone (2:1).*

IR spectra of the MASGA complexes: LHZ (1:1), (2:1) and (4:1) contain the following characteristic frequencies: in the region of 1733 sm$^{-1}$ refers to the valence oscillation of the carbonyl of the lactone ring; in the region of 1700 sm$^{-1}$, the valence oscillation of carbonyls of the carboxyl groups of the monoammonium salt of glycyrrhizic acid is manifested, and the frequency of 1640 sm$^{-1}$ refers to the valence oscillation of the carbonyl located next to the double bond.

It should be noted that in the IR spectrum of the GA complex: LHZ absorption frequencies in the region of 1650–1740 sm$^{-1}$ differ in values and shape from those in the IR spectrum of the MASGA complex: LHS. This indicates that in the formation of the MASGA complex: LHZ involves the carboxyl groups of the glucuronic part of the monoammonium salt of glycyrrhizic acid, hydroxyl groups and the carbonyl group of lagohyrzine.

Thus, based on the data of IR spectra, it can be concluded that lagohirzine and MASGA form molecular complexes due to intermolecular hydrogen bonds between the carboxyl groups of MASGA and the hydroxyl groups of lagohirzine, a hydrogen bond is formed between the carbonyl of the lactone ring with the functional groups of the monoammonium salt of glycyrrhizic acid. Further, their gmostatic activity was studied in comparison with the complexes of Lagohirzine and lagohilin with GA and MASGA.

### Study of the hemostatic activity of molecular complexes

A comparative assessment of the hemostatic effect of molecular complexes was investigated: GA: L (4:1) (RGK-1), GA: LHZ (2:1) (RGK-2), MASGA: L (4:1) (RGTS-3), MASGA: LHZ (2:1) (RGTS-4), MASGA: LHZ (4:1) (RGTS-5) in parenchymal bleeding.

The bleeding time was determined 60 minutes after intraperitoneal administration of molecular complexes at a dose of 0.5 mg/kg on white non-linear rats of both sexes with a body weight of 160–20 g, 6 heads in each group (6 groups). The average bleeding duration time for each group of animals and the reliability of the differences between the experimental groups and the control were calculated. The obtained data were processed statistically. This test reflects the vascular-platelet mechanism of hemostasis and is determined by the number and condition of platelets (their ability to adhere and aggregate) (Baltina L.A., Kondratenko R.M., Flexter O.B., Murinov Yu.I., Zarudiy F.S., Ismagilova A.F., Tolstikov G.A., 2001; Bappadiya Roy, Saha A., Esterrani A., Nandi A.K., 2008).

As can be seen from the data shown in (Fig. 1), 60 minutes after the administration of RGK-1, the blood clotting time decreased by 56% – from 360 ± 20 seconds. up to 165 ± ± 10.0 seconds. With the introduction of RGTS-2, bleeding time was reduced by 45% (197.16 seconds), RGTS-3- by 72% (100.10 seconds), RGTS-4-by 30% (253.20 seconds) and RGTS-5- by 76% (85.6 At the same time, the greatest effect was observed with the introduction of molecular complexes of the monoammonium salt of glycyrrhizic acid with lagochilin in a ratio of 4:1 (RGTS-3) and the monoammonium salt of glycyrrhizic acid with lagochyrzine 4:1 (RGTS-5).

The difference between the molecular associations of HA with lagohilin and lagohirzine was small, only 20%. But they were 2–2.5 times inferior in hemostatic action to the associates of IASC (4:1). A similar pattern can be seen when studying the amount of blood loss. So in the control this indicator was 220 ± 20 mg, and the introduction of the RGC-1–43 ± 2 mg, RGC-2–25 ± 2 mg, RHC-3–10 ± 1 mg, rgts-4–59 ± 3 mg and RHC-5–10 ± 1 mg. At the same time, the maximum decrease in blood loss was observed with the introduction of RG-3 and RG-5 (by 90%).

Thus, in comparison of HA complexes with MASGK complexes, a uniquely higher hemostatic activity of complexes obtained on the basis of MASGK is shown. This is due to the fact that MASGC has a critical concentration of micelle formation lower than that of the GC itself. That is, in this case, there are certain differences in the diffusion of the drug from this associate into the bloodstream, so we observe differences in the hemostatic activity of these molecular complexes. If we compare the molecular associate of HA: LHZ (4:1) with the similar associate of MASGK: LHZ (4:1), it is clear that the complex with MASGK is 2 times more active than the complex of HA: LHZ. In addition, the molar ratios of the starting substances also play an important role in the manifestation of hemostatic activity, and this is clearly seen in the example of comparing the complexes of IASC: LHS in a ratio of 2:1 and 4:1. In a 2:1 ratio, the activity is very low, and in a 4:1 ratio, this activity increases 3 times. This suggests that the lagohyrzine molecule, in a 4:1 ratio, is in a more bioavailable form than in a 2:1 ratio.

### The experimental part

TLC on the plates of the brand "SILUFOL" was used for identification. Solvent systems for TLC: I- ethanol: chloroform 1:3, II- water: Acetonitrile: acetone 3:4:2, III- methanol: chloroform 1:3. Iodine vapor was used as a developer. Silica gel with a particle size of 100/160 was used for column chromatography. The IR spectra were taken on the Sistem- 2000 IR Fourier spectrometer of the Perkin-Elmer company on tablets with KBr.

**Preparation of GA:** Purified GA and MASGA (95–96%) were obtained from MASGA with a base substance content of 72–75% according to the method (Dalimov D.N., Isaev Yu.T., Sayitkulov A.M., 2001).

**Preparation of lagohirzine from lagohilin;** Lagohirzine from lagohilin was obtained using a previously known technique (Zaynutdinov U.N., Islamov R., Dalimov D.N., Abduraxmanov T.R., Matchanov A.D., Vipova N.L., 2002). Mp. =188–190 °C.

**The preparation of lagohirzine complexes with GA** (1:2) and (1:4) is carried out according to the method B.

**Preparation of lagohirzine complexes with MASGA (1:1) (G);** 0.0586 g (0.0002 mol) of lagohirzine is dissolved in 10 ml of 96% ethyl alcohol and then, 100 ml of distilled water is poured into a 500 ml conical flask equipped with a reverse refrigerator and a stirrer and 0.149 (0.0002 mol) MASGA is added with intensive stirring and when heated 80–82 °C. After complete dissolution, 90ml of 96% ethanol is added. With intensive stirring, an alcoholic solution of lagohirzine is added drop by drop. The mixture is intensively mixed for 10–12 hours at a temperature of 60–70 °C. After that, the alcohol is distilled on a rotary evaporator. The aqueous residue is dried by freeze drying. Mp. = 182–184 °C with decomposition.

**The preparation of lagohirzine complexes with MASGA** (1:2) and (1:4) is carried out according to the method of G.

### Study of hemostatic activity

Studies to determine the hemostatic effect were carried out in a thermostat with an open door at a temperature of + 30 °C. At this temperature, the animals were kept for at least an hour before the start of the experiment. The tip of the tail, about 10–12 mm long, is cut off with sharp scissors. A sheet of filter paper is brought to the stump of the tail. The paper is pre-dried in a thermostat (to a constant weight) and weighed. The blood flowing from the tail is evenly distributed on filter paper, after which it is dried and weighed again. The duration of bleeding is marked by a stopwatch from the moment the first drop of blood appears until the bleeding stops completely. The amount of blood loss is estimated by the weight of the dry residue in milligrams.

### Conclusion:

Thus, supramolecular complexes of MASGA with lagohirzine were obtained in various molar ratios, some physico-chemical and spectral characteristics were studied, and the hemostatic properties of the obtained complexes were studied in a comparative aspect and it was shown that they depend on the nature of the complexing agent and on the molar ratio of the starting substances.

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# ON THE PROBLEM OF STUDYING THE FUNCTIONAL STATUS OF THE PARAMETERS OF THE BLOOD CIRCULATION SYSTEM IN WOMEN OF REPRODUCTIVE AGE IN THE CONDITIONS OF THE REPUBLIC OF KARAKALPAGSTAN

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**Abstract**

The article discusses the issues of studying the functional status of circulatory system parameters in women of reproductive age in the conditions of Karakalpakstan. Reproductive health is one of the main indicators of the health status of the population and is an important criterion for the health of the population and the changes occurring in it. The most significant changes in hemodynamic parameters were changes in systolic and diastolic blood pressure, which reached the greatest values in the oldest age group.

**Keywords:** *Karakalpakstan, reproductive health, circulatory system, parameters, flexibility.*

## Introduction

In modern biomedical research, the need to use reliable diagnostic tests to monitor health status is of particular importance.

Currently, different methods of assessing the state of adaptation are used to determine the health status of different population groups within populations. One of the most convenient methods is to determine the parameters of the circulatory system – a simple and effective way to assess the functional state of the cardiovascular system and its adaptation (Baevskiy R. M., 2014).

In the conditions of the Republic of Karakalpakstan, the problem of adaptation of the organism to unfavorable external conditions does not lose its relevance, questions about the hormonal mechanisms of maintaining AP are still not well studied. The circulatory system plays a special role in creating a uniform exchange and communication environment of the body, ensures the process of information signal exchange, creates communication channels by synthesizing and transporting intercellular communication molecules (cytokines and autocoids – growth). factors, leukotrienes, prostaglandins) and redistribution of energy information flows (Abdirov Ch.A., Agadjanyan N. A., Severin A. E., 1993), (Abdurahmonova F. M., Abdurahmonov F. M., 2002).

Adaptive reactions give the organism the ability to adequately respond to changes in its internal and environmental conditions.

Among them, there are compensatory-adaptive reactions, the mechanisms of which are activated in cases where the violation of a certain structure or function of the body ensures its preservation due to the strengthening of other structural and functional formations of the body (Abdirov Ch.A., Agadjanyan N. A., Severin A. E., 1993).

The blood system, which performs various functions in the body, quickly responds to any deviations from the norm of homeostasis indicators and thus activates the body's adaptive-compensatory reaction mechanisms (Volozhin A. I., Subbotin Yu.K., 1998).

Women between the ages of 20 and 45 living in different regions of the Republic of Karakalpakstan (Nukus, Amudarya, Karauzyak districts) participated as research objects. A total of 98 women of childbearing age were examined. The women served were divided into 3 age groups: Group 1–20–29 years old (n=51), Group 2–30–39 years old (n=30) and Group 3–40–45 years old (n=36). Also, age groups were related to the place of residence of the examined women (Table 1).

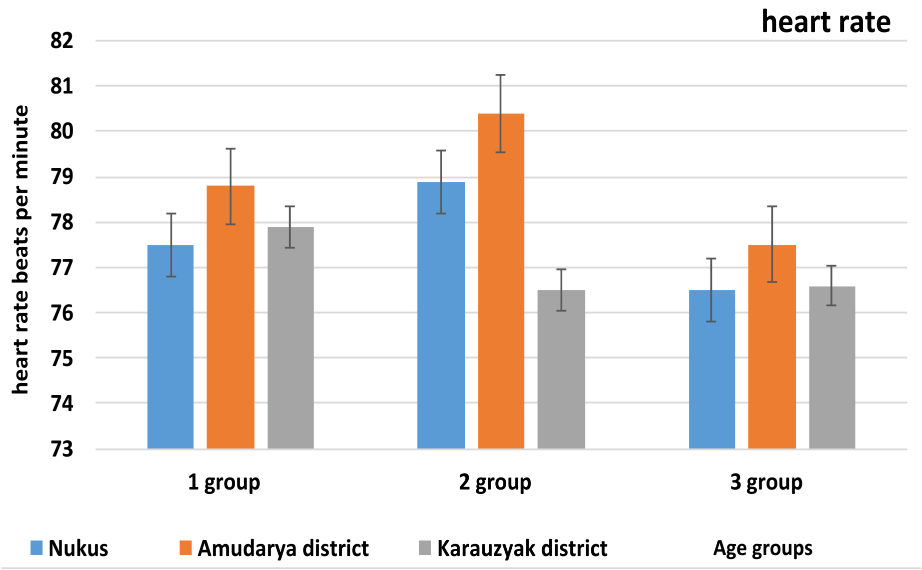
**Table 1.** *The number of women of reproductive age living in the Republic of Karakalpakstan who participated in the survey*

| Young groups | Nukus district | Amudarya district | Amudarya district |
|---|---|---|---|
| 20–29 (n young = 51) | 15 | 18 | 18 |
| 30–39 young (n = 47) | 16 | 15 | 16 |
| Contingent of 40–45–year-old reproductive age women living in the Republic of Karakalpakstan who took part in the survey (n = 53) | 20 | 18 | 15 |
| **Total: 151** | **51** | **51** | **49** |

At present, the state of the environment and living conditions have been proven to be the main factor in the formation of medical pathology with an undoubted leadership over all other factors. Studying the characteristics of the geographical distribution of diseases is the first step in the search for the connection between pathology and ecology (Abdurahmonova F. M., Abdurahmonov F. M., 2002).

Specific indicators of peripheral blood circulation in women in three compared groups are shown in the (figure. 1–3).

**Figure 1.** *Status of heart rate parameters in examined women (n=98)*



Blood circulation Reproductive health is one of the main indicators of the state of public health and is an important criterion of public health and the changes taking place in it. The reproductive system of the body is particularly sensitive to the external effects of any, even low-intensity, harmful environmental factors, including subthreshold factors, and hypothalamo-pituitary regulation is particularly sensitive to the effects of radiation (Baevskiy R. M., 2014).

The circulatory system is an extensive network of organs and vessels responsible for transporting blood, nutrients, hormones, oxygen, and other gases in and out of cells. Without a circulatory system, the body cannot resist disease or maintain a stable internal environment (Abdirov Ch.A., Agadjanyan N. A., Severin A. E., 1993), (Abdurahmonova F. M., Abdurahmonov F. M., 2002).

**Figure 2.** *Status of blood pressure parameters in examined women (n=98)*

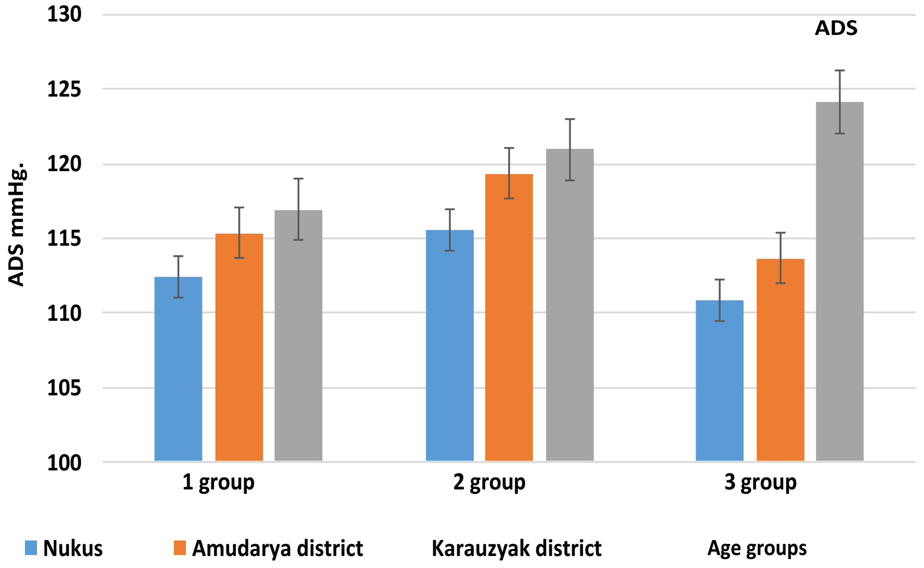
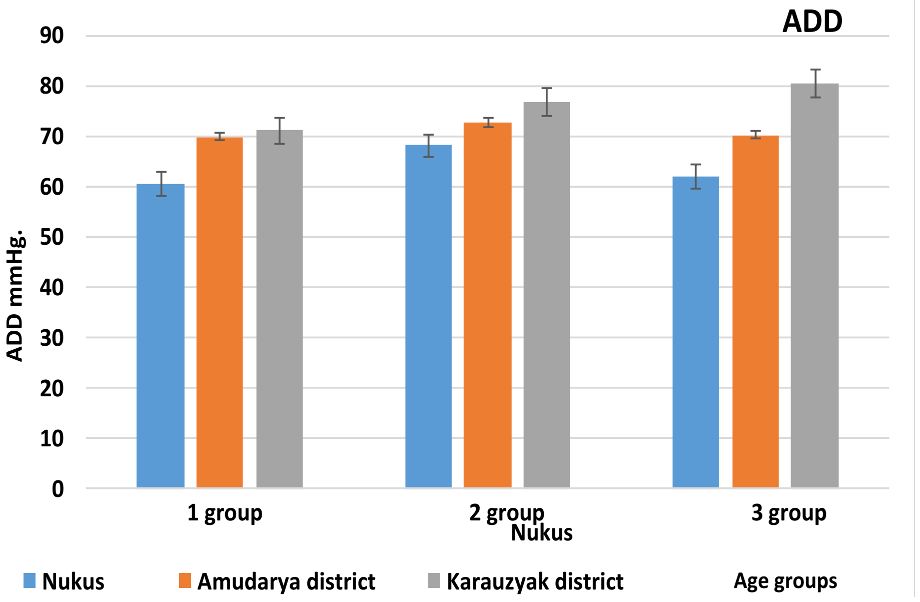


**Figure 3.** *Status of blood pressure parameters in examined women (n=98)*



It turned out that the blood pressure and heart rate of young women of the 1st group (20–29 years old) living in all three regions of the Republic of Karakalpakstan are close to each other and do not differ much from each other. Individual changes primarily concern blood pressure, which is explained by the appearance of people with arterial hypertension and vegetative-vascular dystonia. Later, a gradual increase in blood pressure began in all groups, this anomaly was less noticeable in women, as a control group.

Blood pressure reached 124 ± 4.2 mmHg in the control group and the older group (40–45 years old) in Karauzyak district. or exceeded, and in nulliparous women ADP was 116.8±2.0 mmHg. In the same group, diastolic pressure increased significantly. At this age stage, a large number of systolic and diastolic pressures were in the control group. This is another argument in favor of the importance of the social factor that increased the greatest tension in the circulatory system.

In the second age group (30–39 years old), we again observe a stable state of the chronotropic function, which turned out to be stable in other groups. The transition from the third to the fourth age group is accompanied by a statistically significant increase in systolic and diastolic pressure in women in the prosperous region. It is evident that this is the stage in which the tension and disturbance of the adaptability of the young blood circulation occurs.

The oldest age group (40–45 years) was characterized by the most significant changes in systolic and diastolic pressure. The most characteristic and at the same time multidirectional values of ADS and ADD were in women from Karauzyak region.

Thus, the most significant facts of hemodynamic parameters change are the shift of systolic and diastolic blood pressure, which reached the greatest values in the oldest age group. Hypertensive reactions prevailed in all groups, but were more pronounced in women living in Amudarya and Karauzyak regions of the Republic of Karakalpakstan.

The condition of blood circulation indicators in the examined healthy women and women with reproductive dysfunction is similar in terms of the number of functional disorders in the form of arterial hypertension and vegetative-vascular dystonia, which is a possible explanation of this social factor. Reveals the secret adaptive properties of hemodynamics. Severity of negative changes in parameters of nonspecific and antihypoxic protection, dysfunction of the reproductive field and circulatory system increases with age and depends on the place of residence.

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## Section 2. Chemistry

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# DEVELOPMENT OF IONOSELECTIVE ELECTRODES FOR RAPID DETECTION OF DRUGS BASED ON DODECOMOLIBDOPHOSPHATE IONOPHORES

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## Abstract

Ion-selective electrodes with a plasticized membrane based on dodecomolibdophosphate acid were developed for the express analysis of ori products. The main electrochemical parameters of the developed ISE: the linear range of the signal (of the electrode function), the slope of the electrode function, the lowest limit of ion detection, etc.determined.

**Keywords:** *Medicines, ionometric method, ion selective electrode, ionophore compound dodecomolybdophosphate acid, dibazol, pyridoxine*

## Introduction

In the world, much attention is paid to the control of the production and composition of medicinal substances. In this direction, certain fundamental and practical results have been achieved in Uzbekistan. In the republic, much attention is being paid to the further reform of the healthcare sector, first of all, to reducing the level of morbidity and increasing the life expectancy of the population (Decree of the President of the Republic of Uzbekistan, Decree No. PF-5590 of 07.12.2018).

In this direction, it is urgent to rapidly develop methods of monitoring the composition of pharmaceutical products and the technological processes of their production, to develop modern operational methods of analyzing the composition of products, and to create high-performance sensors that solve various problems based on them.

Ionometric method of determination of drugs is one of the most common methods. Instruments based on this method (ionomers) are available in almost every laboratory, so information about ionometric methods of determination of various medicinal substances is of particular importance.

Potentiometry based on the use of an ion-selective electrode is an alternative to time-consuming and expensive methods such as high-performance liquid chromatography, capillary electrophoresis and spectral analysis due to the simplicity and speed of the equipment in the analysis conditions (Matveychuk, Yu.V., 2016; Matveychuk, Yu.V., 2017). Therefore, one of the important tasks of analytical chemistry is to create and research new high-performance ISE based on existing electrode active substances for drug analysis. Such electroactive substances include dodecomolybdophosphate heteropolyacids (GPK) and their derivatives, which are significantly superior to natural ionophores in terms of selectivity and sensitivity (Ceresa A., Sokalski T., Pretsch E., 2001; Smirnova A. L., Khitrova V. L., Shvake A., 2000; Kharitonov S. V., 2001).

**The purpose of this work** development of membrane ion-selective electrodes based on dodecomolybdophosphoric acid (DDMF) for rapid analysis of medicinal substances and testing their indicators. In order to achieve the goal set in the work, the synthesis of soluble electrode-active compounds based on drugs with DDMF was carried out.

The technology of obtaining plasticized membranes with the resulting ionophores was developed, researches were conducted in the field of establishing the effect of the composition of the drug-detecting ISE membrane on its indicators.

## Methodology and materials

Dodecaphosphomolybdate acid and medicinal preparations as an electrode-active substance (ionophore) in work: dibazol and pyridoxinecompounds obtained on the basis of

Synthesis of ionophores was carried out at room temperature (25 °C). Aqueous solutions of relevant drugs (dibazole and pyridoxine) were added to aqueous solutions of dodecophosphomolybdate heteropolyacid (GPK) heated to $25^0$C with constant stirring. After some time, a white precipitate will settle. The precipitate was then separated from the liquid, washed with cold distilled water and dried for one day. Taking into account the tribasicity of dodecaphosphomolybdate acid and the possibility of protonation of three molecules of dibazole, the molar ratio of GPK and dibazole was taken as 1:3, respectively.

The table shows the results of the synthesis of ion associates based on DDFM and pyridoxine and dibazole drugs.

**Table 1.** *Results of synthesis of ionophores based on dodecaphosphomolybdate acid and drugs (pyridoxine, dibazole)*

| | Gross formula of EAS | Initial salt | Color of EAS | Product yield,% | Solubility of obtained EAC |
|---|---|---|---|---|---|
| Starting component – 25 ml of 0.01 M $H_3[R(Mo_3O_{10})_4]2H_2O$ solution | | | | | |
| 1 | $(Pyridoc)_3[R(Mo_3O_{10})_4]2H_2O$ | pyridHCl | Light green | 96.7 | Low solubility |
| 2 | $(Dib)_3[R(Mo_3O_{10})_4]2H_2O$ | DibHCl | Dark green | 90.5 | Low solubility |

ISE plasticized film membranes were prepared by dissolving PVCH, dioctyl phthalate plasticizer, and electrode active compound (EAC) in tetrahydrofuran with constant stirring. The EAC content was 1–5% by weight of the membrane; The ratio of PVC-plasticizer was 1:2 by weight. The electrode consisted of a cylindrical Teflon body with a plasticized membrane attached to its tip. ~1.5 mL of internal reference solution containing potential-determining ion ($1\cdot10^{-5}$–$1\cdot10^{-4}$ M) and 1–2 drops of 3M KCl was introduced into the electrode body. A silver wire covered with a layer of silver chloride was taken as an internal reference electrode.

## The obtained results and their discussion

Inspection of pointers and electrode calibration of ISE developed on the basis of dodecaphosphomolybdate acid and drug was carried out in the following order. Before measuring the potential, the electrodes were prepared in the prescribed manner. Tests were carried out on the example of ion-selective electrodes (dib.-DDFM, pyridok.-DDFM) prepared using ionophores developed on the basis of drugs (Dibazol and pyridoxine hydrochloride) and dodecaphosphornomolybdate acid. The electrode potential was determined by measuring the EC of the investigated electrode system in accordance with the state standard 8.213–76 at a temperature of 25 ⁰C, using dibazole and pyridoxine hydrochloride solutions with a concentration of $10^{-2}$ mol/l.

The dependence of the signal value of the developed electrodes for detecting different drugs on the concentration of the detected substance in the solution was studied in the concentration range of $10^{-1}$–$10^{-7}$ mol/l. The results of this experiment are presented in (Table 2).

**Table 2.** *Dependence of the signal of ISEs developed on the basis of dodecomolybdophosphate on the concentration of the detectable drug in the solution (n=5, r=0.95)*

| Ion concentration, mol/l | Drug matching signal of ISE, mV | | | | | |
|---|---|---|---|---|---|---|
| | Dibazol hydrochloride | | | Pyridoxine hydrochloride | | |
| | x ± Δx | S | Sr*10² | x ± Δx | S | Sr*10² |
| $10^{-1}$ | 277.2±1.8 | 1.45 | 0.52 | 229.1±2.2 | 1.77 | 0.77 |
| $10^{-2}$ | 232.1±1.9 | 1.53 | 0.66 | 171.1±1.7 | 1.37 | 0.8 |
| $10^{-3}$ | 184.3±1.6 | 1.29 | 0.7 | 115.2±1.8 | 1.45 | 1.26 |
| $10^{-4}$ | 138.4±1.4 | 1.13 | 0.81 | 60.2±0.7 | 0.56 | 0.93 |
| $10^{-5}$ | 95.0±1.0 | 0.8 | 0.85 | 34.3±0.2 | 0.16 | 0.47 |
| $10^{-6}$ | 72.3±1.1 | 0.88 | 1.22 | 15.4±0.4 | 0.32 | 2.09 |
| $10^{-7}$ | 63.2±1.1 | 0.88 | 1.40 | 11.5±0.1 | 1.77 | 0.77 |

The main electrochemical parameters of any ISE include parameters such as the linear range of the signal (of the electrode function), the slope of the electrode function, the lowest limit of ion detection, the selectivity coefficient, and the dynamics of the electrode signal (response time). All parameters of the signal, except dynamics, are determined from the expression E-pA, the dependence of the electrode potential on pA. Using the indicators of the ISE, a conclusion is made about the performance of the electrode. The main characteristic of the measuring electrode is the range of performance of the electrode function (Nernst equation) — this is the part of the range of the concentration dependence of the signal in the range of ion detection, which has a straight line. Based on the results of determining the performance interval of the Nernst region of the membrane electrode developed on the basis of dodecophosphomolybdate and the corresponding drug, it was shown that the developed electrodes have the ability to detect drugs in the concentration ranges of $10^{-1}$–$10^{-5}$ mol/l, depending on the ionophore content. It was observed that the highest detection range of the studied ISEs corresponds to the ISE developed based on pyridoxine and DDFM. The results of determining the electrode function of this electrode are presented in (Figure 1), and we can see that the developed electrode has the ability to detect pyridoxine in the concentration ranges from $10^{-1}$ mol/l to $10^{-5}$ mol/l, depending on the ionophore content.

Another indicator of a drug-selective electrode is the slope of its electrode function (K), which is:

$$K=X/U$$

is found by Eq. In this case, X = ΔE and U = Δp A.

According to the Nernct coefficient, the theoretically calculated value is 58 mV for singly charged ions and 29 mV for doubly charged ions.

Slope percentage of electrode function:

$$A\%= K/58 \cdot 100$$

is determined by Eq., and this indicator is used to determine if the electrode is working properly. From the results of determining the slope of the electrode function of the ISE developed on the basis of EAC containing Dibazole-DDFMK (Figure 2), the value of K was analyzed: K= ΔE/ΔpA=231/4.11=56.2 a and the percentage of the slope = (56.2/58)100 = =96.9%. The results of the study of the slope of the electrode function for the electrodes developed on the basis of membranes with different compositions are presented in (Table 3).

**Figure 1.** *Performance interval of the electrode function of the ISE developed on the basis of Pyridox-DDFMK, which identifies the drug pyridoxine*

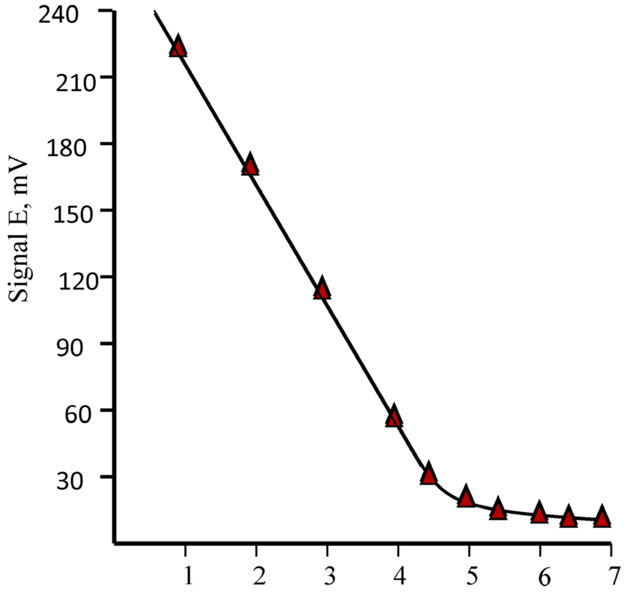


**Figure 2.** *The slope of the electrode function of ISE developed on the basis of EAB containing Dibazole-DDFMK*

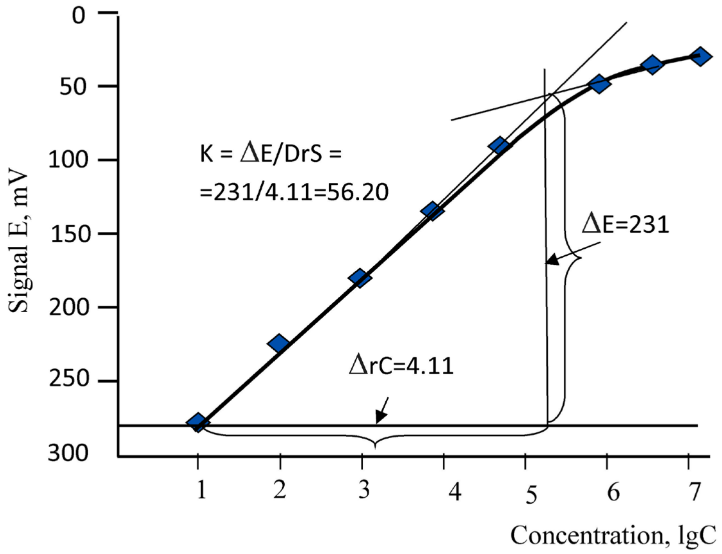


**Table 3.** *Values of the slope magnitude of the electrode function corresponding to the respective ions of the developed ISEs (n=5, P=0.95, Cion = 10–2 mol/l)*

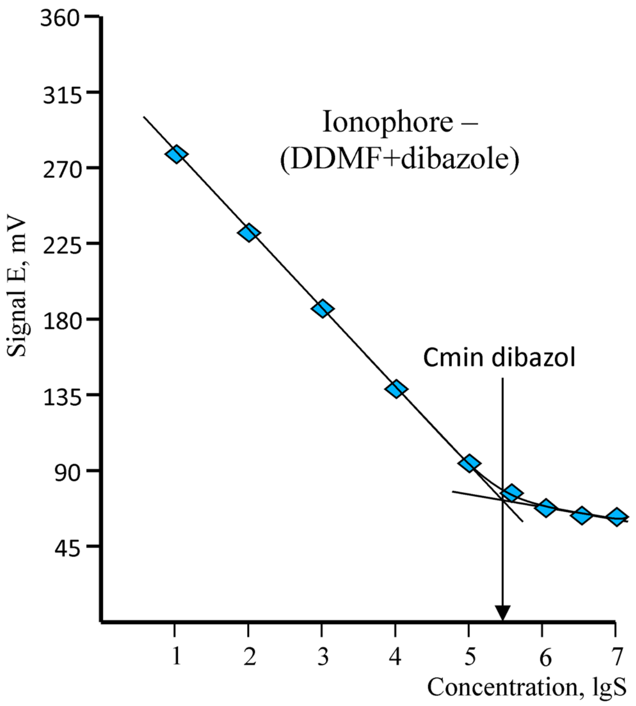
| t/r | The composition of the ionophore | A detectable component | The magnitude of the slope of the electrode function (x±Δx), mV | The percentage of the slope of the function of the electrode (x±Δx),% |
|---|---|---|---|---|
| 1. | $Pyr_3\,[R(W_3O_{10})_4]2H_2O$ | Pyridoxine | 51.8±0.2 | 87.9±0.5 |
| 2. | $Dim_3\,[R(W_3O_{10})_4]2H_2O$ | Dibazol | 56.2±0.1 | 96.9±0.4 |

From the table, we see that the highest value of the slope of the electrode function, equal to 96.9%, corresponds to $Dim_3[R(W_3O_{10})_4]2H_2O$, and the lowest value of this function equal to 87.9% corresponds to $Pir_3[R(W_3O_{10})_4]2H_2O$.

The lower limit of ion selective electrodes in drug detection is one of the main parameters of the electrodeThe linear parts of the E — pA relationship are extrapolated to determine the ion detection limit ($C_{min,\ p}$) that determines the potential. In this case, the intersection point from which the detection limit of the ion is obtained corresponds to the value of $-lgC_{min,\ p}$ on the abscissa axis. It was found that the detection limit of pyridoxine selective electrode (pyr-DDFMK) and dibazole selective electrode (Dib-FMK) corresponds to the range of $10^{-5}$–$10^{-6}$ mol/l.

Figure 3 below shows the results of finding the lowest concentration values of the detectable ion in the determination of dibase using the developed ISE

**Figure 3.***Dib3[R(Mo3O10)4]2N2O lower limit of detection of dibazole using ISE with composition*



## Conclusions

**As a result of the research** for the express analysis of ori products, ion-selective electrodes with plasticized membranes based on dodecomolybdophosphate heteropolyacid were developed and the effects of various factors on their indicators were investigated.

The optimal conditions for the synthesis of poorly soluble electrode-active compounds (EACs) of medicinal substances with DDMF anion were selected and the electroanalytical properties of the obtained EACs were evaluated.

Methods for obtaining plasticized membranes based on synthesized ionophores have been developed. Effect of EAC physicochemical properties, membrane and solution composition on potentiometric characteristics of ISEstudied.

The main electrochemical parameters of the developed ISE: the linear range of the signal (of the electrode function), the slope of the electrode function, the lowest limit of ion detection, etc.determined.

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# SYNTHESIS OF UNSYMMETRICAL MOCHIVENES BASED ON NUCLEOPHILIC COUPLING REACTIONS OF SECONDARY CYCLIC AMINES WITH O-TOLUYL ISOCYANATE

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## Abstract

In the article, for the first time, the methods of synthesis of 1,3-substituted urea derivatives based on m-toluyl isocyanate and 2-aminothiazoles with different functional groups were explained, methods of obtaining 1,3-substituted urea derivatives, reaction mechanisms were studied in depth. The structure of the obtained substances has been proven by analysis of modern physical research methods.

**Keywords:** *synthesis of 1,3-substituted urea, m-toluyl isocyanate, 2-aminothiazoles, substituted urea derivatives, reaction mechanisms, structure, analysis, physical research method*

## Introduction

Substituted ureas attract attracted attention due to its diverse applications in industry, technology, agriculture, and medicine (Kucheryavy V.I., Lebedev V.V., 1970; Vishnyakova T.P., Golubeva I.A., Glebova E.V., 1985; Mashkovsky M.D., 2012). They are widely used in as pesticides and plant growth regulators, are effective additives for hydrocarbon fuels for various purposes, oils, and polymeric materials; are used as medicines and dyes. To date, a huge amount of factual material has been accumulated on synthesis methods, properties and the use of substituted ureas. In 1982 a review has been published (Afanasyev V.A., Dzhamanbaev J.A., Zaikov G.E., 1982; Fukui H., Tanivoto F., Kitano H., 1965; Gerhardt G.W., 1968; Hoffman A.W., 1849; Davis T.L., Blanchard H.C., 1929; Pat. 2673859 USA, IPC 7 07 C 273/18, C 07 C 273/00. 1954; US3161677 (1964) (Schlatter R.); Chem. Abst., 1965; Kuliyev A.M., Abdinova A.B., 1958) examining methods synthesis, structure and properties of a specific class ureas – derivatives of hydrocarbons with urea fragments. It has been established that sulfonylureas, even at very low concentrations, are capable of inhibiting acetolactate synthetase, the first specific enzyme in chains of biosynthesis of isoleucine and valine, which leads to block cell division. Urea derivatives have different selectivity of action depending on their buildings. Thus, sulfonylureas such as chlorsulforone act mainly on dicotyledons plants, and isoproturon acts on foxtail and selective for wheat. Due to the availability of a decently wide range of herbicide preparations Based on urea derivatives, it is possible to use them to control weeds in various crops. Most urea derivatives are soil herbicides. Of significant interest are 1,2,3-thiadiazolylureas, some of them have strong herbicidal effect and can be used as defoliants. From this group substances, the drug has found practical application Thidiazuron defoliant for fine fiber cotton, for which no other effective defoliants have yet been found. By mechanism of action this group of substances can be classified as cytokinins. Along with insecticides, fungicides and herbicides, zoocides were found among the derivatives. The most powerful zoocides are arylpyridyl ureas, of which practical use found N-nitrophenyl-N'-(pyridyl-3-methyl)-urea used as a dietary supplement (0.5% active ingredient). As zoocides Monoarylguanidines have also been proposed. In the first half of the twentieth century, the ability of antibacterial sulfonamide drugs to lower blood sugar was accidentally discovered. Then a targeted search for sulfonamide derivatives with a pronounced hypoglycemic effect began. It has since been synthesized three generations of sulfonylurea's. Despite significant advances in the field diabetology achieved over the past 20 years, diabetes continues to be a problem relevant in almost all countries of the world. To By 2025, there will be 380 million people with diabetes in the world. These numbers sounded at the World Diabetes Congress in Cape Town in 2006. It is predicted that by 2025 7% of the population will suffer from diabetes peace. Moreover, type 2 diabetes mellitus is predicted to scientists, will be 92–97%. Diabetes causes approximately 3.8 million deaths each year, and most of them die from its complications. U In 75% of patients with type 2 diabetes mellitus (DM2), the cause of high mortality is cardiovascular diseases, including coronary disease heart disease, stroke, atherosclerosis of peripheral arteries, arterial hypertension.

## Methods and Results

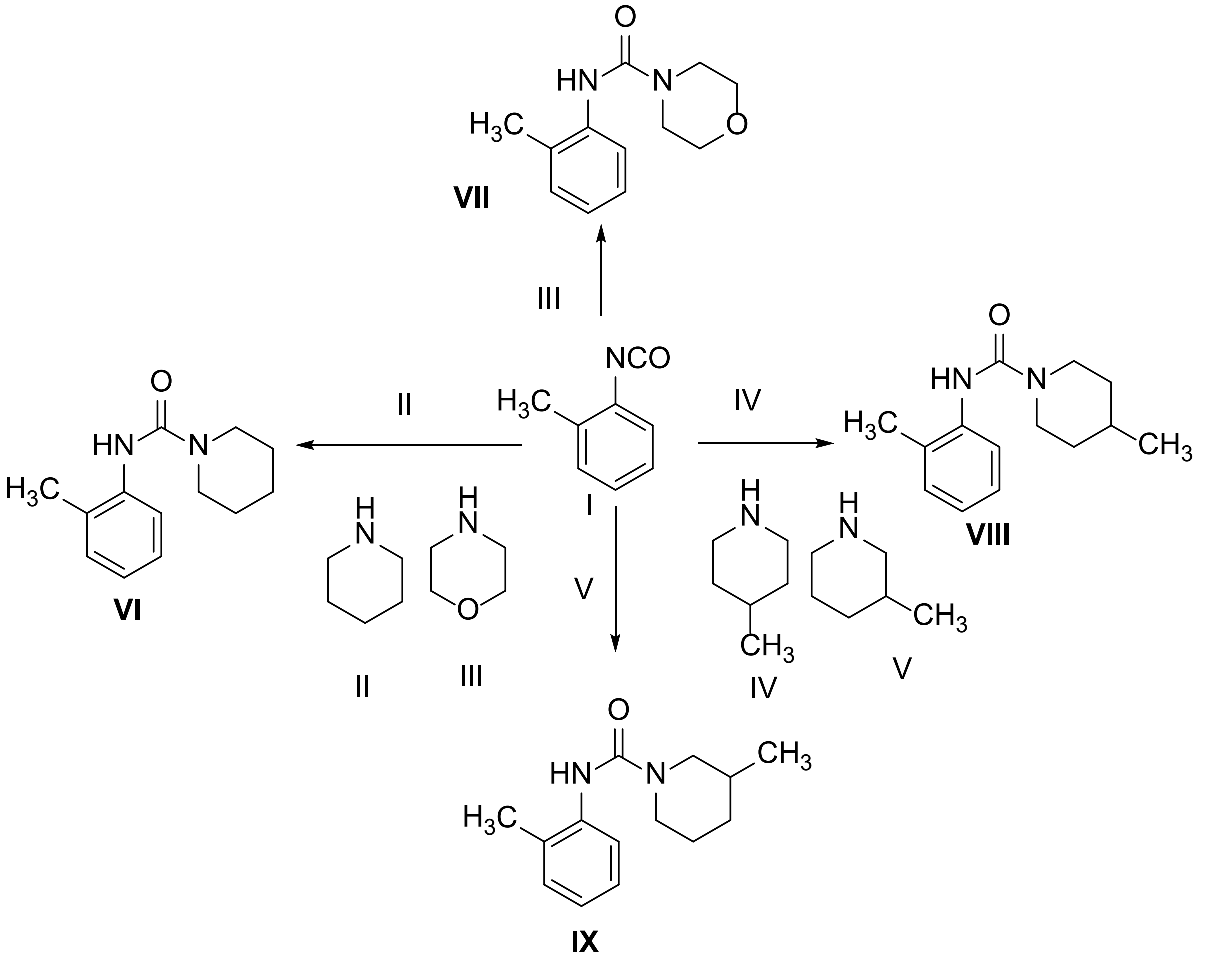
For the synthesis of sulfonylureas, isocyanates are most often used, which, when interacting with sulfonamides, form sulfonylureas. To obtain isocyanates, the reaction of primary aliphatic or aromatic amines with phosgene, which is a chemical warfare agent. The original isocyanates are also compounds with increased toxicity. The reactivity of urea is typical for amides: both nitrogen atoms are nucleophiles, that is, urea forms salts with strong acids, nitrates to form Nnitrosourea, and halogenates to form Nhalogen derivatives. Urea alkylates, forming the corresponding N-alkylureas $RNHCONH_2$, interacts with aldehydes, forming derivatives of 1-amino alcohols $RC(OH)NHCONH_2$, in under harsh conditions, urea is acylated by acid chlorides of carboxylic acids to form ureides (N-acylureas). Sulfonylureas increase insulin secretion by stimulating beta cells pancreas (therefore they act until until the reserves of the insulin synthetic function of the pancreas are depleted). In As a result, the sensitivity of beta cells is restored and the number of insulin receptors increases. The main importance in the treatment of type 2 diabetes mellitus belongs to sulfonylurea drugs, which began to be used in clinical practice. practice since the mid-60s. A wide variety of hypoglycemic derivatives sulfonylureas are associated with radical differences at the phenolic ring. Sulfonylureas are divided into drugs 1 and 2 generation. Today, first generation sulfonylurea drugs (tolbutamide, chlorpropamide) are practically not used. Currently, there are several main methods for producing substituted ureas, Among them are the following: 1) Interaction of amino compounds with organic isocyanates; 2) Interaction of amines and alkyl halides with alkali metal cyanates; 1) Interaction of primary and secondary amines with phosgene; 2) Interaction of amines with urea and nitrosourea; 3) Interaction of urea with various connections; 4) Carbonylation of amines to substituted ones urea; 5) Synthesis of substituted ureas from amides acids (Hoffmann rearrangement).

One of the simplest methods for producing urea derivatives is the interaction of the amine with isocyanates. Isocyanates are organic compounds containing the functional group $-N = C = O$. Isocyanates, being heterocumulenes, are active electrophilic reagents. When interacting with primary and secondary amines, they form substituted ureas, with alcohols they form carbamates (urethanes), and are hydrolyzed with water to amines and carbon dioxide.

The most common synthesis method isocyanates – reaction of amines with phosgene (phosgenation of amines), the reaction occurs in an inert solvent through the intermediate formation of carbamoyl chlorides. The reaction occurs both in organic solvents and without them. The process temperature depends on the structure of the starting isocyanates and amine. In

Tertiary amines or organic tin compounds can be used as catalysts. At When the process is carried out correctly, the product is obtained in almost quantitative yield. The isocyanates required for this method are obtained from reactions of the corresponding amines with phosgene, such as usually at elevated temperatures. This method is the main industrial method for the synthesis of isocyanates. Sulfonylisocyanates are prepared similarly. Other acyl isocyanates with better yields are formed from amides and oxalyl chloride. On the basis of various solvents, we continued our scientific work and synthesized urea derivatives based on toluene isocyanates. The reaction scheme is expressed as follows.

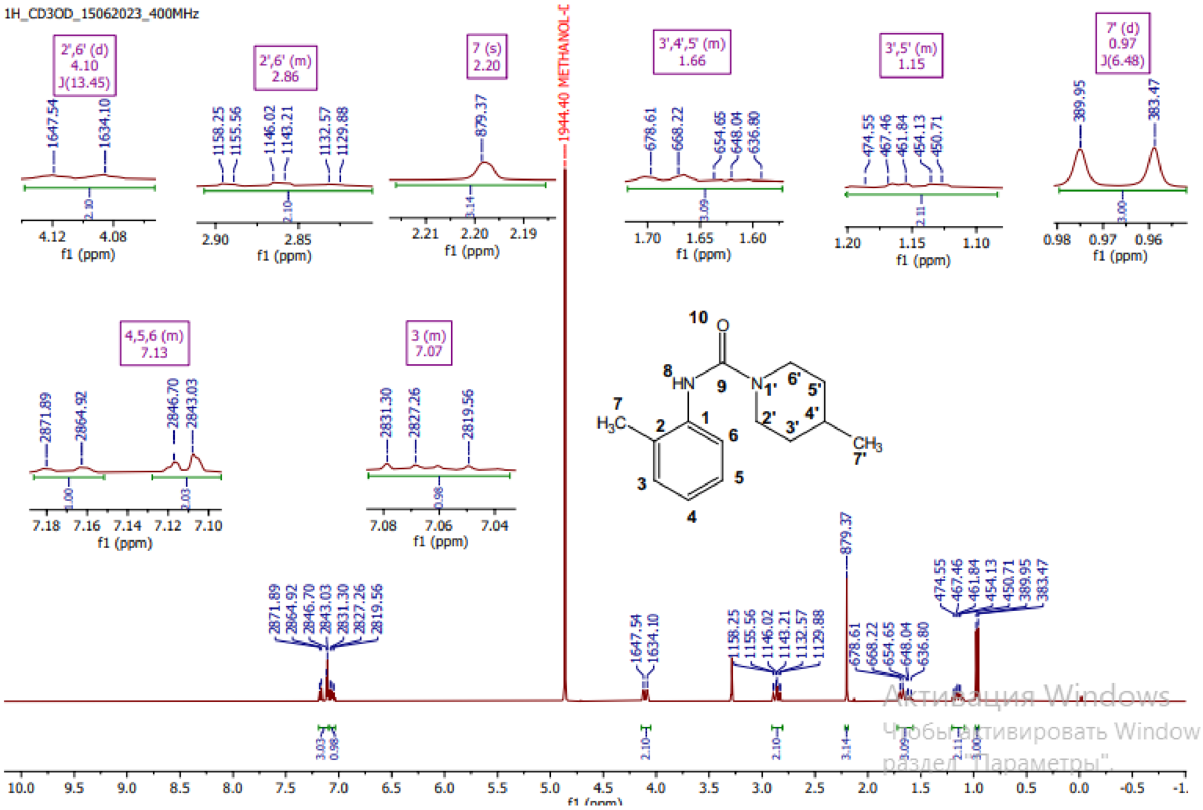
**Figure 1.**



1,3-substituted aryl-heteryl urea derivatives were synthesized in good yields. -IR, $^1$H-NMR, $^{13}$C-NMR spectra of the obtained substances were analyzed.

The structure of 8 substance molecules was determined using $^1$H NMR physical research methods (Fig. 1)

**Figure 1.** *8 substance molecules was determined using $^1$H NMR*



### Experimental Part

### Synthesis of N-(toluyl)piperidine-1-carboxamide.

0.385 ml (3 mmol, 0.413 g, r=1.073 g/ml) of o-toluyl isocyanate, 0.383 ml (3.88 mmol, 0.33 g, $\rho$=0.862 g/ml) of piperidin were added to a 100 ml round-bottom flask. 10 ml of acetone was poured into the flask and mixed on a magnetic stirrer. After the substances were completely dissolved, 0.414 g (3 mmol) of potash was added and mixed. The flask was stirred on a magnetic stirrer connected to a reflux condenser for 4–8 hours. The progress of the reaction was monitored by thin layer chromatography. The reaction mixture was left at room temperature overnight. The precipitate was filtered, extracted with $K_2CO_3$ and dried to give 0.487 g. (73%) product was obtained. The $^1$H NMR-spectrum data of substance I presented below fully prove the structure of this substance: 1.63(6H, m, Het-C-H-3′,4′,5′), 2.20(3H, c, Het-$CH_3$), 3.47 (4H, m, Het-C-H-2′, 6′), 7.06(1H, м, Ar-4), 7.11(2H, d, J=2.9, Ar-5,6), 7.17(1H, d, Ar-3). **$^{13}$C NMR ($CD_3OD$):**157.27, 137.26, 134.31, 130.08, 126.63, 125.87, 47.03, 45.04, 25.67, 24.26, 16.83.

### Synthesis of N-(toluyl)morphaline-4-carboxamide

0.247 ml (2.65 mmol, 0.353 g, r=1.073 g/ml) o-toluyl isocyanate, 0.3 ml (3.32 mmol, 0.3 g, $\rho$=1.007 g/ml) morphalin 10 ml acetone 0.445 g (2.65 mmol) $K_2CO_3$ was obtained. 0.36 g product was obtained with 62% yield. $^1$H NMR **($CDCl_3$):** 2.21(3H, d, J=0.9, Ar-$CH_3$), 3.48(4H, м, Het-C-H-2′,6′),3.69(4H, м, Het-C-H-3′,5′), 7.08(1H, d, $J_1$=7.4, $J_2$=5.3, Ar-4), 7.13(2H, м, Ar-5,6), 7.19(1H, d, J=7.6, Ar-3). **$^{13}$C NMR ($CD_3OD$):**157.46, 136.89, 134.33, 130.16, 126.60. 125.69, 66.38, 48.31, 44.30, 16.81.

### Synthesis of 4-methyl-N-(toluyl) piperidine-1-carboxamide.

0.37 ml (3 mmol, 0.398 g, r=1.073g/ml) o-toluene isocyanate, 0.442 ml (3.7mmol), 0.37 g, $\rho$=0.838g/ml) 4-methylpiperidine 10 ml acetone 0.414g (3 mmol) of $K_2CO_3$ was obtained. 0.622 g product was obtained with 89% yield. **$^1$H NMR ($CDCl_3$):** 0.97(3H, d, J=6.5, Het-$CH_3$), 1.15(2H, м, Het-C-H-3′,5′), 1.66(3H, м, Het-C-H-3′,4′,5′), 2.20(3H, c, Ar-$CH_3$), 2.86(2H, м, Het-C-H-2′,6′), 4.10(2H, d, J=13.4, Het-C-H-2′,6′), 7.07(1H, м, Ar-3), 7.13(3H, м, Ar-4,5,6).**$^{13}$C NMR ($CD_3OD$):**

157.25, 137.24, 134.31, 130.08, 126.63, 125.88, 125.47, 47.03, 44.43, 33.99, 30.98, 20.94, 16.83.

### Synthesis of 3-methyl-N-(toluyl) piperidine-1-carboxamide

0.343ml (2.76 mmol, 0.343g, $\rho = 1.073$g/ml) o-toluyl isocyanate, 0.405 ml (3.45 mmol), 0.342g, $\rho = 0.845$g/ml) 3-methylpiperidine, 10 ml acetone 0.35g (2.76 mmol) of $K_2CO_3$ was obtained. 0.59 g product was obtained with 96% yield. **$^1$H NMR (CDCl$_3$):**0.93(3H, , J=6.7, Het-CH$_3$), 1.16(1H, м, Het-C-H-4′), 1.62(1H, м, Het-C-H-4′,5′,5′), 1.85(1H, м, Het-C-H-3′), 2.53(1H, м, Het-C-H-2′), 2.85(1H, м, Het-C-H-2′), 3.99(2H, т, J=13.0), 7.09(3H, м, Ar-4,5,6), 7.15(1H, d, J=7.7, Ar-3). **$^{13}$C NMR (CD$_3$OD):**157.07, 137.24, 134.17, 130.15, 126.59, 125.46, 51.63, 44.64, 32.98, 31.15, 25.14, 18.25, 17.17.

## Conclusion

Outlined basic methods of obtaining zameshchennyx urea, kak semi-products and synthesis of biologically active preparations (herbicides, fungicides, root preparations, saccharosnijayuschich preparations and others). Opredeleny sposoby polucheniya naibolee dostupnyx aryl-, heteroarylurea. Named basic principle synthesis of zameshchennyx urea, used in the synthesis of biologically active compounds.

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# SYNTHESIS OF UREA DERIVATIVES BASED ON SUBSTITUTED2-AMINOTHIAZOLES AND SOME AROMATIC ISOCYANATES

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## Abstract

Synthesis of 1,3-substituted urea derivatives based on m-toluyl isocyanate and 2-aminothiazoles with different functional groups. Analysis of the structure of the obtained substances in modern physical research methods. 1,3-substituted urea derivatives were synthesized based on m-toluyl isocyanate and 2-aminothiazoles. The structure of the synthesized substances was proved by IR, $^1$H NMR, $^{13}$C NMR, spectral analysis.

**Keywords:** *m-Toluyl isocyanate, DMFA,* $K_2CO_3$*, 2-(3-(m-tolyl)urea)-4,5-dihydrothiazole-4-carboxylic acid, 1-(benzo[d]thiazol-2-yl)-3-(m-tolyl)urea*

## Introduction

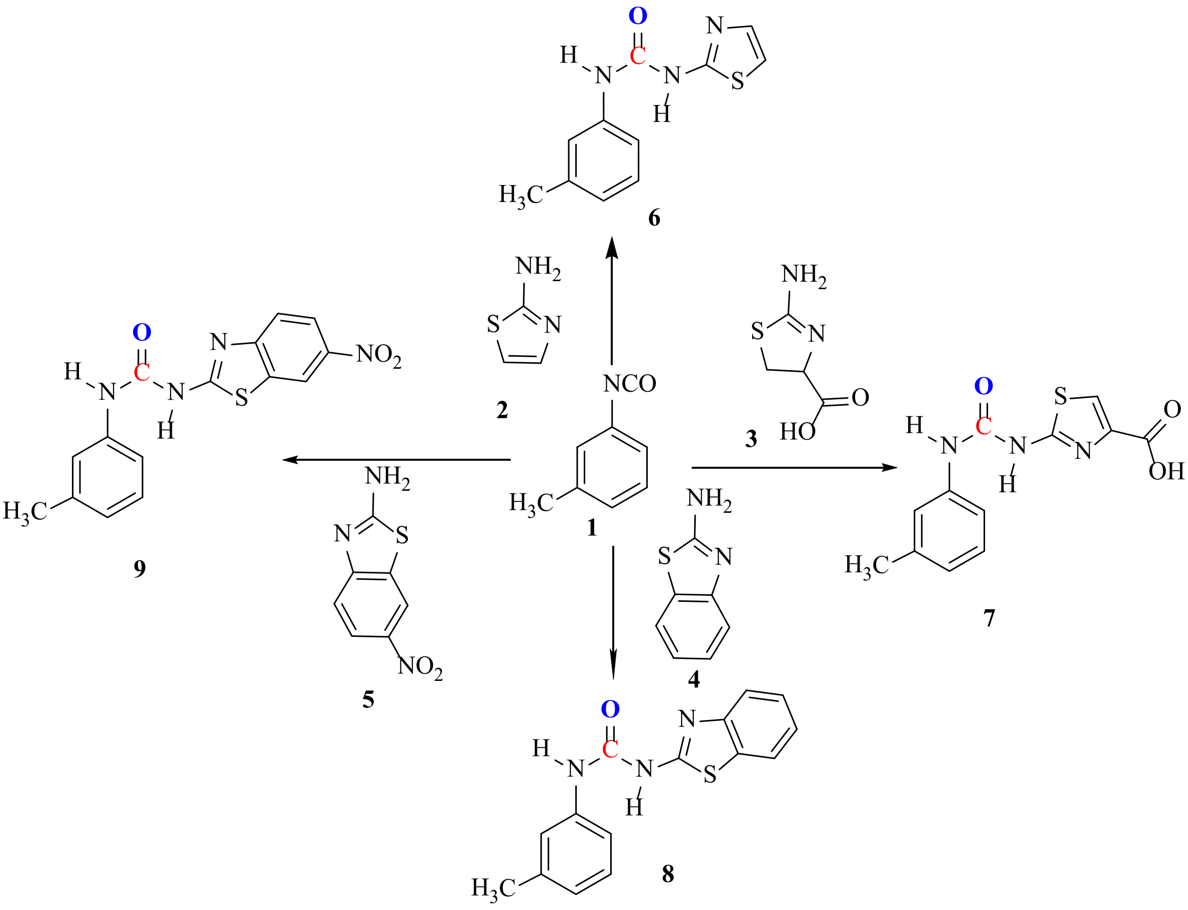
Modern search in the currently intensively developing chemistry and properties of urea compounds attract the attention of many researchers both in Uzbekistan and abroad (Suresh, G., Nadh, R.V., Srinivasu, N., Yennity, D., 2018; Das, D., Sikdar, P., Bairagi, M., 2016; Alexandru, M.-G., Velickovic, T.C., Jitaru, I., Grguric-Sipka, S., Draghici, C., 2010). This is due, on the one hand, to those rich capabilities of diphenyl, azo-diphenyl, bis-urea, polyhydrocarbon groups in molecules of organic macrocompounds, and on the other hand, with valuable practical using the properties of organic substances themselves compounds diphenyl, azodiphenyl groups, as well as bis-urea bonds. There are many examples where the introduction of azo-, phenyl, diphenyl bridging bonds would lead to the emergence of various kinds of biological, pharmacological, physiological activity, as well as the ability to inhibit corrosion of metals, coatings, and stabilizers for halogen-containing polymers, impregnations, and also as an anti-aging vulcanization of rubbers, the creation of solvation theories for intensifying the processes of dyeing and printing fabrics from natural and chemical fibers in liquid ammonia and organic solvents.

At present, the synthesis of urea derivatives kept by various pharmacophoric groups and the study of their biological activities are important tasks. Because among the compounds of this class there are many drugs against diabetes, cancer, hepatitis and many other diseases, as well as substances with high fungicidal and herbicidal activity in agro-industry. That is why the synthesis of 1,3-substituted ureas, determination of their structure by physico-chemical methods and study of their biological activity are considered urgent issues.

## Method and Results

Interest in the chemistry of 2-aminothiazole in recent years is explained by the discovery of a wide spectrum of biological activity in a number of its derivatives. In addition, the 2-aminothiazole molecule, which has a large number of reaction centers, cannot but interest chemists (Décor, A., Grand-Maître, C., Hucke, O., O'Meara, J., Kuhn, C., Constantineau-Forget, L., Brochu, C., Malenfant, E., Bertrand-Laperle, M., Bordeleau, J. 2013; Jalani, H.B., Pandya, A.N., Pandya, D.H., Sharma, J.A., Sudarsanam, V., Vasu, K.K., 2013; Stefanska, J., Nowicka, G., Struga, M., Szulczyk, D., Koziol, A.E., Augustynowicz-Kopec, E., Napiorkowska, A., Bielenica, A., Filipowski, W., Filipowska, A., 2015; Elshaarawy, R.F., Mustafa, F.H., Sofy, A.R., Hmed, A.A., Janiak, C., 2019; Chen, Y.-Y., Gopala, L., Bheemanaboina, R.R.Y., Liu, H.-B., Cheng, Y., Geng, R.-X., Zhou, C.-H., 2017; Prakash, A., Malhotra, R. 2018; Cordeiro, Y., Ferreira, N.C., 2015). Therefore, the goal of our work was to obtain new heterocyclic compounds based on 2-aminothiazole using the halocyclization reactionBased on the following reactions, 1,3-substituted urea derivatives were synthesized based on m-toluyl isocyanate and 2-aminothiazoles with different functional groups. As a result of the action of molecules of substances 2, 3, 4, 5 of raw material 1, corresponding products 6, 7, 8, 9 were synthesized in high yield.

**Figure 1.**



m-Toluyl isocyanate and 2-aminothiazoles 1,3-substituted urea derivatives were synthesized. Potash was used as a catalyst in the reaction. Factors affecting the course of the reaction were determined.

1,3-substituted aryl-heteryl urea derivatives were synthesized in good yields. -IK, 1H-NMR, 13S-NMR spectra of the obtained substances were analyzed.

## Experimental Part

**1-(THIAZOL-2-YL)-3-(M-TOLYL) UREA**. 0.71 ml (5.48 mmol, 0.73 g, $\rho$ = =1.033 g/ml) of m-toluyl isocyanate (1), 0.55 g (5.49 mmol) of 2-aminothiazole (2) were taken in a 100 ml round-bottom flask. 10 mL of DMFA was added to the flask and mixed on a magnetic stirrer. After the substances were completely dissolved, 0.83 g (6 mmol) of potash was added and mixed. The flask was stirred on a magnetic stirrer connected to a reflux condenser for 4–8 hours. The progress of the reaction was monitored by thin layer chromatography. The reaction mixture was left at room temperature overnight. The precipitate was filtered off, extracted with K2SO3 and dried to give 1.08 g. (84%) product (6) was obtained. Rf =0.62 (benzene: methanol – 5:1). 1N YaMR (m.u CD3OD): 10.03 (1H, c, NH-), 7.70 (1H, c, Ar-H-2), 7.62 (1H, d, J=5,72, Ar-H-6), 7.48 (1H, d, J=3.64, Het H-4), 7.20 (1H, t, J=7.80, $H_{Ar}$-5), 6.98 (1H, d, J=3,64, $H_{Het}$-5), 6.83 (1H, d, J=7.54, $H_{Ar}$-4), 2.15 (3H, c, Alk-$CH_3$). **$^{13}$C NMR ($CD_3OD$):** 162.7 ($C_{Het}$-2), 153.9 (C=O C), 140.5 (Ar-C-3), 139.6 ($C_{Ar}$-1), 138.3 ($C_{Het}$-4), 129.8 ($C_{Ar}$-5), 124.7 ($C_{Ar}$-4), 121.0 ($C_{Ar}$-4), 117.0 ($C_{Ar}$-6), 112.7 ($C_{Het}$-5), 21.4 ($C_{CH3}$).

**2-(3-(M-TOLYL)UREA)THIAZOLE-4-CARBOXYLIC ACID**. 0.45 ml (3.5 mmol, 0.46 g. $\rho$ =1,033 g/ml) m-toluene isocyanate (1), 0.46 g (3.19 mmol) 2-aminothiazole-4-carboxylic acid (3), 10 ml DMFA and 0.45 g (3.26 mmol) of $K_2CO_3$ was obtained. 0.31 g of product (7) was obtained in 62% yield. . $R_f$ = 0.77 (benzene: methanol–5:1). **$^1$H NMR (δ, m.u., $CD_3OD$):** 10.74 (1H,-COOH-H), 8.49 (1H,-NH-H), 7.69 (1H, c, $H_{Het}$-4), 7.25 (1H, c, $H_{Ar}$-6), 7.19 (1H, d, J=9.22, $H_{Ar}$-2), 7.09 (1H, t, J=7.80, $H_{Ar}$-5), 6.76 (1H, d, J=7.80, $H_{Ar}$-4), 2.30 (3H, $H_{CH3}$). **$^{13}$C NMR($CD_3OD$):** 162.03 ($C_{–COOH}$), 160.66 ($C_{Het}$-2), 152.56 ($C_{–C=O}$), 142.46 ($C_{Het}$-4), 139.57 ($C_{Ar}$-3), 139.08 ($C_{Ar}$-1), 129.56 ($C_{Ar}$-5), 124.50 ($C_{Ar}$-2), 122.13 ($C_{Ar}$-4), 120.33 ($C_{Ar}$-6), 116.91 ($C_{Het}$-5), 21.89 ($C_{Alk-CH3}$). **IR (ν, $cm^{-1}$):** 3114 (C–H aryl), 2977 ($CH_2$ for the group), 676, 694 (C–S-C), 1614 (–C=N-), 1681 (>C=O), 676 (Ar (=C-H deform.t.), 1379 (–C–$CH_3$), 1121, 1107 (–C-OH).

**1-(BENZO[d]THIAZOL-2-YL)-3-(M-TOLYL)UREA.** 0.7 ml (5.4 mmol, 0.72 g, $\rho$ =1.033 g/ml) of m-toluyl isocyanate (1), 0.81 g (5.4 mmol) of 2-aminobenzothiazole (4), 10 ml of DMFA and 0, 77 g (5.6 mmol) of $K_2CO_3$ were obtained. 1.27 g of product (8) was obtained in 83% yield. Liquid=340–342oC. Rf =0.76 (benzene: methanol – 5:1). 1HYaMR (m.u., CD3OD): 10.58 (1H, c, -NH), 8.82 (1H, c, -NH), 7.74 (1H, c, Het-H-4), 7.57 (1H, d, J=6.49, $H_{Ar}$ –7), 7.28 (2H, m, $H_{Het}$ –5.6), 7.23 (1H, d, $H_{Ar}$-3), 7.13 (2H, m, $H_{Ar}$ –2,6), 6.77 (1H, d, J=7,53, $H_{Ar}$ –4), 2.32 (3H, c, $H_{Alk-CH3}$). **$^{13}$C NMR ($CD_3OD$):** 160.73 ($C_{Het}$-1), 152.26 ($C_{–C=O}$), 139.66 ($C_{Ar}$-3), 139.13 ($C_{Ar}$-1), 132.78 ($C_{Het}$-7a), 129.60 ($C_{Ar}$-5), 126.54 ($C_{Ar}$-2), 124.53 (C-5), 120.70 ($C_{Ar}$-6), 120.32 ($C_{Het}$-4), 116.92 ($C_{hetaeryl}$-7), 21.91 (). **IR (ν, $sm^{-1}$): IR-(ν, $sm^{-1}$):** 3049 (C–H aryl), 728 (C–S-C), 1725 (>C=O), 690 (Ar (=C-H), 1451 (–C–$CH_3$), 1194, 1194 (–C-OH).

**1-(6-NITROBENZO[d]THIAZOL-2-YL)-3-(m-TOLYL)UREA.** 0.26 ml (2.03 mmol, 0.27 g., $\rho$ =1.033 g/ml) m-toluyl isocyanate (1), 0.4 g (2.05 mmol) 2-amino-6-nitrobenzothiazole (5), 10 ml DMFA and 0.304 g (2.2 mmol) of $K_2CO_3$ were obtained. 0.4 g of product (9) was obtained in (59%) yield. Liquid=240–242oC. Rf =0.65 (benzene: methanol – 5:1). 1H NMR(m.u., CD3OD): 10.23 (1H, c, NH-), 8.86 (1H, d, J=2,47, $H_{hetaeryl}$-4), 8.22 (1H, dd, J=8.90, J=2.40, hetaeryl-6), 7.74 (1H, c, $H_{hetaeryl}$-7), 7.66 (2H, t, J=8.51, $H_{Ar}$-2,6), 7.24 (1H, t, J=7.80, $H_{Ar}$-5), 6.89 (1H, d, J=7.54, $H_{Ar}$-4), 2.18 (3H, c, $H_{Alk-CH3}$). **$^{13}$C NMR ($CD_3OD$):** 167.3 (C-1), 154.5 ($C_{C=O}$), 144.0 ($C_{getr}$-3a), 140.1 ($C_{getr}$-5), 139.7 ($C_{Ar}$-7a), 133.3 ($C_{getr}$-7), 129.9 ($C_{Ar}$-1), 125.3 ($C_{Ar}$-5), 122.7 ($C_{Ar}$-2), 121.5 ($C_{Ar}$-4), 119.7 ($C_{getr}$-6), 119.3 ($C_{Ar}$-6), 117.9 ($C_{getr}$-4), 21.3 ($C_{CH3}$-C). **IR (ν, $sm^{-1}$):** 3130 (C–H aryl), 747 (C–S-C), 1685 (>C=O), 642 (Ar (=C-H 1447 (–C–$CH_3$), 1549 (–C-$NO_2$).

## Consulsion

The heterocycles of 2-aminothiazole scaffolds occupy a dominant part in organic/medicinal chemistry in relation to their reactivity and biological activity and mostly act as pharmacophores. The present review summarizes the literature reports of the various synthetic routes for 2-aminothiazole-containing molecules with four different biological activities (namely, anticancer, antioxidant, antimicrobial and anti-inflammatory activities). The presented information in this review is valuable for future innovation.

This review highlighted the recently synthesized 2-aminothiazole-containing compounds within the last thirteen years ago. Further, the synthetic strategies developed for the admission of the recent 2-aminothiazole derivatives (N-substituted, 3-substituted, 4-substituted, multi-substituted, aryl/alkyl substituents or acyl/other substituents) were presented. The reported literature revealed several synthetic pathways of those 2-aminothiazoles related to four different biological activities (anticancer, antioxidant, antimicrobial and anti-inflammatory activities). It is hoped that this review will be useful in displaying the rationalistic designs of 2-aminothiazole-based medical synthetic pathways.

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# STUDYING THE RHEOLOGICAL PROPERTIES AND FILTERABILITY OF INTERMEDIATE, FINAL SOLUTIONS AND SUSPENSIONS FORMED WHEN PROCESSING SULFATE-CONTAINING SALTS

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## Abstract

The purpose of the research was to study the rheological properties and filtration rate of glaserite and schenite suspensions depending on the L:T ratio. Experimental studies have established that changes in the density and viscosity of the formed suspension depend on the L:S ratio and the characteristics of the constituent salts of the system. And the filtration rate of glaserite suspensions fluctuates in the ranges of 1937.10÷2056.33 kg/h · m$^2$, and for the solid phase - with an increase in the L:S ratio, an increase in filtration rate is observed, which ranges from 404.98-2165.80 kg /h · m$^2$. When using chenite suspensions, the filterability in the liquid phase is in the range of 1940.10÷2234.90 kg/h · m$^2$. The solid phase filtration rate fluctuates in the ranges of 550.81÷1996.25 kg/h · m$^2$, sediment moisture is on average 2.5–2.6% lower, and the filtration rate is on average 100–200 kg/h · m$^2$ more than glaserite sediments.

**Keywords:** *system, rheology, density, viscosity, filtration, potassium sulfate, fertilizers, sulfate salts, astrakhanite, schenite, glaserite*

## Introduction

The potash industry is characterized by very high rates of development. Potassium is one of the most important nutrients for agricultural crops. Its deficiency in the soil leads to a significant decrease in soil fertility (№ PP–3236 dated August 15, 2017).

In terms of the amount of potassium fertilizers used per hectare of arable land, our country lags behind developed countries. A persistent deficit in potassium fertilizers has determined their primary use for industrial crops and vegetables. The yield of grain crops is determined mainly by the natural fertility of the soil. Wider use of potash fertilizers for grain crops is one of the most important reserves for their productivity (Osichkina R.G., Popov V.S., Tilyakhodzhaev Kh. –№5003.–12.07.84).

The specialization of Uzbekistan in cotton production necessitated the creation of the development of the chemical industry in the republic and its subordination, first of all, to the tasks of intensifying cotton growing. For each hectare of crops, an average of 220–250 kg of nitrogen, 100–120 kg of phosphorus pentoxide and 90–70 kg of potassium fertilizers ($K_2O$) are applied. As a result of an agrochemical survey of irrigated lands in Uzbekistan, it was found that on 75% of the areas of cotton–growing areas and as a result of the use of insufficient doses of potassium fertilizers, the removal of potassium with the harvest and annual leaching of saline lands, the soils are depleted of exchangeable potassium.

The need for potash fertilizers in cotton growing in Uzbekistan alone reaches more than 800 thousand tons. The need to increase the norms of potassium fertilizers has been proven not only for cotton, but also for other crops (corn, melons, fodder), which will further increase the demand for potash approvals (Kurnakov N.S., Shoikhet D.N., SFHAD 984). Chlorine–free potassium fertilizers can also be obtained from glauconite and through complex processing of alunites and syenites. However, further research is needed to select the most rational ways of processing and using this type of raw material (Sokolovsky A.A., Unanyants T.P., 1977.).

Further development should be achieved by enterprises producing potassium fertilizers by converting potassium chloride into sulfate salts chenite, langbeinite, glaserite, astrakhanite, etc. Several schemes for the conversion production of potassium sulfate have been proposed, in particular options where iron sulfate, an industrial waste, is used as a sulfate component – inorganic pigments and etching of metals with sulfuric acid. This method as a whole also does not solve the problem of chlorine–free potassium fertilizers (Sedletsky V.I. 1982, Popov B.S., Osichkina R.G., Dep. VINITI. –No. 3232. 1981).

In the Central Asian region, it is possible to organize the production of potassium sulfate on the basis of local raw materials of sulfate salts (thenardite, mirabilite, astrakhanite) from the salt deposits of the Aral Sea region (Akkala, Kushkanataw), located in Karakalpakstan. Reserves of mirabilite at the Akalinskoye deposit alone exceed 2 billion tons (Mirzakulov Kh.Ch., Zhuraeva G.Kh., Kh.Ch. Mirzakulov, G.Kh. Zhuraeva. 2014).

## Methods and materials

The density of solutions was determined by the hydrometric method according to GOST (state standard) 18481 for general purpose with a division scale of 1 kg/m$^3$ (0.001 g/cm$^3$), the error in the accuracy of the results ranges from 0.0005 to 0.02 g/cm$^3$ (GOST (state standard) 18995.1–73. 2004). Viscosity was determined using a glass capillary viscometer brand VPZ (hanging level viscometer) in the temperature range 10–40 ºC, the maximum deviation of which is ± 0.02 mm (GOST (state standard) 10028–81. 2005).

The sodium and potassium content in the obtained samples was determined according to the GOST (state standard) 20851.3–93 method (GOST 20851.3–93. 1995). To determine the composition of an aqueous solution, sulfate ions were determined according to the GOST (state standard) 8.315 method (GOST 24024.12–81. 1981) and the permissible relative error of the certified value was no more than 1.0%. Chlorine ion was determined according to the GOST (state standard) 4245–72 methods.

## Results and discussion

For the purpose of physical and chemical substantiation, development of scientific foundations and technology for processing salt deposits of the Aral region and obtaining potassium sulfate from sulfate deposits with the establishment of optimal process parameters, an analysis of the solubility diagram of the system $2Na^+, 2K^+, Mg^{2+}, // SO_4^{2-}, 2Cl^- - H_2O$ was carried out by isothermal method at temperatures 25, 50 and 75 °C (Kucharov B.Kh., Zakirov B.S., A.U. Erkaev, Z.K. Toirov, O.A. Mursaev, N.A. Ulashova. 2016, Kucharov B.Kh., Zakirov B.S., Erkaev A.U., Orakbaev A.A., Reymov, 2019, Kucharov B.Kh., Zakirov B.S., Erkaev A.U. 2019).

From the results of a theoretical analysis of the solubility diagram, it was revealed that the salt deposits of the Aral region mainly consist of sodium or magnesium sulfates and/or their mixtures at certain ratios.

We also investigated the physicochemical properties of sulfate deposits of the Aral region (astrakhanite, glaserite and schenite), which can serve as raw materials for the production of potassium sulfate (N.A. Ulashova, Kucharov B. Kh., Erkaev A. U., Zakirov B. S. 2024).

Based on a theoretical analysis of the solubility diagrams of $2Na^+, 2K^+, Mg^{2+}//SO_4^{2-}, 2Cl^-$–$H_2O$ at 0; 25 and 75 °C, to provide practical recommendations for the processing of salt deposits, we have proposed a technological scheme. The process of producing glaserite and shenite is carried out using potassium chloride from "Dekhkanabad Potash Fertilizer Plant" JSC and astrakhanite from salt deposits of the Aral region. The technological process includes the following main stages:

– loading a circulating solution, potassium chloride, astrakhanite into the reactor, followed by conversion at 75 °C;

– filtration and separation of glaserite from the solution;

– adding the calculated amount of potassium chloride to the mother solution and dissolving;

– filtration and separation of chenite from the solution at 0 °C;

This article provides data on the study of the rheological properties and filtration ability of the suspension that is formed during the complex processing of astrakhanite, glaserite and schenite with potassium chloride.

When studying the rheological properties of the resulting suspension of glaserite and schenite, the L:S ratio was maintained from 1:1 to 4:1, and the temperature varied from 0 to 45 °C.

The obtained data are shown in (tables 1 and 2) and (figures 1–4). The tables and figures show the rheological properties of glaserite and chenite solutions and suspensions. Table 1 shows that the rheological properties of glaserite suspensions with increasing temperature to 25 °C – the viscosity and density of the suspensions gradually increase, a further increase in temperature leads to a decrease in the density and viscosity of the formed suspensions. It was revealed that at L:S = 2:1 and L:S = 4:1, the maximum values of these parameters are observed at low temperatures than at high temperatures. This fact gives us reason to assume that the formation or splitting of double crystalline hydrates with single salts occurs.

**Table 1.** *Rheological properties of the suspension during the conversion of $Na_2SO_4$ with potassium chloride*

| **No.** | **Ratio** | **Density, $\rho$ g/cm$^3$, at temperature, °C.** | | | **Viscosity, $\eta$, mm$^2$/sec at temperature, °C.** | | |
|---|---|---|---|---|---|---|---|
| | L:S | 10 | 25 | 40 | 10 | 25 | 40 |
| 1 | 1:0 | 1.213 | 1.210 | 1.205 | 1.750 | 1.642 | 1.412 |
| 2 | 1:1 | 1.282 | 1.271 | 1.260 | 2.231 | 2.091 | 1.635 |
| 3 | 2:1 | 1.242 | 1.282 | 1.240 | 2.220 | 1.932 | 1.528 |
| 4 | 3:1 | 1.240 | 1.243 | 1.230 | 2.022 | 1.851 | 1.417 |
| 5 | 4:1 | 1.237 | 1.288 | 1.221 | 1.830 | 1.733 | 1.322 |

**Table 2.** *Rheological properties of the suspension during the conversion of $MgSO_4$ with potassium chloride*

| **Sample number corresponds to the table 1** | **Density, $\rho$, g/cm$^3$, at temperature, °C** | | | | **Viscosity, $\eta$, mm$^2$/sec, at temperature, °C** | | | |
|---|---|---|---|---|---|---|---|---|
| | 0 | 15 | 30 | 45 | 0 | 15 | 30 | 45 |
| 1 | 1.144 | 1.140 | 1.138 | 1.134 | 1.930 | 1.8130 | 1.6769 | 1.4870 |
| 2 | 1.147 | 1.149 | 1.158 | 1.166 | 1.990 | 1.9040 | 1.8740 | 1.7060 |
| 3 | 1.155 | 1.158 | 1.170 | 1.170 | 2.144 | 2.0377 | 1.9607 | 1.4550 |
| 4 | 1.175 | 1.177 | 1.170 | 1.179 | 2.419 | 2.3940 | 2.3500 | 2.1449 |
| 5 | 1.224 | 1.235 | 1.221 | 1.217 | – | – | – | – |

**Figure 1.** *Change in suspension density depending on process parameters*

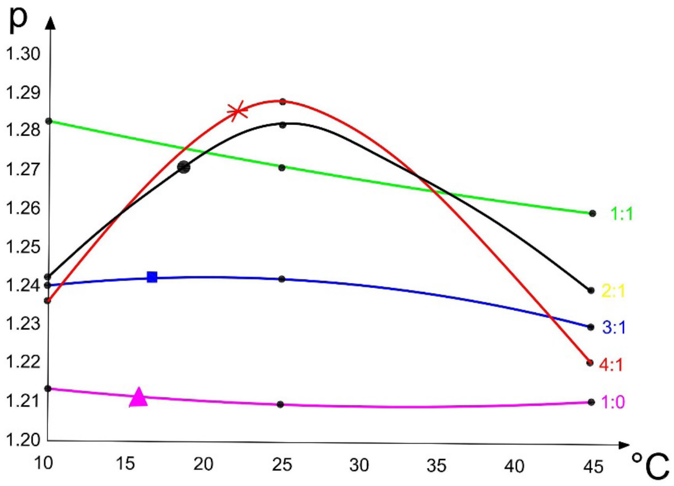
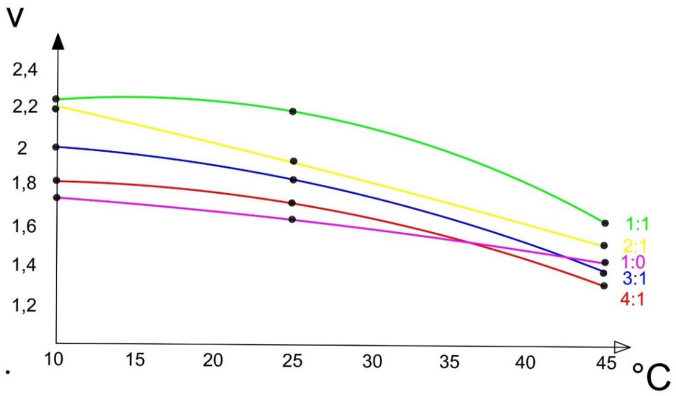


**Figure 2.** *Change in suspension viscosity depending on process parameters*



From (Table 2) it follows that with an increase in temperature from 0 to 45 °C and with an increase in the S:L ratio from 1:1 to 4:1, the viscosity of the suspension decreases. When the S:L ratio changes from 1:1 to 4:1 and the temperature from 0 to 45 °C, an increase in the density of the formed suspensions is observed, which is in the range of 1.134 – 1.1224 g/sm$^3$. This is probably due to the characteristics of the constituent salts included in this system.

Table 2 shows the rheological properties of the schenite suspension.

**Figure 3.** *Change in suspension viscosity depending on process parameters*

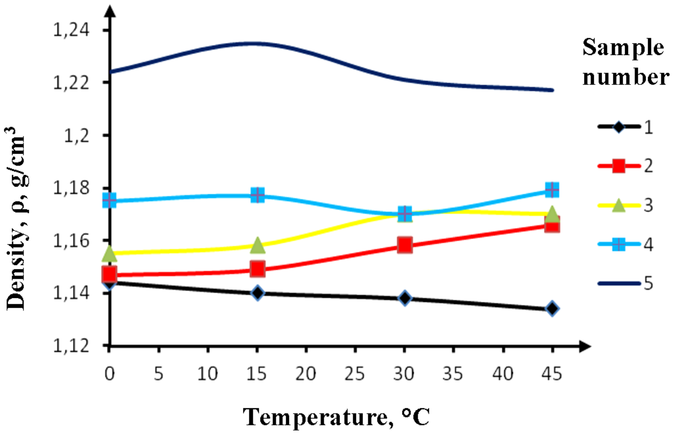


**Figure 4.** *Change in suspension viscosity depending on process parameters*

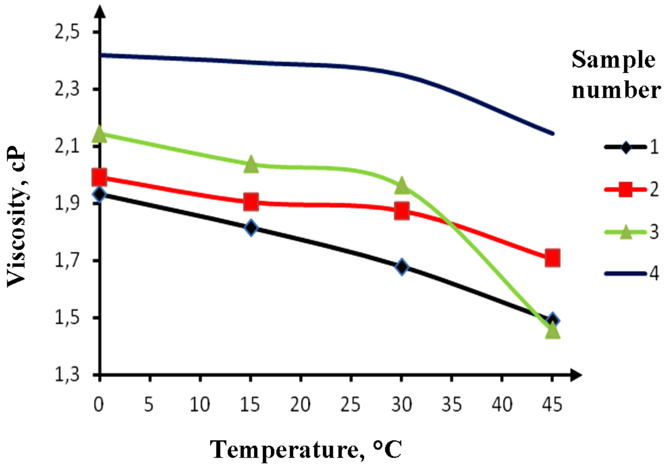


**Table 3.** *Filtration rate of glaserite and shenite suspensions, kg/($m^2$ . hour)*

| Sample numbers correspond to numbers table 1 | When using $Na_2SO_4$. | | | When using $MgSO_4$. | | |
|---|---|---|---|---|---|---|
| | By liquid phase | According to the solid phase | Solids moisture, % | By liquid phase | According to the solid phase | Solids moisture, %. |
| 2 | 1953.35 | 404.98 | 14.690 | 1940.10 | 580.81 | 11.72 |
| 3 | 2056.33 | 545.43 | 10.790 | 2037.51 | 802.65 | 12.61 |
| 4 | 1984.40 | 1021.16 | 16.601 | 2234.90 | 1186.20 | 10.56 |
| 5 | 1937.10 | 2165.80 | 7.720 | 2134.90 | 1969.25 | 7.23 |

Table 3 presents data on the filterability of glaserite and chenite suspensions.

As follows from the data obtained, within the studied limits of L:S variation, the filtration rate of glaserite suspensions in the liquid phase fluctuates in the ranges of 1937.10÷2056.33 kg/h · $m^2$, and in the solid phase, with an increase in the L:S ratio, an increase in the rate is observed filtration, ranging from 404.98–2165.80 kg/h · $m^2$. Precipitation humidity ranges from 7.72–16.60%.

When using chenite suspensions, the filterability in the liquid phase is in the range of 1940.10÷2234.90 kg/h · $m^2$. A similar pattern is observed in the solid phase: the filtration rate fluctuates in the ranges of 550.81÷1996.25 kg/h · $m^2$, the sediment moisture is on average 2.5–2.6% lower, and the filtration rate is on average 100–200 kg/h · $m^2$ more than in glaserite sediments.

## Conclusion

Thus, in order to provide practical recommendations for the production of potassium sulfate from sulfate salt deposits, we studied the rheological properties and filtration rate of suspensions that are formed during the complex processing of astrakhanite, glaserite and schenite with potassium chloride. It was established that changes in the density and viscosity of the formed suspensions depend on the L:S and the characteristics of the constituent salts of the system.

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# STUDY OF THE EFFECT OF $CO_2O_3$ AND CUO ADDITIVES ON GROUND ENAMEL OBTAINED IN THE $SIO_2$–$AL_2O_3$–$NA_2O$–$K_2O$–CAO–$B_2O_3$–NIO SYSTEM

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## Abstract

The purpose of this work is to study the effect of $Co_2O_3$ and CuO additives on the adhesion of ground coatings obtained in the $SiO_2$–$Al_2O_3$–$Na_2O$–$K_2O$–CaO–$B_2O_3$–NiO system. To achieve this goal, glasses were synthesized in the $SiO_2$–$Al_2O_3$–$Na_2O$–$K_2O$–CaO–$B_2O_3$–NiO system based on local raw materials with the addition of $Co_2O_3$ and Cu O. Ground enamels based on these glasses were obtained and their properties were studied. The obtained data show that the most important influence on the adhesion to the steel surface is exerted by copper, nickel and cobalt oxides. The conclusion is made about the predominant effect of copper oxide on the bond strength and about the positive effect of the combined presence of copper and cobalt oxides.

**Keywords:** *Adheison, ground enamel, enamel coatings, the system*

## Introduction

The adhesion of enamel to metal is one of the main characteristics of the metal-enamel system; it determines the stability of the system even before the product enters service. After firing enameled products, a firmly bonded coating is obtained on the metal. The practice of enameling, as well as a large number of research papers, has established the dependence of adhesion strength on a number of factors, such as stresses in the enamel layer, elasticity of enamel and metal, thickness of the enamel layer and other factors (Petzold, A., Peschmann, G., 1990).

The ground creates the best conditions for the adhesion of the coating enamel to the protected surface. The adhesion must be strong enough so that the coating layer cannot be separated from the metal by chemical or mechanical action. The adhesion strength is the force normally directed towards the substrate–enamel interface and necessary for the complete separation of the coating from the substrate over the entire test surface. However, a rupture can occur along one of the interface boundaries (adhesive separation), or along the intermediate layer material and even along the coating or substrate material (cohesive rupture). A mixed nature of the gap is also possible [4]. Therefore, after preliminary preparation of its surface, ground enamel is applied to the product, dried and fired at 850–900 °C, and then the cover enamel is applied in one or more layers with drying and subsequent firing at the same temperature (Bobkova, N. M., Dyatlova, E. N., Kunitskaya, T. S., 1987).

The adhesion of ground glass enamel to steel depends on the duration of contact of the melt with the surface of the substrate, i.e. on the firing time of the enameled product. The increase in adhesion over time has a limit. At a certain point, it stops and the further continuation of the firing becomes harmful. A thick layer of scale is formed under the coating layer which easily breaks off from the steel along with the enamel (Pashchenko, A.A., 1983).

The quality of the enamel coating significantly depends on the adhesion strength to the metal, therefore, it is desirable to enhance the adhesive properties of the ground with metal (Shimizu, T., Jiang, Z.H., Shirasak, M., 1998).

The adhesion strength of enamel to metal depends on several factors, such as the type of metal and enamel, coating thickness, application method, firing conditions, and others (Bragina, L.L., Zubekhin, A.P., 2003).

The strong adhesion of enamel to metal is ensured with adjustments of the thermal coefficient of linear expansion (TCLE) of metal and enamel. The TCLE of the enamel should be less than that of the metal, otherwise there will be a violation of the continuity of the layer. Tensile stresses occur during the cooling of the spread enamel (Kazmina, O.V., Elistratova, A.V., 2022).

To obtain high-quality enamel coatings, ground enamels must meet the following requirements: ensure good adhesion strength of the enamel layer to the metal, decompose iron oxides formed during the firing of ground coatings, contribute to the presence of iron in the melt in the form of divalent ions, have surface tension and viscosity to spread well, moisten oxidized metal and contribute to release of gases, have an insignificant thickness of the burnt coating (Technology of fibrous materials and coatings. 2014).

As a result of the study of multicomponent glass-enamel coatings on stainless steel, the prospects of creating coatings by introducing ions of alkaline earth elements with different ion radius into the borosilicate glass mesh (Novoselova, P.N., Semin, M.A., 2011) are shown.

A study of multicomponent enamels based on borosilicate glasses has been carried out (Ehrt, D., 2013; Topper, B., Möncke, D., Youngman, R.E., Valvi, Ch., Kamitsos, E.I., Christos, P.E. Varsamis, Ch.E.I., 2023).

The study of the dependence of the adhesion strength in the steel – glass enamel coating system on the specifics of the steel oxidation process. Temperature and time conditions of heat treatment is one of the most important factors in enameling, in particular 2C/1F technology. Since the enamel interacts with the metal during firing and forming a boundary zone that provides adhesion, the bond strength of the coating with the metal depends on the composition, structure of this zone and the firing temperature. The bonding process is associated with the formation of a double electric layer at the enamel boundary with an oxide film, not with pure metal. Oxygen plays an important role in the interaction processes, as well as the adhesion oxides included in the enamel, due to which electrochemical reactions occur at the phase interface, during which a transition layer is created that provides strong adhesion of enamel to metal (Soshina, T.O., Muxamadyarova, V.R., 2019; Yatsenko, E.A., 2010).

Based on the results of the study of the field of glass formation in the $K_2O$-RO-$B_2O_3$-$Al_2O_3$-$SiO_2$-$TiO_2$-$P_2O_5$-F system, the synthesis of single-layer glass-crystalline white enamel coatings was carried out. As a result of the research, a technology has been developed for the production of white glass-crystalline enamel coatings on household steel products with improved aesthetic and consumer properties (Klimova L.V., 2017).

The possibility of using glasses based on the basic $Na_2O$–CaO–$TiO_2$–$SiO_2$ system to obtain fluorine-free, non-porous ground coatings has been investigated. The optimal concentration of adhesion oxide (CoO) in the developed enamel compositions has been established. The influence of suspending materials on the characteristics of ground enamel coatings has been determined and their rational amount has been established. The obtained fluorine-free, boron-free ground coatings are recommended for production tests to use them in the enameling of steel products for household purposes (Bely, Ya. I., Kislichnaya, R. I., Nagornaya, T. I., Naumenko, S. Yu., Pavlova, K. V., 2013).

The composition of a complex adhesion promoter has been developed, containing a minimum amount of expensive CoO and NiO oxides and providing the necessary reactivity of the ground melt at a firing temperature of 800–820 °C due to the additional introduction of $MnO_2$ and $Fe_2O_3$. The specificity of the processes was revealed and the optimal conditions for the formation of the low–carbon steel system were established – a low-melting two-layer enamel coating of single firing at a temperature of 820 °C based on the developed soil, which contains a complex adhesion promoter in combination with white titanium enamel (Shalygina, O. V., 2006).

Today, to achieve saving it is customary to carry out single-layer enameling method to protect the metal surface from corrosion. However, single – layer enameling is not without drawbacks: The main focus that should be given to the mechanical properties is the adhesion of the glass-enamel coating to the metal, which is ensured by the oxides of Ni and Co. If these oxides are not present in the enamel, it is impossible to ensure adhesion during single-layer enameling. Ni and Co oxides should not be added to the coating enamel, since these oxides have a negative impact on human health. The advantage of double-layer enameling is the possibility of using Ni and Co oxides in the composition of the ground enamel, providing adhesion and using a coating enamel without the participation of these oxides.

Glasses were synthesized in the $SiO_2–Al_2O_3–Na_2O–K_2O–CaO–B_2O_3–NiO$ system. The physical and technical properties of synthesized glasses are determined. Compositions that meet the regulatory requirements for ground enamels have been identified. The possibility of synthesizing ground enamels for household metal products based on local raw materials has been proved (Naimov, Sh.B., Aripova, M. H., 2023).

The purpose of this work is to study the effect of $Co_2O_3$ and CuO additives on the adhesion of soil coatings obtained in the $SiO_2–Al_2O_3–Na_2O–K_2O–CaO–B_2O_3–NiO$ system.

## Materials and Methods

Chemical analysis of the raw materials and synthesized glasses was determined on an energy-dispersive X-ray fluorescence spectrometer Rigaku CG EDXRF (USA).

The structure of the ground coating was studied using a scanning electron microscope JEOL JSM–IT200LA (Japan).

Glass melting was carried out in a gas laboratory furnace in fireclay crucibles with a capacity of 250 ml at a temperature of 1350 °C with exposure for 1 hour. The melt was poured into water and frit granules were obtained.

The temperature coefficient of linear expansion is determined on BK – POL16. Qingdao Chengyu Testing Equipment Co., Ltd.

The ground coating on DC04EK steel was obtained in accordance with the procedure given in (GOST 52569–2018).

The spreadability and adhesion strength of the soil coating to the base were determined in accordance with the methodology given in (GOST 52569–20180.

For the synthesis of glasses in the $SiO_2–Al_2O_3–Na_2O–K_2O–CaO–B_2O_3–NiO$ system, the following raw materials were used: quartz sand from the Samarkand deposit; limestone of the Kashkadarya deposit; clay shale of the Jerdanak deposit (Table 1); technical soda ash GOST 5100–85; o.s.h. boric acid; pure CuO, pure NiO and Co2O3.

To obtain ground enamel, we used the addition of ch.o. grade fireproof clay.

**Table 1.** *Chemical composition of raw materials*

| Raw materials | Mass content of oxides,% | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $SiO_2$ | $Na_2O$ | $K_2O$ | CaO | $Al_2O_3$ | $B_2O_3$ | MgO | $Fe_2O_3$ | NiO | $Co_2O_3$ | CuO | п.п.п |
| Quartz sand | 92.5 | – | 1.03 | 0.271 | 3.51 | – | 0.04 | 0.087 | – | – | – | – |
| Limestone | 0.31 | – | 0.034 | 53.40 | 0.23 | – | 5.03 | 0.06 | – | – | – | 40.94 |
| Clay shale | 0.676 | – | 0.04 | 0.525 | 96.8 | – | – | 0.84 | – | – | – | 1.12 |

### Results

The chemical composition of the synthesized glasses is given in Table 2.

From the obtained data, it can be concluded that copper oxide has a predominant effect on adhesion strength and the positive effect of the combined presence of copper and cobalt oxides. Nickel content also affects the strength of the steel surface – the decrease in the amount of CuO is compensated by an increase in the NiO content (composition No. 3). Thus, we can conclude that the oxides of copper, nickel and cobalt have the most important influence on the amount of adhesion to a steel surface, which to a certain extent coincides with the results obtained by other researchers (Yatsenko, E.A., 2002; Chumakov, A.A., 2019; Yatsenko E. A., Dzyuba E. B., Veropakha N. V., 2012; Ken Chen, Minghui Chen, Fuhui Wang, Shenglong Zhu, 2017; Leila Samiee, Hossein Sarpoolaky, Alireza Mirhabibi, 2007).

**Table 2.** *Chemical composition of synthesized glass (frit)*

| Composition index | Mass content of oxides,% | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $SiO_2$ | $Al_2O_3$ | $Na_2O$ | $K_2O$ | CaO | $TiO_2$ | $P_2O_5$ | MgO | NiO | $Co_2O_3$ | CuO | $Fe_2O_3$ | $B_2O_3$ |
| № 1 | 43.4 | 8.12 | 23.5 | 0.6 | 7.61 | 0.05 | – | 0.118 | 0.807 | 0.324 | 0.974 | 0.114 | 14.2 |
| № 2 | 44.2 | 8.95 | 22.4 | 0.562 | 7.67 | 0.05 | – | 0.116 | 0.429 | – | 0.677 | 0.12 | 14.64 |
| № 3 | 44.0 | 7.67 | 25.5 | 0.555 | 8.1 | 0.05 | – | 0.113 | 0.750 | – | 0.356 | 0.1 | 12.73 |
| № 4 | 48.2 | 8.9 | 22.2 | 0.653 | 7.69 | 0.07 | – | 0.121 | 0.815 | 0.314 | 0.653 | 0.135 | 10.1 |
| № 5 | 54.0 | 4.89 | 23.9 | 0.794 | 7.84 | 0.141 | 0.671 | 0.122 | 0.704 | 0.214 | – | 0.132 | 6.8 |
| № 6 | 52.0 | 3.73 | 29.0 | 0.684 | 6.21 | 0.08 | 0.705 | 0.659 | 0.721 | 0.223 | – | 0.127 | 6.5 |
| № 7 | 47.9 | 4.14 | 23.3 | 0.851 | 7.82 | 0.144 | 1.31 | 0.128 | 0.712 | 0.168 | – | 0.11 | 13.58 |
| № 8 | 47.2 | 4.71 | 25.9 | 0.804 | 5.61 | 0.115 | 1.33 | 0.06 | 0.671 | 0.151 | – | 0.121 | 13.48 |
| № 9 | 42.3 | 5.53 | 25.7 | 0.615 | 5.64 | 0.06 | 0.681 | – | 0.665 | 0.202 | – | 0.113 | 18.5 |

To obtain a ground enamel coating, a slip was prepared from finely ground frit, quartz sand and refractory clay. The properties of the resulting ground enamels are given in Table 3.

**Table 3.** *Properties of ground enamels*

| Composition index | Temperature coefficient of linear expansion $\cdot 10^6$, $K^{-1}$ | Spreadability, mm | Strength of adhesion to steel surface |
|---|---|---|---|
| № 1 | 120 | 37 | 4,5 |
| № 2 | 117 | 30 | 4,0 |
| № 3 | 124 | 47 | 4,0 |
| № 4 | 115 | 37 | 4,5 |
| № 5 | 127 | 37 | 3,0 |
| № 6 | 140 | 45 | 3,5 |
| № 7 | 122 | 45 | 3 |
| № 8 | 129 | 44 | 2,5 |
| № 9 | 127 | 57 | 3 |

It can be noted that the predominant influence on the TCLE is exerted by the total content of oxides of alkali and alkaline earth metals and the total content of silicon and aluminum oxides. As can be seen from the data presented, compositions No. 1–4 and No. 7 meet the requirement for the value of thermal expansion coefficient of GOST 52569–2018. Therefore, we exclude compositions No. 6 and No. 8 from further studies.

**Table 4.** *Compliance of the resulting ground enamels with the flowability requirements*

| Spreadability, mm according to "GOST R52569–2018 FRITTS Technical specifications" | Composition index | Spreadability, mm |
|---|---|---|
| Relatively refractory, component, 25–40 mm | № 1 | 37 |
| | № 2 | 30 |
| | № 4 | 37 |
| | № 5 | 37 |
| Medium fusibility, component, 35–55 mm | № 7 | 45 |
| | № 3 | 47 |
| Relatively low-melting, component, 50–70 mm | № 9 | 57 |

It has been established that the synthesized compositions of ground enamel, when compared with the standard requirements correspond to the following groups in terms of spreadability: No. 1, 2, 4 and 5 are relatively refractory; No. 3, No. 6-No. 8 – to medium fusibility; No. 9 – to a relatively low-melting type. The latter result is easily explained by the high content of boron oxide in the composition of the ground enamel.

Figure 1 shows an electron microscopic image of ground enamel composition No. 1 on a steel surface. The intermediate layer that appeared at the contact between the soil and steel, which provided good adhesion, is clearly visible.

**Figure 1.** *Electron microscopic image of ground enamel composition No. 1 on a steel surface*

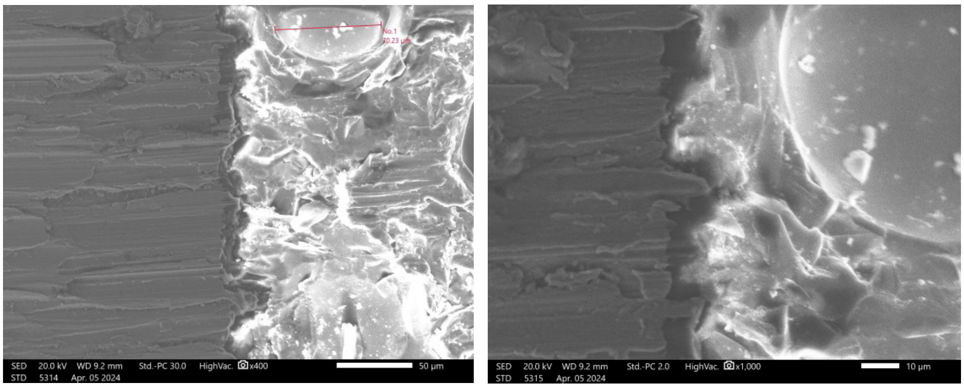
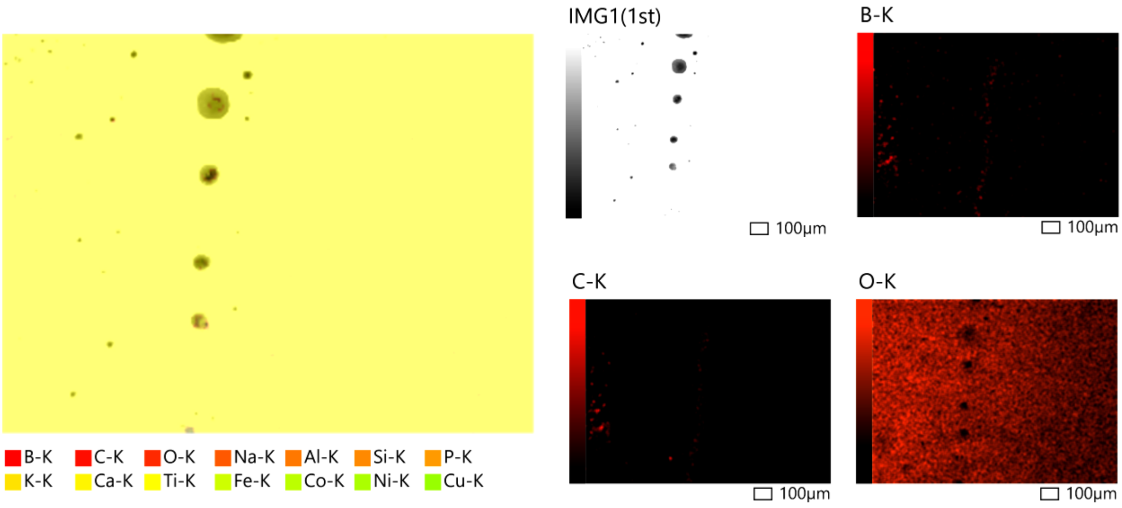
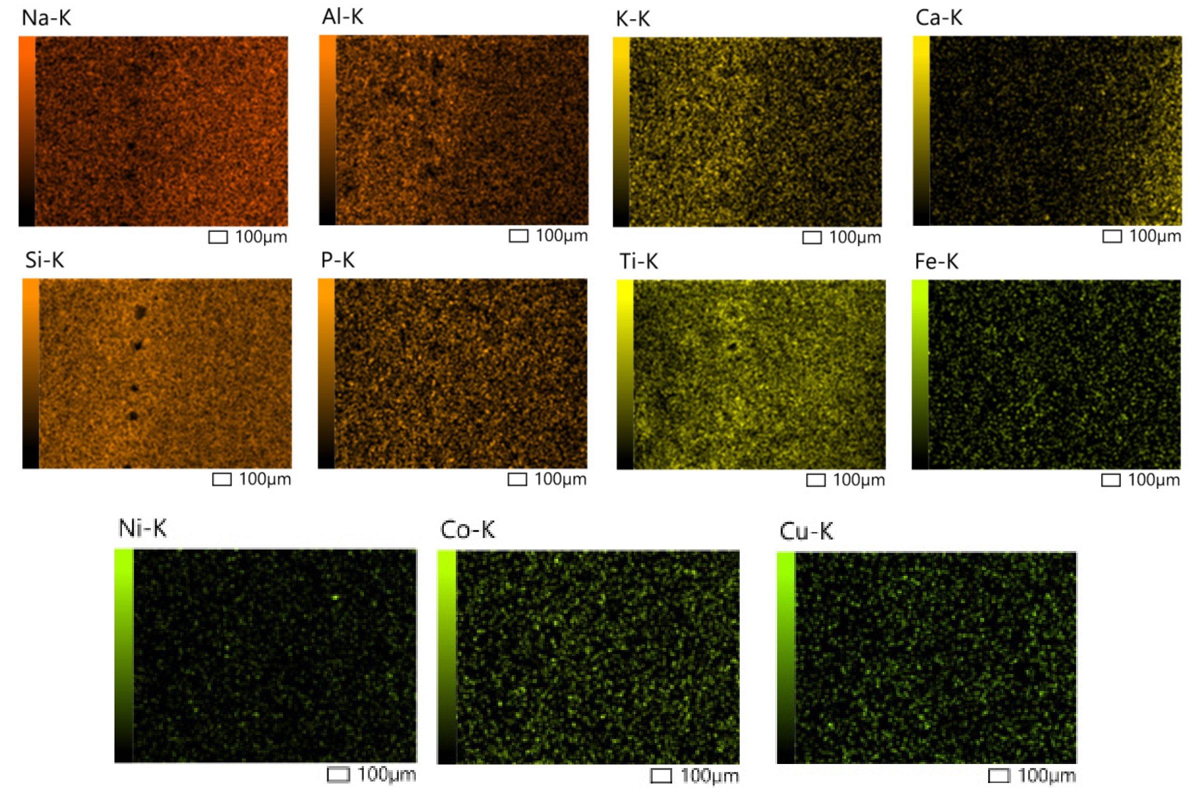
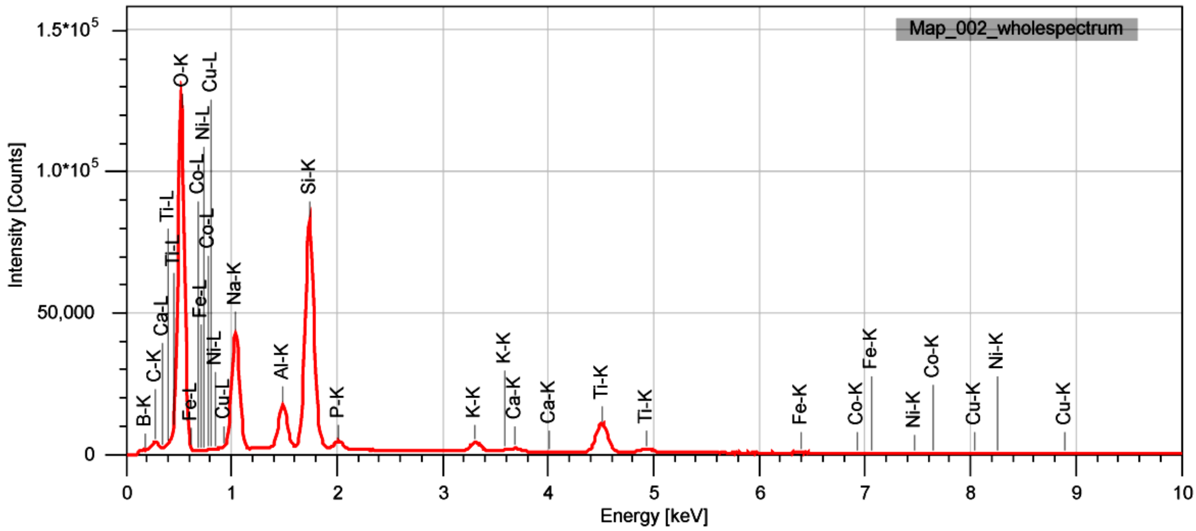


Figure 2 shows the results of EDS analysis of the surface layer of ground enamel

**Figure 2.** *EDS analysis of the surface layer of ground enamel*







EDS analysis of the soil surface showed that the soil elements are distributed predominantly evenly over the entire surface. The exception is boron, presumably due to enhanced penetration into the coating.

## Discussion

The synthesis of glasses in the $SiO_2$–$Al_2O_3$–$Na_2O$–$K_2O$–CaO–$B_2O_3$–NiO system based on local raw materials with the addition of $Co_2O_3$ and CuO and the production of ground enamels based on these glasses showed that copper, nickel and cobalt oxides have the most important influence on the amount of adhesion to a steel surface. It was concluded that copper oxide has a predominant effect on adhesion strength and the positive effect of the combined presence of copper and cobalt oxides.

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# CREATION OF A MODIFIED SOLID-PHASE MATRIX BASED ON GELATIN MEMBRANE FOR DETERMINATION METAL IONS

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**Abstract**

The immobilization of disodium salt of 4-hydroxy-3-(4-sulfonato-1-naphthyl- azo)-1-naphthalene sulfonate onto photographic membrane, the optimal conditions for the sorption of the reagent and the process of complexation on a layer of gelatin membrane were investigated. A new solid-phase spectrophotometric method for determination copper ions in model mixtures simulating real objects has been proposed and metrologically improved results have been obtained.

**Keywords:** *solid-phase spectrophotometry, gelatin membrane, organic reagent, optical density*

## Introduction

Expanding the range of solid-phase reagents is a necessary condition for the delaboration of new methods of chemical analysis. Solid-phase reagents are obtained mainly by immobilization of reagents or fixing of it's functional analytical groups on the surface of various sorbents. The analysis procedure using a solid-phase reagent involves the extraction of the analyte into the modified sorbent phase due to it's interaction with the modifier-reagent, i.e. carrying out solid-phase extraction and further determination of the isolated component directly in the sorbent phase or in the eluate after its desorption. Concentration and separation of analytes can significantly to increase the sensitivity and selectivity of analytical determinations; This important advantage of solid-phase reagents has found application in diffuse reflectance spectroscopy, colorimetry, solid-phase spectrophotometry, fluorimetry, and visual test analysis (Zolotov, Yu.A., et al., 2007; Moliner-Martinez, Y., et al., 2005; Zaporozhets, O.A., et al., 2005; Ngeontaea, W., et al., 2007). Specific interest are solid-phase reagents (indicator agents) presented for monitoring of integral (generalized) indicators that make it possible to quickly assess the quality of natsural, technical, waste and drinking waters and other objects.

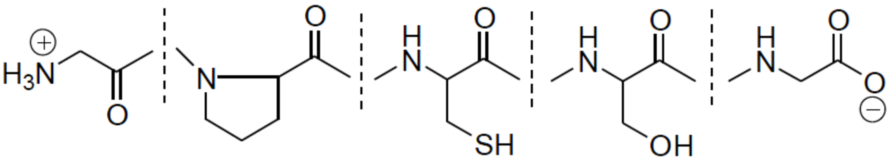
Solid-phase reagents are also used as indicators and test systems for determination of total acidity and  hardness, total content of metals, phenols, nitrites and nitrates, Fe(II, III), reducing agents, polycyclic aromatic hydrocarbons, aromatic amines and pesticides, active chlorine, iodine, total antioxidant activity, and phosphorus content, chemical and biological oxygen consumption (Zaitsev, V.N. 1997; Tikhomirova, T.I., et al., 2011; Dmitrienko, S.G., et al., 2010; Dini, F., et al., 2011; Kim, H.N., et al., 2011; Amelin, V.G., et al., 2011; Takagai, Y., 2010; Vilozny, B., et al., 2011; Boling, M. et al.,  2010; Savvin, S.B., et al., 2008; Dedkova, V.P., et al., 2011; Nan, Wu, et al., 2009; Comes, M., et al., 2008; Koncki, R., et al., 2001; Mohr, G.J., et al., 1999; Descalzo, A.B., et al., 2007).

For these same purposes in some cases (Nicole, L., et al., 2004; Amelin, V.G., et al., 2001; Choi, M.M.F., et al., 2001; Hashemi, P.A., et al., 2008; Sadananda, D., et al., 2011; Jiang, J., et al., 2009; Fries, K., et al., 2008; Dedkova, V.P., et al., 2010; Amelin, V.G., et al., 2009; Li, C.M., et al., 2009) gelatin membrane, hardened gelatin gel and other matrices are used.

**Gelatin membrane.** In addition to transparent synthetic polymer matrices, membranes based on the biological polymer gelatin are widely used in modern analytical chemistry as suitable means for the immobilization of analytical reagents.

By it's chemical nature gelatin is considered as polydispersion submolecular polypeptide (Fig. 1.), consisting of amina ocid residues, from which, on average, more than 30% are bound on the basis glycine (El-Sheikh, A.H., Al-Degs, Y.S. 2013).

**Figure 1.** *Fragment of a gelatin molecule*



Gelatin is a fairly hydrophilic polymer. At gelatin action on aqueous solution, in it's mass, along with the solvent, dissolved chemical also are included. The penetration of water into the gelatin mass causes its isotropic swelling. Hydration of gelatin is achieved primarily through the formation of hydrogen bonds with water molecules. Hydration capacity is also provided by many anionic and cationic groups containing in gelatin macromolecules (Dinç, E., et al., 2002; Sahraei, R., et al., 2013).

In this work by immobilizing reagents in the gelatin layer of photographic membranes, solid-phase reagents were obtained for monitoring of various parameters of environmental objects. Gelatin layers of Agfa photographic membranes were used as a sorbent for the immobilization of HR for the determination of copper ions.

According to date of (Sahraei, R., et al., 2013; Kaur, A.D., Gupta, U. 2012; Unsal, Y.E., et al., 2012) spectrophotometric determinations with using of complexing and redox solid-phase reagents based on gelatin membranes, the sensitivity, they can be compared with standard methods of spectrophotometric analysis.

## Experimental part

**Reagents and solutions.** $9.95 \cdot 10^{-4}$M (0.05%) aqueous solution of disodium salt of 4-hydroxy-3-(4-sulfonato-1-naphthyl-azo)-1-naphthalene sulfonate (HR) and $1.574 \cdot 10^{-2}$ M copper(II) solutions, prepared from metallic copper, a universal Britton-Robinson buffer solution was prepared by mixing $H_3BO_3$, $CH_3COOH$ and $H_3PO_4$ at 0.04 M each and adding 0.2 M NaOH to achieve the appropriate pH value (Lurie, Yu.Yu. 1989) we used.

Matrices from photographic membranes were prepared by removing silver salts from the gelatin layer by using solutions of a special reagent (Razzoqova, S., et al., 2024; Khaitov, J.K., et al., 2024; Yunusov, Kh.E., et al., 2022).

**Equipment.** Absorption spectrums were recorded on a UV-VIS 500 spectrophotometer, and the optical density (OD) of solutions was measured on a KFK-3 concentration photocolorimeter. The pH of solutions was controlled by pH/ISE Meter pH meter with an accuracy of ± 0.05 units. A universal buffer solution was used to maintain constant pH (5.87–6.11). Based on the experimental data, pH = 5.95 was chosen as optimal.

## Results and its discussion

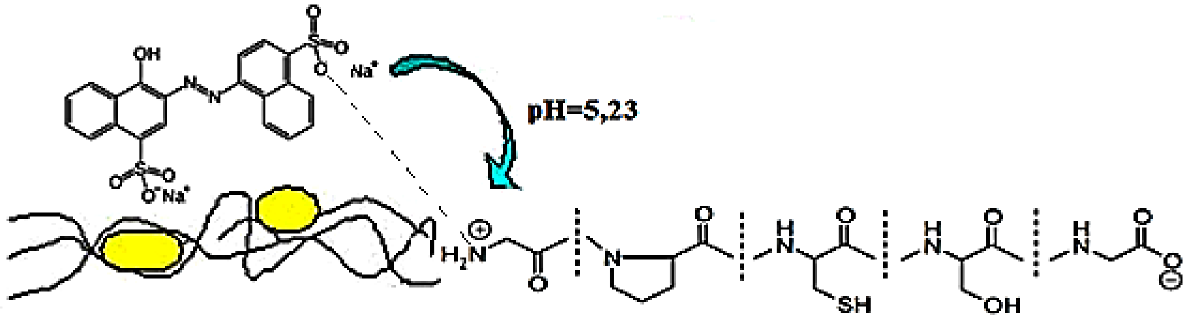
The research results were studied by methods of physico-chemical analysis (photometry, spectrophotometry, solid-phase spectrophotometry).

**Determination of the amount of immobilized reagent:** For determination the amount of immobilized reagent on the matrix layer 0.05% solutions of HR were prepared and their concentration was calculated in mol/l. Comparative solutions of various concentrations were placed in a 25 ml volumetric flask, 5.0 ml of a universal buffer solution with a predetermined pH value 5.23 was poured on top, by double-distilled water was added to the mark and mixed. Then the optical density (OD) of the resulting solutions was measured on a UV-VIS 500 spectrophotometer in order of increasing concentration according to Table 1. Then the same solutions were placed in a glass beaker with a capacity of 50 ml, pre-cleaned photographic membranes with dimensions of 2 × 0.5 cm were placed and left for certain time. After 40 minutes the membranes were removed from the solution, washed with bidistilled water and dried at room temperature in air, after which the OD of the solution was determined in a glass beaker. The amount of immobilized reagent on the photographic membrane (Im-HR) was calculated based on the results shown in Table 1.

**Table 1.** *Results of calculating the amount of reagent immobilized on the membrane (pH =5.23, l = 520 nm, ℓ = 0.10 cm, n-3)*

| No. | $V_{HR,}$ ml | $C_{HR}\cdot 10^{-4}$ mol/l | A, prior to immobilization | À, after immobilization | $\Delta\bar{A}$ | $C_{HR}\cdot 10^{-4}$ mol/l after immobilization | $C_{Im\text{-}HR}\cdot 10^{-4}$ mol/l |
|---|---|---|---|---|---|---|---|
| 1 | 2.2 | 0.876 | 1.8802 | 1.7611 | 0.1191 | 0.7569 | 0.1191 |
| 2 | 2.4 | 0.9552 | 1.9136 | 1.7897 | 0.1239 | 0.8311 | 0.1239 |
| 3 | 2.6 | 1.035 | 1.9790 | 1.8512 | 0.1243 | 0.9107 | 0.1243 |
| 4 | 2.8 | 1.114 | 1.9920 | 1.8627 | 0.1293 | 0.9847 | 0.1293 |
| 5 | 3.0 | 1.194 | 2.0091 | 1.8393 | 0.1696 | 1.0244 | 0.1696 |
| 6 | 3.2 | 1.274 | 2.4362 | 2.2516 | 0.1846 | 1.0894 | 0.1846 |
| 7 | 3.6 | 1.432 | 2.7440 | 2.4831 | 0.2609 | 1.1721 | 0.2609 |
| 8 | 4.0 | 1.592 | 2.9160 | 2.6531 | 0.2629 | 1.3291 | 0.2629 |
| 9 | 4.5 | 1.791 | 2.9391 | 2.6730 | 0.2659 | 1.5251 | 0.2659 |
| 10 | 5.0 | 1.99 | 2.9630 | 2.6845 | 0.2785 | 1.7115 | 0.2785 |
| 11 | 6.0 | 2.388 | 2.9810 | 2.7005 | 0.2805 | 2.1075 | 0.2805 |
| 12 | 6.5 | 2.587 | 2.9980 | 2.7125 | 0.2855 | 2.3015 | 0.2855 |
| 13 | 7.5 | 2.985 | 3.0120 | 2.7236 | 0.2884 | 2.6936 | 0.2914 |
| 14 | 8.0 | 3.184 | 3.1422 | 2.8531 | 0.2896 | 2.8944 | 0.2896 |
| 15 | 8.5 | 3.383 | 3.3037 | 3.0139 | 0.2898 | 3.0932 | 0.2898 |
| 16 | 9 | 3.582 | 3.7377 | 3.4252 | 0.2925 | 3.2825 | 0.2995 |
| 17 | 10 | 3.98 | 3.8643 | 3.5660 | 0.2987 | 3.6727 | 0.3073 |

**Figure 2.** *Proposed scheme for HR immobilization onto gelatin membrane layer*



From (Fig. 1) it is clear that gelatin molecules are highly anisotropic and asymmetric. The polymer has an infinite network structure consisting from long chains of a limited number of interconnected molecules. The stabilization of this structure is formed mainly by hydrogen bonds and Van der Waals interactions. Gelatin is a typical ampholyte. The isoelectric point (pI) of alkaline gelatin is in the pH range 4.8–5.1, and the pI value of acidic gelatin is in the pH range 7.0-9.5. At such acidity all the main groups of gelatin become positively charged, while most of the carboxyl groups are overdissociated and have the same number of negative charge (Razzoqova, S., et al., 2024).

**Study of the dependence of HR immobilization and copper(II) complexation with Imm-HR on the pH medium of the solution.** Considering that one of the important conditions for the immobilization is the dissolution medium, universal buffer solutions with different pH values were prepared to select the optimal conditions for the immobilization of the reagent and the complexation of Cu(II) with HR. A 25 ml volumetric flask was filled with 1.0 ml of a 0.5% HR solution, with 5.0 ml of a universal buffer solutions with different pH values, filled to the mark with double-distilled water and mixed. The resulting solution was placed in a glass beaker with a volume 50 ml and the membrane was immersed in it. After 40 minutes the membrane was removed from the solution, washed with double-distilled water and dried at room temperature. The OD of the immobilized membrane was measured in various mediums (pH) in comparison with the immobilized membrane on KFK-3 at $\lambda_{max}$=520 nm, ℓ=0.10 cm. The results obtained are presented in (Table 2 and Figure 3).

**Table 2.** *Immobilization of HR on a layer of photographic membrane depending on the pH medium ($\lambda_{max}$ = 520 nm, ℓ = 0.10 cm, n = 3)*

| | | | | | | |
|---|---|---|---|---|---|---|
| pH | 3.05 | 3.44 | 3.95 | 4.23 | 4.83 | 5.05 |
| A | 0.021 | 0.056 | 0.099 | 0.119 | 0.135 | 0.177 |
| pH | 5.23 | 5.48 | 5.65 | 5.95 | 6.05 | 6.22 |
| A | 0.182 | 0.176 | 0.151 | 0.144 | 0.076 | 0.044 |

**Table 3.** *Immobilization of Cu(II) complex with HR on a layer of photographic membrane depending on the pH medium ($\lambda_{max}$ = 630 nm, ℓ = 0.10 cm, n = s3)*

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| pH | 3.05 | 3.25 | 3.66 | 3.95 | 4.23 | 4.54 | 4.83 | 5.05 | 5.95 |
| A | 0.034 | 0.072 | 0.146 | 0.164 | 0.158 | 0.131 | 0.077 | 0.034 | 0.017 |

**Figure 3.** *Study of HR immobilization on a layer of photographic membrane depending on the pH medium*

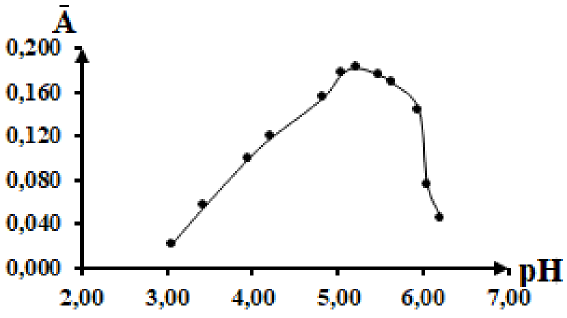
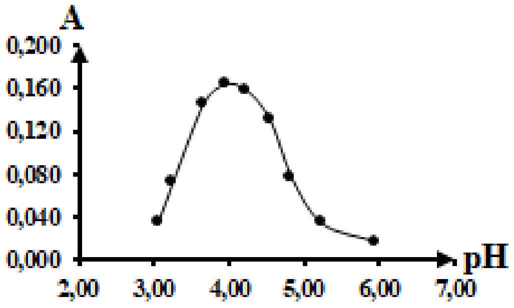


**Figure 4.** *Immobilization of the Cu(II) complex with HR on a layer of photographic membrane depending on the pH medium*



In 25 ml volumetric flask 1.0 ml of Cu(II) solution with a concentration of 100.0 µg/ml and 5.0 ml of a universal buffer solution with different pH were added, the flask was filled to the mark with double-distilled water and mixed. The resulting solution was placed in a glass beaker with a volume of 50 ml and the immobilized membrane was immersed in it. After 30 minutes the membrane was removed and washed with double-distilled ion water. The OD of the complex of the immobilized membrane with the Cu(II) ions in comparison with the immobilized membrane was measured at KFK-3 ($\lambda_{max}$ = 630 nm, ℓ = 0.10 cm. The results obtained are presented in (Table 3 and ion Figure 4).

**Determination of stability of HR immobilized on photographic membrane and it's complex with Cu(II), in time.** To determine the stability of the immobilized HR and obtain reproducible results, the stability of the optical density of the immobilized membrane over time was studied.

**The methodology of work.** 1.0 ml of 0.5% HR solution was added to a measuring flask with a capacity of 25 ml, 5.0 ml of a universal buffer solution pH = 5.23 was added and the flask was filled to the mark with bidistilled water,and was mixed. The resulting solution was placed in a glass beaker with capacity 50 ml and the membrane was statically immersed in it. After a certain time the membrane was removed from the solution, washed with distilled water and dried at room temperature. Thus, the OD of the immobilized membrane compared to the pure membrane was measured in KFK-3 at λ = 520 nm, ℓ = s0.10 cm. The results are presented in (Table 4 and on Figure 5).

**Table 4.** *Stability of the immobilization of the reagent on the photographic membrane over time (HR–0.5%, pH =5.23, l = 520 nm ℓ = 0.10 sm, n = 3)*

| **$t_{min}$** | **5** | **10** | **15** | **20** | **25** | **30** | **35** | **40** | **45** | **50** | **55** |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **A** | 0.123 | 0.045 | 0.058 | 0.087 | 0.111 | 0.132 | 0.152 | 0.182 | 0.182 | 0.182 | 0.182 |
| **$t_{min}$** | **60** | **65** | **70** | **75** | **80** | **85** | **90** | **95** | **100** | **105** | **110** |
| **A** | 0.182 | 0.182 | 0.182 | 0.182 | 0.182 | 0.182 | 0.182 | 0.182 | 0.182 | 0.182 | 0.182 |

In order to obtain reproducible results at the determination of copper (II) with HR, the stability of the OD solution of a complex compound formed on a layer of immobilized membrane was studied.

**A method for determining the stability of a complex formed on an immobilized membrane over time:** 1.0 ml of Cu(II) ion solution with a concentration of 50.0 mkg/ml, 5.0 ml of universal buffer solution with pH = 5.23 were added to a 25 ml volumetric flask, then the flask was filled with bidistilled water to the mark and mixed.

**Figure 5.** *Time stability of the copper(II) complex formed on the immobilized membrane with HR (Cu(II) = 50 mkg, HR–0.05%, pH = 3.95 ($\lambda_{max}$= 630, ℓ = 0.10 nm, n = 3)*

| **$t_{min}$** | **5** | **10** | **15** | **20** | **25** | **30** | **35** | **40** | **45** |
|---|---|---|---|---|---|---|---|---|---|
| A | 0.0026 | 0.053 | 0.083 | 0.112 | 0.141 | 0.164 | 0.164 | 0.164 | 0.164 |
| **$t_{min}$** | **50** | **55** | **60** | **65** | **70** | **75** | **80** | **85** | **90** |
| A | 0.164 | 0.164 | 0.164 | 0.164 | 0.164 | 0.164 | 0.164 | 0.164 | 0.164 |

**Figure 5.** *Shows the dependence of the stability of the immobilization of the reagentHR on membrane from time*

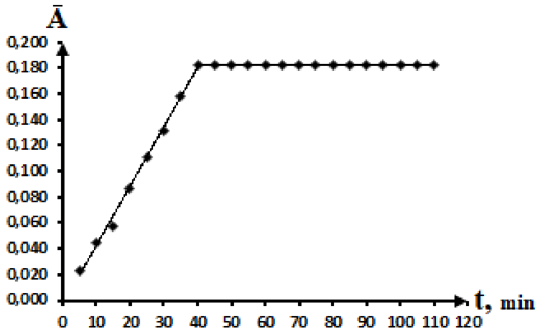
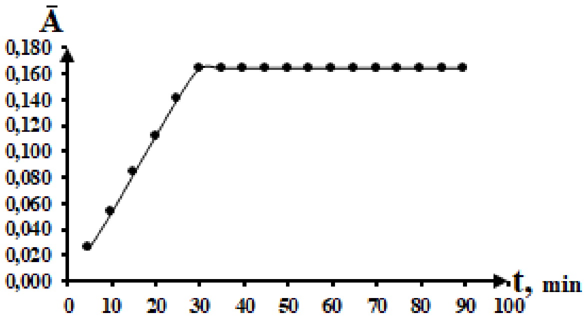


**Figure 6.** *Stability of the copper(II) complex formed on an immobilized membrane with HR from time*



The resulting solution was placed in a glass beaker with a capacity of 50 ml and a membrane immobilized with HR was immersed in it. In this order, the OD of the complex was measured, compared with the immobilized membrane on KFK-3 at λ = 630 nm, ℓ = 0.10 sm. The results are presented in Table 5 and on (Figure 6).

The results of (Table 2.19 and Figure 2.12), as well as (Table 2.20 and Figure 2.13), have shown that the immobilization of the HR reagent on the membrane increased to 40 minutes and remained unchanged for thirty days or more. The results show that the OD of the complex formed on the immobilized membrane with HR has increased to 30 minutes and then remains unchanged for several days (even more than a month). This allows to get reproducible results.

**Determination of the optimal absorption wavelength of Imm-HR and it's complex with copper (II) on the memrane.**

Considering that each process molecule absorbs light of a certain wavelength during immobilizated , the immobilization of HR on the film and it's complex formed with copper(II) ions is carried out according to the method (Moliner-Martinez, Y., et. al., 2005).

**The methodology of work:**

1.0 ml of 0.5% HR solution was placed in 25 ml volumetric flask, 5.0 ml of a universal buffer solution with pH = 5.23 was poured, the flask was filled to the mark with bidistilled water and shaken. The resulting solution was placed in 50 ml glass beaker and the membrane was immersed in it.

Through 30 minutes the membrane was removed from the solution, washed with bidistilled water and dried at room temperature and the OD was measured at the optimal wavelength. The results are presented in (Table 6 and on Figure 7).

**Table 6.** *The results of choosing the optimal absorption wavelength of a membrane immobilized by HR in comparation with pure membrane ($C_{HR}$– 0.5%; pH = 5.23; ℓ = 0.10; n = 3)*

| **λ, nm** | **320** | **330** | **340** | **350** | **360** | **370** | **380** | **390** | **400** |
|---|---|---|---|---|---|---|---|---|---|
| **A** | 0.033 | 0.046 | 0.05 | 0.052 | 0.054 | 0.052 | 0.05 | 0.048 | 0.046 |
| **λ, nm** | **410** | **420** | **430** | **440** | **450** | **460** | **470** | **480** | **490** |
| **A** | 0.045 | 0.042 | 0.042 | 0.043 | 0.051 | 0.072 | 0.093 | 0.119 | 0.143 |
| **λ, nm** | **500** | **510** | **520** | **530** | **540** | **550** | **560** | **570** | **580** |
| **A** | 0.161 | 0.177 | 0.182 | 0.172 | 0.152 | 0.13 | 0.111 | 0.077 | 0.029 |

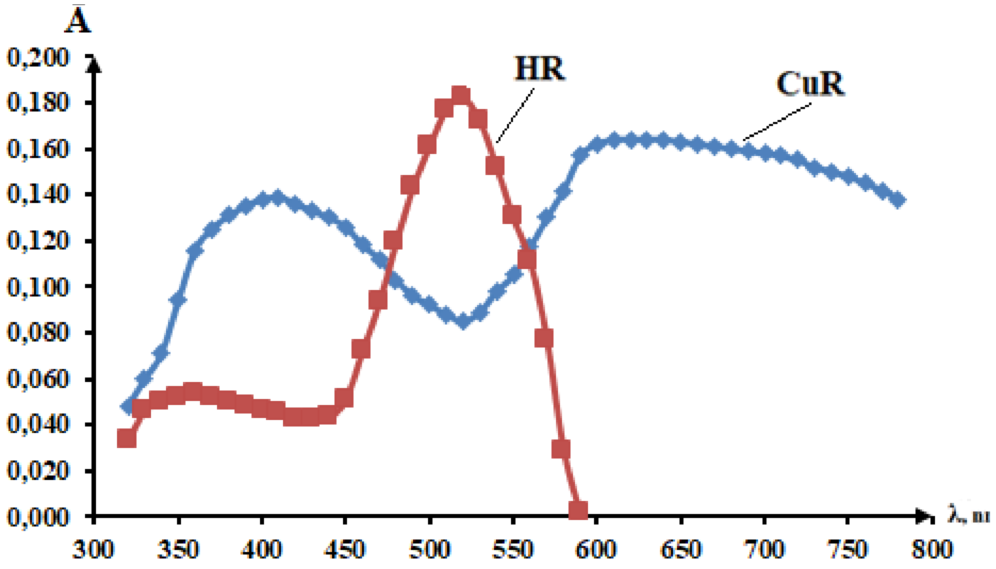
**A method for determining the optimal absorption wavelength of a copper(II) complex on an immobilized membranbe:** 1.0 ml of Cu(II) solution with a concentration of 50.0 micrograms/ml, 5.0 ml of a universal buffer solution with pH = 3.95 were added to a measuring flask with a capacity of 25 ml and distilled water was added to the mark on the flask. The resulting solution was placed in a 50 ml glass and an immobilized membrane was immersed in it. After 30 minutes, the membrane was removed from the solution, dried in air and OD was measured in various light filters. As a comparison, a membrane immobilized by HR only without metal was used. The measurement results are presented in (Table 7 and Figure 7).

**Table 7.** *The results of determining the optimal absorption wavelength for the copper(II) complex with HR on an immobilized memebrane ($C_{HR}$–0.5%; Cu(II)–50 мкг; pH = 3.95; ℓ = 0.10; n = 3)*

| **λ, nm** | **320** | **330** | **340** | **350** | **360** | **370** | **380** | **390** | **400** |
|---|---|---|---|---|---|---|---|---|---|
| A | 0.048 | 0.06 | 0.071 | 0.094 | 0.116 | 0.125 | 0.131 | 0.135 | 0.138 |
| **λ, nm** | **410** | **420** | **430** | **440** | **450** | **460** | **470** | **480** | **490** |
| A | 0.139 | 0.136 | 0.133 | 0.13 | 0.126 | 0.118 | 0.112 | 0.103 | 0.096 |
| **λ, nm** | **500** | **510** | **520** | **530** | **540** | **550** | **560** | **570** | **580** |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| A | 0.092 | 0.088 | 0.085 | 0.089 | 0.098 | 0.105 | 0.117 | 0.13 | 0.142 |
| **λ, nm** | **590** | **600** | **610** | **620** | **630** | **640** | **650** | **660** | **670** |
| A | 0.157 | 0.162 | 0.164 | 0.164 | 0.164 | 0.164 | 0.163 | 0.162 | 0.161 |
| **λ, nm** | **680** | **6900** | **700** | **710** | **720** | **730** | **740** | **750** | **760** |
| A | 0.16 | 0.159 | 0.158 | 0.157 | 0.155 | 0.152 | 0.15 | 0.148 | 0.145 |

**Figure 7.** *Results of choosing the optimal absorption wavelength of the immobilized HR and it's Cu(II) complex on the membrane*



It can be seen from the results obtained that the immobilization of the membrane with a reagent obtained at pH = 5.23 and absorption compared to a pure membrane showed the highest optical densitys at $\lambda_{max}$ = 520 nm. It was found that the maximum light absorption of the copper(II) complex on the immobilized membrane is observed at pH = 3.95 and has the highest optical density at $\lambda_{max}$ = 630 nm.

**The study of the subordination to the Booger-Lambert-Beer law of the Cu(II) whiz HR complex immobilized on membrane.**

The behavior of the solution of the complex formed on an immobilized membrane with HR was studied under selected optimal conditions.

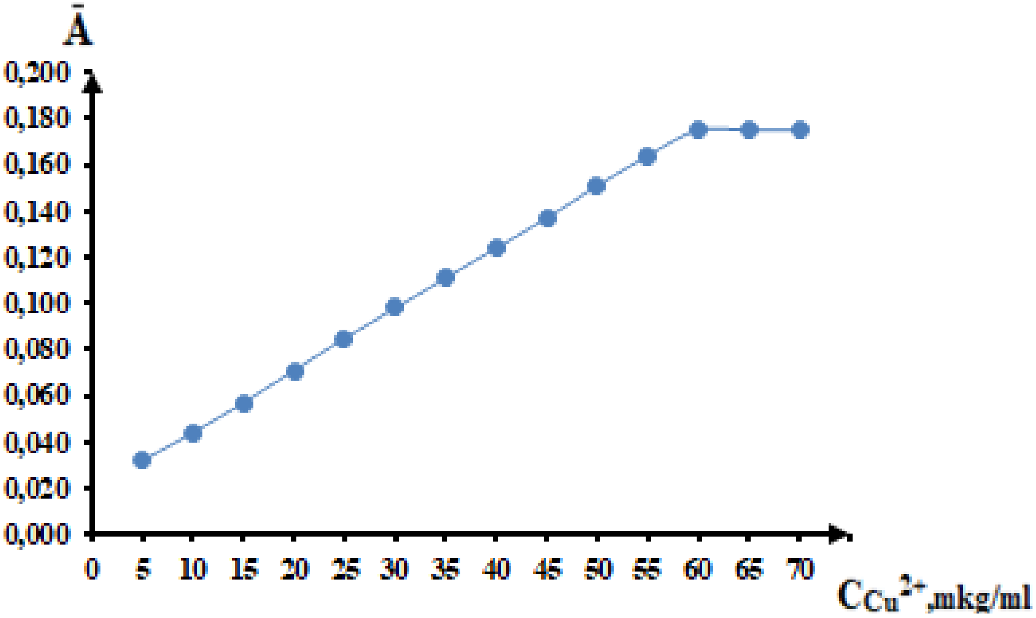
**Working methods:** A Cu(II) solution with a concentration of 50 mkg/ml in various quantities was placed in a measuring flask with a capacity of 25.0 ml, 5.0 ml of a universal buffer solution with a pH of 3.95 was poured, supplemented with bidistilled water to the label of the flask, poured into a glass beaker, the immobilized membrane was immersed in the same solution, after 30 minutes the membrane was removed from the solution, They were washed with distilled water and dried at room temperature in air, then the membrane OD was measured on KFK-3, the light absorption thickness was ℓ =0.10 sm in a light filter with $\lambda_{max}$ = 630 nm. The results are presented in (Table 8 and on Figure 8).

**Table 8.** *Investigation of the subjection to the Buger-Lambert-Ber law of the Cu(II) c HR complex immobilized on membrane ($C_{HR}$– 0.5%; pH = 3.95; ℓ = 0.10; n = 3)*

| No. | Aliquot part, ml | The amount of Cu(II) in the aliquot, mkg | $A_{comp}$ |
|---|---|---|---|
| 1 | 0.5 | 5.0 | 0.032 |
| 2 | 1.0 | 10.0 | 0.044 |
| 3 | 1.5 | 15.0 | 0.057 |
| 4 | 2.0 | 20.0 | 0.071 |
| 5 | 2.5 | 25.0 | 0.085 |

| No. | Aliquot part, ml | The amount of Cu(II) in the aliquot, mkg | $A_{comp}$ |
|---|---|---|---|
| 6 | 3.0 | 30.0 | 0.098 |
| 7 | 3.5 | 35.0 | 0.111 |
| 8 | 4.0 | 40.0 | 0.124 |
| 9 | 4.5 | 45.0 | 0.137 |
| 10 | 5.0 | 50.0 | 0.151 |
| 11 | 5.5 | 55 | 0.164 |
| 12 | 6.0 | 60 | 0.175 |
| 13 | 7.0 | 65 | 0.175 |
| 14 | 7.5 | 70 | 0.175 |

**Figure 8.** *The dependence of the optical density on the amount of added* $Cu^{2+}$



It can be seen from the results obtained that the area of subordination to the Buger-Lambert-Ber law was observed in the range of 5.0-55 micrograms of Cu(II) in 25.0 ml solution. At a higher concentration a deviation from the linear dependence was observed.

**Study of the spectral characteristics of the copper(II) complex with HR immobilized on membrane.**

The absorption spectrum of the copper(II) complex formed on the immobilized membrane with HR were measured under selected optimal conditions.

**Working methods:** standard Cu(II) solutions with a concentration of 50.0 micrograms/ml, 5.0 ml of a universal buffer solution with a pH of 3.95 and bidistilled water to the label of the flask were added to measuring flasks with a capacity of 25.0 ml. The resulting solution was placed in a 50 ml glass and a membrane immobilized with HR was immersed, after 30 minutes it was washed with distilled water and dried in air. The absorption spectra of a membrane immobilized by the reagent were compared with a membrane without immobilization. The results are presented on (Fig. 9 and in Table 9). According to the absorption spectrum shown on (Fig. 9), the maximum absorption region of the HR reagent in the membrane is located at λ = 520 nm, and the maximum absorption region of the copper complex in the membrane was observed at λ = 630 nm. The Mendelian sensitivity of the method was calculated using the formula:

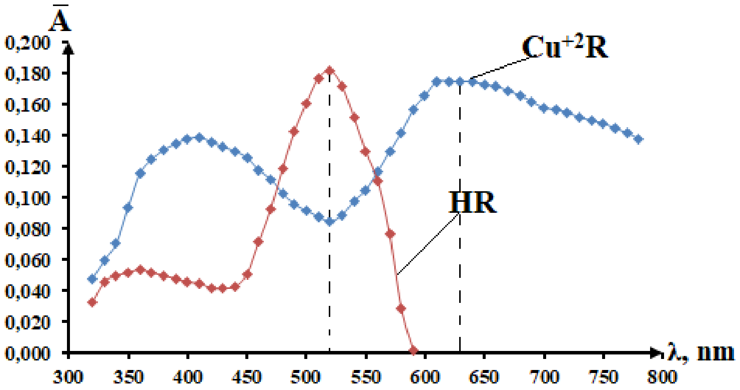
$$S_{s.b.s} = \frac{Q \cdot \ell \cdot 0.001}{A \cdot 25} = \frac{50 \cdot 0.10 \cdot 0.001}{0.175 \cdot 25} = 0.00114\, mkg / sm^2$$

**Table 9.** *Spectral description of HR and it's complex with copper (II) (Cu(II)–50 mkg/ml, ℓ = 3.0 cm, $\lambda_{max}$ = 600 nm, pH = 8.40, n = 3*

| Colur | pH (HR) | pH (MeR) | $\lambda_{max}$, nm MeR | HR | Δλ | $C_{Cu(II)}$ mkg/ 25 ml | $C_{Cu(II)}$, mol/l | A | Sendel sensitivity of mkg/sm² |
|---|---|---|---|---|---|---|---|---|---|
| Reddish -brown | 5.23 | 3.95 | 630 | 520 | 110 | 50 | $3.147 \cdot 10^{-5}$ | 0.175 | 0.00114 |

It was determined that the sensitivity of the delaborated method is equal to 0.00114 mkg/cm².

**Figure 9.** *Absorption spectrum of the HR reagent and it's complex with copper(II)*



As can be seen from the results obtained, the contrast ratio of the reaction is 110 nm and the sensitivity of the Sendel sensivity 0.00114 mkg/ sm² respectively, which indicates on the high sensitivity of the delaborated method.

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# REGULATION OF THE PROPERTIES OF HYDRATION STRUCTURES IN CLAY GYPSUM COMPOSITIONS

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## Abstract

The purpose of this study was to study the properties of various binders based on gypsum rocks of the Ustyurt deposit. The ways of regulating the properties of various gypsum binders using additives and rational use of natural gypsum for the production of a low-temperature binder with a gypsum type of hardening and a high-temperature gypsum binder for rocks with a predominance of the clay-carbonate part are determined.

The processes of thermal decomposition of gypsum in the temperature range of 100––1000 °C are considered and its modeling compositions, the phase composition of the products formed, the basic patterns of structure formation depending on the phase composition of the initial binder are established. Heat treatment of clay gypsum (100–1000 °C) allows you to change the nature of the structure-forming component, including the sulfate or clay part in the hydration process

**Keywords:** *Hydration, structure formation, clay-gypsum, additive, strength, roasting, solution, heat treatment, alumina, gypsum binder, surfactants*

## Introduction:

Scientific and practical results have been achieved in the Republic in obtaining new local materials and directional changes in the properties of existing compositions.

Karakalpakstan is a republic of a country remote from industrialized regions. There is no production of cement and surfactants, and their transportation is economically impractical. At the same time, the territory of Karakalpakstan is rich in raw materials for the production of local binders and mineral additives to Portland cement, in particular gypsum, limestone, etc (Segalova, 1963, Voljenskiy, 1971) In this regard, scientific work is developing the direction of obtaining gypsum-based binders of various nature and in-depth study of the processes of their hydration structure formation and their properties. The formation of hydration hardening structures, depending on the type, nature and composition of the binder, are present in the additive system. This is largely due to the complex nature of the processes occurring in nonequilibrium multicomponent systems consisting of polymineral binders.

The processes of hydration of clinker and other minerals included in the composition of binders, as well as hydration structure formation (hydration structure formation) in these systems are interrelated (Mixeev, 1957; Ferronskoy, 2004).

## Objects and methods of research

The objects of research in this work were gypsum minerals of the Ustyurt deposit (Republic of Karakalpakstan). Gypsum stones of the studied deposits of the Republic of Karakalpakstan belong to rocks of sedimentary origin, the content of calcium sulfate in them ranges from 60 to 98%. Meanwhile, the large reserves of clay gypsum explored on its territory remain unused for the time being due to the low concentration of gypsum in the rock (Turemuratov, 2019; Abilova, 2019).

Gypsum, which is a natural mixture of two-water gypsum with clay minerals, belongs to such binders. Of particular interest is for Karakalpakstan, where it uses imported gypsum materials. The relatively high solubility, well-defined crystal structure of hydrate phases, the formation of which is not complicated by hydrolysis, makes gypsum binders a convenient object of study.

Thermogravimetric research methods were performed on a Paulik-Paulik-Erdey system derivatograph. The method allows us to qualitatively and quantitatively determine the changes in the characteristic enthalpy, the heat content of substances.

Comprehensive laboratory studies included the study of structure formation in concentrated suspensions (pastes), the interaction of additives with the liquid phase and the determination of the phase composition of the resulting products using thermographic methods. The process of hydration of the sulfate and clay parts in the heat treatment of clay gypsum (100–1000 °C) allows you to change the nature of the structure-forming component.

## Results and their discussions

Heat treatment of clay gypsum at a temperature (100–1000 °C) allows you to change the nature of the structure-forming component, including the sulfate or clay part in the hydration process. This serves as a determining factor in the kinetics of formation and the properties of emerging structures. In accordance with the peculiarities of the chemical nature of the structure-forming component, the regulation of these processes should be carried out in various ways (Asamatdinov, 1977; Abilova, 2018).

For low-temperature gypsum binders, the hardening of which is based on the interaction with water of the semi-hydrate, the determining factor limiting the maximum strength that can be provided by the nominal composition of the binder is the stresses caused by the rapid release of hydrate in volume at high supersaturation. The reduction of the latter, therefore, must be considered as the main task in regulating hydration structure formation in this case. In practice, it can be solved by influencing the processes of dissolution of the semihydrate in the direction of slowing down.

In our experiments, such minerals as calcium hydroxide were used as additives affecting the dissolution of semihydrate, which were introduced in amounts from 0.5 to 10% into dry binders or with mixing water.

Measurements of the kinetics of structure formation in concentrated suspensions with the addition of $Ca(OH)_2$ (1–20%) showed that its presence in the system begins to manifest itself after 24 hours (Table 1). At the same time, the strength increases as the content of $Ca(OH)_2$ in the mixture increases, passing through the maximum at higher concentrations than The activity of the clay part of gypsum is higher, which is determined by the temperature of its firing (Picture 1). For gypsum-based compositions (550 and 700 °C) containing a clay component in the most active form, maximum strength is achieved in mixtures with 5–10% $Ca(OH)_2$.

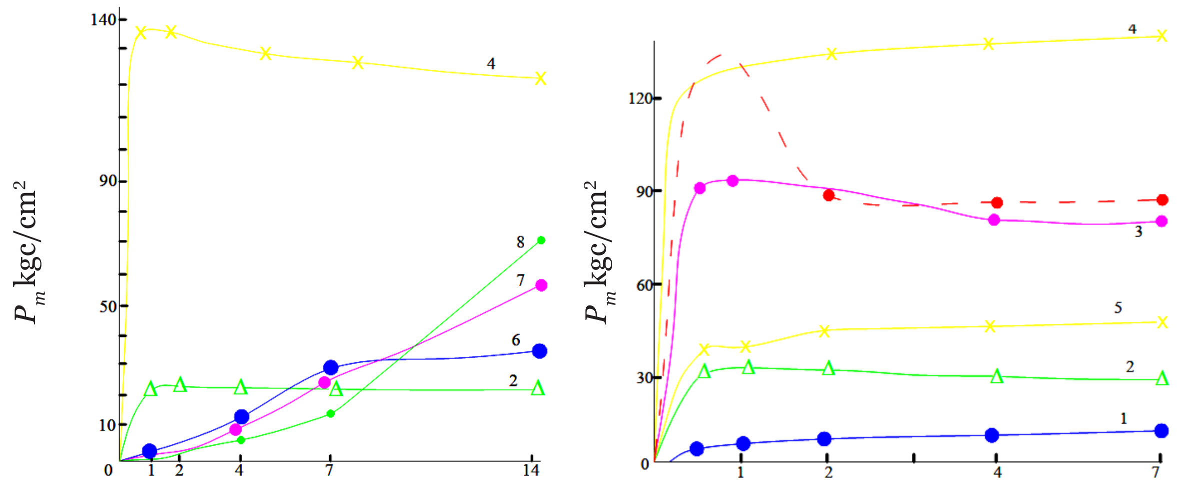
Conductometric studies and analyses of the liquid phase of semi-hydrate suspensions or low-temperature gypsum binders in the presence of $Ca(OH)_2$ indicate a slowdown in the release of dihydrate into the solid phase due to lower supersaturation, and thermogravimetric analysis showed a decrease in the degree of hydration of the binder in the presence of $Ca(OH)_2$. In correlation with this, there is also a sharp inhibition of structure formation with this additive. Over time, the role of lime in the processes of gypsum structure formation changes qualitatively. If at the initial moment the additive serves as a retarder of the hydration of the gypsum part, then over time it begins to exhibit the properties of the chemically active component in relation to the clay reagent, contributing to the formation of additional hydration structures due to interaction with it.

**Table 1.** *Effect of $Ca(OH)_2$ on the kinetics of structure formation of gypsum fired at different temperatures, Rm kgf/cm² (W/H = 0.50)*

| Firing temperature, °C | Supplement $Ca(OH)_{2,\,в\%}$ | The timing of the measurement | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Min | | | | Hour | | | | day | | | | | |
| | | 1 | 5 | 15 | 30 | 1 | 2 | 4 | 6 | 1 | 2 | 4 | 7 | 14 | 28 |
| 500 | 0 | 0 | 0 | 0 | 0.11 | 0.17 | 0.19 | 0.27 | 0.30 | 0.63 | 0.69 | 2.9 | 5.8 | 6.2 | 14 |
| | 1 | 0 | 0 | 0 | 0.14 | 0.16 | 0.19 | 0.21 | 0.28 | 3.9 | 15.6 | 17.1 | 23.3 | 41 | 85 |
| | 5 | 0 | 0 | 0 | 0.13 | 0.15 | 0.16 | 0.27 | 0.36 | 7.6 | 35 | 87 | 189 | 232 | 239 |
| | 10 | 0 | 0 | 0 | 0.11 | 0.12 | 0.14 | 0.31 | 0.35 | 8.5 | 31 | 90 | 183 | 198 | 214 |
| 700 | 0 | 0 | 0 | 0 | 0.06 | 0.13 | 0.21 | 0.23 | 0.25 | 0.49 | 0.55 | 2.8 | 4.6 | 12 | 19 |
| | 1 | 0 | 0 | 0 | 0.06 | 0.11 | 0.14 | 0.17 | 0.64 | 1.3 | 11.5 | 14.1 | 18.5 | 18.9 | - |
| | 5 | 0 | 0 | 0 | 0.09 | 0.16 | 0.27 | 0.39 | 0.97 | 2.9 | 48 | 125 | 154 | 191 | 209 |
| | 10 | 0 | 0 | 0 | 0.07 | 0.12 | 0.17 | 0.19 | 0.32 | 7.2 | 23 | 112 | 120 | 181 | 221 |
| | 20 | 0 | 0 | 0 | 0.08 | 0.16 | 0.19 | 0.15 | 0.47 | 8.7 | 34.9 | 153 | 174 | 189 | 191 |
| 1000 | 0 | 0 | 0 | 0 | 0 | 0.10 | 0.22 | 0.29 | 0.38 | 0.51 | 0.52 | 0.61 | 2.6 | 3.8 | 14 |
| | 1 | 0 | 0 | 0 | 0 | 0.09 | 0.11 | 0.2 | 0.23 | 3.1 | 8.2 | 17 | 27 | - | - |
| | 5 | 0 | 0 | 0 | 0 | 0.10 | 0.11 | 0.11 | 0.13 | 2.2 | 11.1 | 20 | 29 | 75 | 152 |
| | 10 | 0 | 0 | 0 | 0 | 0.09 | 0.11 | 0.11 | 0.12 | 2.3 | 5.3 | 14.1 | 29 | 80 | 167 |
| | 20 | 0 | 0 | 0 | 0 | 0.08 | 0.12 | 0.12 | 0.20 | 0.21 | 0.20 | 3.2 | 6.1 | 15.3 | 153 |

**Picture 1.** *Kinetics of structure formation in concentrated gypsum suspensions depending on the firing temperature: 1–130 °C, 2–150 °C, 3–160 °C, 4–190 °C, 5–250 °C, 6–600 °C, 7–800 °C, 8–1000 °C*

a) b)



In compositions hardening on the basis of insoluble anhydrite, on the contrary, unlike semi-hydrate, stress does not play a significant role, achieving maximum strength is limited by low supersaturation and, accordingly, very slow hydrate formation. Therefore, an increase in the solubility of the structure-forming phase is of the greatest importance for such systems (Ivanov, 2008; Abilova 2019). The strength of high-temperature gypsum binders depends mainly on the number and composition of hydraulically active phases. For the Ustyurt gypsum we are considering, it is limited by the low content of calcium compounds in the sulfate part.

With an increase in the firing temperature of gypsum to 1000 °C the optimal dosage of $Ca(OH)_2$ in the initial period shifts to the area of lower concentrations (1–5%), increasing to 10% over time. This is in accordance with a sharp decrease in the activity of alumina and its transition to inactive β and α modifications during heat treatment, as well as with alumina-containing active mineral additives.

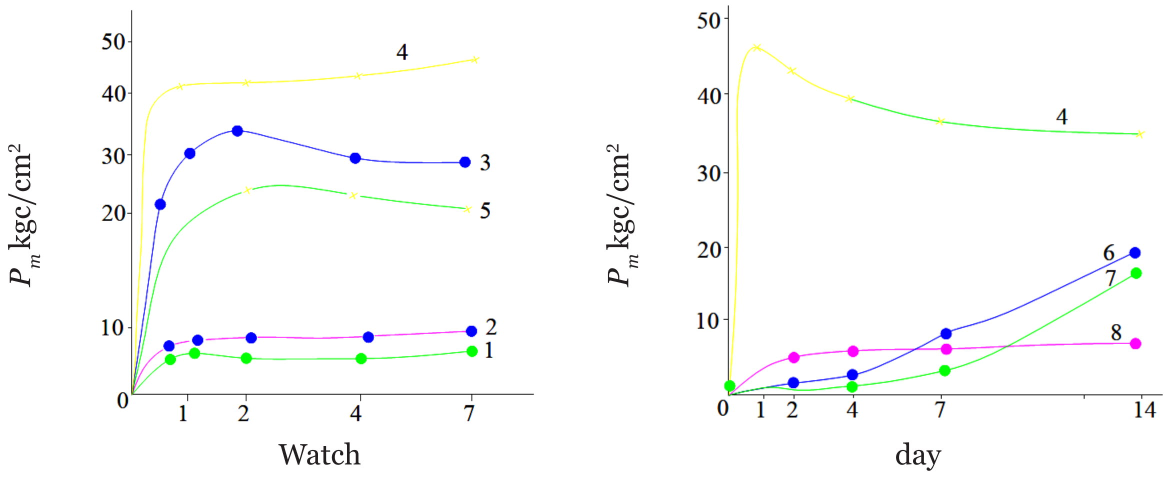
The same patterns are manifested in clay-gypsum mixtures (Table.2) where structures of noticeable strength arise in the presence of $Ca(OH)_2$ already in the first hours.

**Table 2.** *The effect of $Ca(OH)_2$ on the structure formation of clay-gypsum mixture $P_m$, kgs/sm² (W/H = 0.50)*

| Firing temperature, °C | Supplement Ca $(OH)_2$ | Timing measurement | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | min | | | | hour | | | | day | | | | | |
| | | 1 | 5 | 15 | 30 | 1 | 2 | 4 | 6 | 1 | 2 | 4 | 7 | 14 | 28 |
| 550 | 0 | 0.01 | 0.09 | 0.10 | 0.11 | 0.17 | 0.18 | 0.45 | 0.61 | 20.9 | 36.2 | 39 | 34 | 36 | 32 |
| | 1 | 0.10 | 0.12 | 0.25 | 0.61 | 5.1 | 5.5 | 8.7 | 22 | 38 | 41 | 42 | 68 | 68 | – |
| | 5 | 0.09 | 0.13 | 0.15 | 0.24 | 0.51 | 2.3 | 5.2 | 6.8 | 45.5 | 84.6 | 85. | 90.5 | 91 | 151 |
| | 10 | 0.12 | 0.14 | 0.16 | 0.24 | 0.45 | 0.46 | 1.9 | 4.8 | 38.9 | 79.8 | 123 | 182 | 218 | 236 |
| 700 | 0 | 0.01 | 0.01 | 0 | 0.06 | 0.19 | 0.15 | 0.18 | 0.21 | 0.53 | 1.42 | 2.25 | 7.4 | 7.1 | 17.2 |
| | 10 | 0.11 | 0.17 | 0.23 | 0.21 | 0.26 | 0.33 | 0.35 | 0.39 | 12.3 | 36.4 | 65 | 189 | 218 | 223 |
| 1000 | 0 | 0.03 | 0.02 | 0 | 0 | 0.16 | 0.15 | 0.16 | 0.17 | 0.24 | 0.56 | 1.6 | 2.32 | 7.1 | 17.8 |
| | 10 | 0.10 | 0.14 | 0.15 | 0.22 | 0.23 | 0.25 | 0.27 | 0.28 | 0.65 | 1.9 | 4.2 | 16.6 | – | 96 |

As can be seen from (picture. 2), the course of the kinetic curves $P_m - \tau$ changes significantly: as the semihydrate disappears in the mixture, instead of a steep rise, indicating the avalanche nature of the process at the initial stages, as a result of the instantaneous occurrence of high supersaturation in suspensions ß–$CaSO_4 \cdot 0.5H_2O$, a slow, a gradual rise in the late stasges of hydration, indicating a change in the nature of the structure-forming component. In firing products above 450 °C, this component is soluble anhydrite, which does not give solutions supersaturated in dihydrate at the first stages of interaction with.

**Picture 2.** *Kinetics of structure formation in concentrated suspensions of clay-gypsum mixture depending on the firing temperature:1–130 °C, 2–150 °C, 3–160 °C, 4–190 °C, 5–250 °C, 6–600 °C, 7–800 °C, 8–1000 °C*

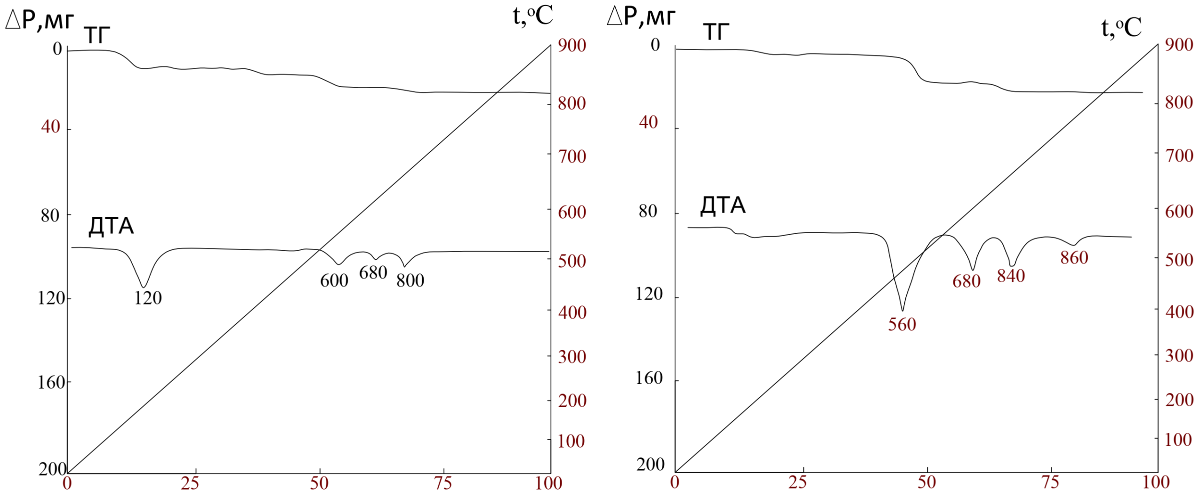


a) b)

With an increase in the temperature of gypsum heat treatment from 500 to 600 °C and the transition to the lime-pozzolan type of hardening, when the chemical activity of the clay component increases as a result of dehydration, the role of calcium hydroxide in creating strength increases.

Measurements of the kinetics of structure formation in concentrated suspensions with the addition of $Ca(OH)_2$ (1–20%) show that its presence in the system begins to manifest itself after 24 hours. At the same time, the strength increases as the $Ca(OH)_2$ content in the mixture increases, passing through the maximum at the higher concentrations, the higher the activity of the clay part of the ganch, which is determined by its firing temperature. For compositions based on ganch 550 and 700 °C, maximum strength is achieved in mixtures with 5–10% $Ca(OH)_2$ (Picture 3).

**Picture 3.** *Diffractograms and derivatograms of initial samples of natural gypsum and clay-gypsum mixtures*



a) b)

Calcium hydroxide added to gypsum binders also affects the kinetics of hydration of its gypsum component. As a result of the interaction with water of anhydrite, which is part of gypsum, burned at 550 °C and above, a dihydrate is formed. For a composition with gypsum 550 °C, the appearance of dihydrate in the solid phase is unambiguously fixed after 5 minutes from the beginning of hydration. In mixtures with gypsum 700 °C, the induction period, during which anhydrite gradually dissolves in water, forming supersaturated solutions and releasing a solid phase, increases to several days. When hydrating burnt gypsum at 1000 °C, this process proceeds even more slowly.

The role of calcium hydroxide introduced into high-temperature gypsum binders (550–700 °C) is not limited to its participation in the formation of new hydrate phases, it also affects the kinetics of hydration of anhydrite.

## Conclusions:

Thus, gypsum binders obtained by firing at a temperature of 550–700 °C from natural gypsum, with the addition of $Ca(OH)_2$, they acquire properties of increased moisture resistance compared to conventional gypsum binders, while maintaining air resistance at the same time. This quality may be of interest in the practical use of large reserves of "low-grade" natural gypsum with a low concentration of gypsum and an increased content of clay.

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# OPTIMIZATION OF THE PROCESS OF EXTRACTION OF THE ABOVE-GROUND PART PLANTS HAPLOPHYLLUM PERFORATUM JUSS

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## Abstract

Studies are presented to study the optimization of the extraction process of the aerial part of the plant Haplophyllum perforatum Jussem. Rutaceae growing on the territory of the Republic of Uzbekistan using the Box-Wilson mathematical method, and optimal conditions for extraction from raw materials were identified, namely extraction with 96% alcohol at a temperature of 35 °C, extraction time – 6 hours.

**Keywords:** *Haplophyllum perforatum, aerial part, optimization, Box-Wilson method, insecticidal activity*

## Introduction

In world practice, synthetic agents are widely used to protect plants from pests. Improper use and increased consumption of chemicals negatively affects human health and the environment. It is known that due to pests, the loss of agricultural products per year is 50–70%. Based on this, the search and implementation of environmentally friendly plant protection products is considered an urgent task.

Recently, interest has increased in plants that produce secondary compounds with repellent, insecticidal and acaricidal effects (Turaeva S. M., Kurbanova E. R., Mamarozikov U. B., Zakirova R. P., Khidirova N. K., 2022). This indicates the possibility of obtaining biological products from them as an alternative to synthetic pesticides (Mamarozikov U. B., Zakirova R. P., Khidyrova N. K. Rakhmatov A., Asatova S., 2017). Previously, we identified the insecticidal activity of the extract of the aerial parts of Haplophyllum perforatum Juss plants against aphids Aphis pomi, S. graminum, Macrosiphum euphorbiae (Mamarozikov U. B., Bobakulov Kh. M., Turaeva S. M., Zakirova R. P., Rakh-matov Kh. A., Abdullaev N. D., Khidyrova N. K., 2019).

This paper presents data on optimizing the extraction process of the aerial part of the plant Haplophyllum perforatum Juss. It is known that the extraction of natural compounds depends on many factors, each of which affects the yield of the final product to a greater or lesser extent.

Therefore, to assess the degree of their influence on extraction, as well as to determine the conditions for the maximum yield of dry extract from leaf plants, the method of mathematical planning of an experiment according to Box–Wilson was used (Hajibaev T. A., Khalilov R. M., Sagdullaev Sh. Sh., 2017; Ibragimov T. F., Khadjibaev T. A., Li A. V., Dzhanibekov A. A., 2015).

## Method and Results

The optimization parameter was the yield of dry extract from the content in the raw material at the first contact of the phases. In all experiments, the amount of raw material and the isolation method were identical.

Based on a priori information (in this case, the results of one-factor experiments), we selected the factors that most influence extraction and established the following main levels and ranges of variation for them (Table 1):

$X_1$ – extraction temperature, °C;
$X_2$ – duration of the process, h;
$X_3$ – amount of ethyl alcohol,

**Table 1.** *Factors and ranges of variation*

| Factor level | Factor | | |
|---|---|---|---|
| | $X_1$ | $X_2$ | $X_3$ |
| Upper | 35 | 28 | 1:12 |
| Average | 30 | 25 | 1:10 |
| Lower | 25 | 22 | 1:8 |
| Variation interval | 5 | 3 | 2 |
| Unit | °C | h | |

Two levels of four factors are established, i.e. full factorial experiment of type 23. A matrix for planning experiments was compiled (**Table 2**) and the results of the experiments were recorded in it.

**Table 2.** *Experiment planning matrix and their results*

| Experience No. | Factor code | | | | $Y_1$ | $Y_2$ | $Y_{av.}$ |
|---|---|---|---|---|---|---|---|
| | $X_0$ | $X_1$ | $X_2$ | $X_3$ | | | |
| 1 | + | + | – | – | | | 6.512 |
| 2 | + | + | – | + | | | 6.615 |
| 3 | + | + | + | – | | | 6.550 |
| 4 | + | + | + | + | | | 6.619 |
| 5 | + | – | – | – | | | 5.560 |
| 6 | + | – | – | + | | | 5.618 |
| 7 | + | – | + | – | | | 5.673 |
| 8 | + | – | + | + | | | 5.810 |

Each of the 8 experiments was carried out in accordance with the compiled matrix, using selected levels of each factor, coded in the matrix with the signs «+» or «–» (upper and lower levels of variation, respectively).

For example, experiment № 1 was performed under the following conditions: plant leaves: extractant alcohol (1:8);

– extracted at a temperature of 35 °C, the duration of phase contact was 22 hours.

Experience № 5. plant leaves: alcohol extractant (1: 8):

– extracted at a temperature of 25 °C, the duration of phase contact was 22 hours and so on in all 8 experiments.

We present the experimental results in the form of a regression equation:

$$Y = b_0+b_1x_1+b_2x_2+b_3x_3;$$

where: $b_0$, $b_1$, $b_2$, $b_3$, are the regression coefficients of the complete quadratic equation.

## Experimental Part

Postulating that the process under study at given intervals of variation of variables can be described by a linear dependence, and using the least squares method, we determined the coefficients:

$$b_i = \frac{\sum_{i=1}^{N} (X_{ij} \times Y_i)}{N} \qquad (1)$$

where: i is the number of experience (1,2...8); j-factor number (1,2...4); Xij – coded value of factors; N – is the number of experiments in the matrix.

Using formula 1, we calculated the values of the regression coefficients:

$b_0 = 6.125$; $b_1 = 0.45$; $b_2 = 0.048$; $b_3 = 0.05$;

Substituting the calculated values of the "b" coefficients into equation 2, we obtained:

$Y = 6.125 + 0.45X_1 + 0.048X_2 + 0.05X_3$

As a result, a mathematical model of the process was established, which is a first-order regression equation. To verify the correctness of the experiment and the adequacy of the resulting model, statistical processing of the data obtained was carried out (Table 3).

**Table 3.** *Statistical analysis*

| $Y_1$ | $Y_2$ | $Y_{av}$ | D | D | $S_i^2$ | $Y_{cal}$ | D | (D |
|---|---|---|---|---|---|---|---|---|
| 6.522 | 6.502 | 6.512 | 0.01 | 0.0001 | 0.0002 | 6.48 | 0.032 | 0.001 |
| 6.605 | 6.625 | 6.615 | 0.01 | 0.0001 | 0.0002 | 6.58 | –0.035 | 0.001 |
| 6.590 | 6.510 | 6.550 | 0.04 | 0.0016 | 0.0032 | 6.57 | –0.02 | 0.0004 |
| 6.649 | 6.589 | 6.619 | 0.03 | 0.0009 | 0.00018 | 6.67 | –0.051 | 0.003 |
| 5.570 | 5.55 | 5.560 | 0.01 | 0.0001 | 0.0002 | 5.58 | 0.02 | 0.0004 |
| 5.608 | 5.628 | 5.618 | 0.01 | 0.0001 | 0.0002 | 5.68 | –0.062 | 0.0038 |
| 5.690 | 5.655 | 5.673 | 0.017 | 0.00029 | 0.00058 | 5.67 | 0.003 | 0.0009 |
| 5.828 | 5.792 | 5.810 | 0.018 | 0.00032 | 0.00064 | 5.77 | 0.04 | 0.0016 |
| Sum |  |  |  |  | 0.00540 |  | – | 0.0121 |

To determine the variation in the values of repeated experiments, we used the variance calculated by the formula:

$$S_i^2 = \frac{\sum_{q=1}^{n} (Y_q - Y_{cp})}{n-1} \qquad (2.3)$$

where: $Y_q$ is the result of a separate experiment; $Y_{cp}$ is its arithmetic mean value; ($n$ –
–1) – the number of degrees of freedom equal to the number of repeated experiments minus one.

For two repeated experiments, formula 2.3 took on the following form:

$$S_i^2 = \frac{2\Delta Y^2}{1} \qquad (2.4)$$

Homogeneity of variance was carried out using the Cochran criterion:

$$G_{экс} = \frac{S^2_{\max}}{\sum_{i=1}^{N} S_i^2} \le G_{кр} \qquad (2.5)$$

$G_{кр} = 0.6798$ [129]

$G_{экс} = 0.12$

$0.12 < 0.6798$

The result obtained corresponds to the conditions of formula 5. The dispersion is homogeneous.

To check the adequacy of the resulting model, the variance of adequacy was first determined.

$$S_{ad}^2 = \frac{\sum_{i=1}^{N} (\Delta Y_i')^2}{f} \qquad (2.6)$$

Then Yras was found; (Table 2)

Based on the results obtained, we find – $Y_i'$ using the formula

$$\Delta Y_i' = Y_{cp} - Y_{pac}; \qquad (2.7)$$

After this, the reproducibility variance was determined using the formula:

$$S_Y^2 = \frac{\sum_{i=1}^{N} \sum_{q=1}^{n} (Y_{iq} - Y)^2}{N(n-1)} \qquad (2.8)$$

where: $i$ =1,2, ..., N

$q$ =1,2,..., n

For two repeated experiments, formula 8 took the form:

$$S_Y^2 = \frac{2\sum_{i=1}^{n}\left(Y_{iq} - Y\right)^2}{N} = \frac{\sum_{i=1}^{N} S_i^2}{N} \quad (2.9)$$

$$S_Y^2 = \frac{0.054}{8} = 0.007$$

We found the variance of adequacy:

$$S_{ad}^2 = \frac{n\sum(Y_{cp} - Y_{pac})^2}{N - q} \quad (2.10)$$

where: $q = K + 1$;

$K$ – number of regression coefficients.

$$S_{ad}^2 = \frac{0.0242}{4} = 0.0061$$

The adequacy of the model was checked using the Fisher criterion:

$$F_{экс} = \frac{S_{ad}^2}{S_Y^2} = \frac{0.0061}{0.007} = 0.87$$

$F_{tab}$ (2.8) = 4,5 for $f_1 = 2, f_2 = 8$

In this case, $F_{ex} < F_{tab}$; 0.87 < 4.5; therefore, the model is adequate.

To check the significance of the coefficients (regression), you need to: find the variance of the regression coefficients $S^2_{bi}$ using the formula:

$$S_{bi}^2 = \frac{S_y^2}{N} = \frac{0.007}{8} = 0.0009$$

Then construct a confidence interval; $\Delta b_i = tS_{bi.}$

Here: t is the table value of the Student's test for the number of degrees freedoms with which $S_{2y}$ was determined at the selected significance level (usually 0.05);

$S_{bi}$ – square error of the regression coefficient.

$$S_{bi} = \pm\sqrt{S_{bi}^2} = \sqrt{0.0009} = 0.03$$

$$\Delta t_{кр} = 3.182$$

$$\Delta bi = t\,x\,S_{bi} = 3.182 \times 0.03 = 0.095$$

The coefficient is significant if its absolute value is greater than the confidence interval (Table 3).

**Table 4.** *Significance of coefficients*

| $b_i$ – values | Symbol | D | Condition values | Results |
|---|---|---|---|---|
| $B_0$=+6.125 | > | 0.095 | pleasure | The coefficient is significant |
| $b_1$=+0.45 | > | 0.095 | pleasure | The coefficient is significant |
| $b_2$=+0.048 | > | 0.095 | pleasure | The coefficient is insignificant |
| $b_3$=+0.05 | > | 0.095 | pleasure | The coefficient is insignificant |

### Conclusion

As can be seen from (table 4), factor $X_1$, turned out to be significant, which is understandable.

One of the tasks of optimizing the extraction process using the method of mathematical experimental planning is to quantify the contribution of each of the selected factors to the extraction result.

It was found that the main influence on the process of extraction of the above-ground part of the plant is exerted by factor $X_1$ – temperature.

A steep ascent was not carried out, since in experiment № 4 the yield obtained was quite acceptable at the first contact of the phases.

Thus, the research carried out using the method of mathematical experimental planning revealed the optimal conditions for extraction from raw materials at the first phase contact, namely extraction with 96% alcohol at a temperature of 35 °C, extraction time – 6 hours.

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# Section 3. Mechanic engineering

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# ANALYSIS OF THE OPERATION OF A HOLLOW THICK-WALLED CYLINDER IN THE CASE OF A STATIONARY AXISYMMETRIC TEMPERATURE LOAD

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**Abstract**

The work of a hollow thick-walled cylinder made of anisotropic material is analyzed in the case of a stationary axisymmetric load acting on it. The law of change in the increment of the temperature load in the cylinder is known. The design scheme is represented by a thick-walled cylinder, the end surfaces of which are rigidly fixed in the axial plane, and there are no fasteners in the radial plane, the cylindrical surfaces are stress-free. It is concluded that only radial and circumferential deformations can be taken into account in the calculations, and relative deformations along the height of the cylinder can be neglected.

**Keywords:** *axisymmetric problem, thermoelectroelasticity, finite integral transformations, stationary action*

## Introduction

Consider a hollow thick-walled cylinder made of an anisotropic material under the action of a stationary temperature load. The mathematical formulation of the problem includes differential equations of equilibrium of the components of the displacement vector and temperature increment, as well as boundary conditions (Grinchenko V.T., Ulitko A.F., Shulga N.A., 1989; Senitsky Yu.E. 2011):

$$\frac{\partial}{\partial r}\nabla U+a_1\frac{\partial^2 U}{\partial z^2}+a_2\frac{\partial^2 W}{\partial r\partial z}=\frac{\partial\Theta}{\partial r}, \qquad (1)$$

$$a_1\nabla\frac{\partial W}{\partial r}+a_3\frac{\partial^2 W}{\partial z^2}+a_2\nabla\frac{\partial U}{\partial z}=a_4\frac{\partial\Theta}{\partial z}$$

$$z=0,h \quad W=0,\ \frac{\partial W}{\partial r}+\frac{\partial U}{\partial z}=0, \qquad (2)$$

$$r=R,1 \quad \frac{\partial U}{\partial r}+a_5\frac{U}{r}+a_6\frac{\partial W}{\partial z}=\left\{\omega_1,\omega_2\right\},$$

$$\frac{\partial W}{\partial r}+\frac{\partial U}{\partial z}=0, \qquad (3)$$

This system of equations is presented in a dimensionless form. We investigate it by using Fourier transforms:

$$U_c(r,n)=\int_0^h U(r,z)\cos(j_n z)\,dz,$$

$$W_s(r,n)=\int_0^h W(r,z)\sin(j_n z)\,dz,$$

$$U(r,z)=\sum_{n=1}^{\infty}\Omega^{-1}U_c(r,n)\cos(j_n z),$$

$$W(r,z)=\frac{2}{h}\sum_{n=1}^{\infty}W_s(r,n)\sin(j_n z), \qquad (4)$$

$$\frac{d}{dr}\nabla U_c-a_1 j_n^2 U_c+a_2 j_n\frac{dW_s}{dr}=F_1, \qquad (5)$$

$$a_1\nabla\frac{dW_s}{dr}-a_3 j_n^2 W_s-a_2 j_n\nabla U_c=a_4F_2;$$

$$r=R,1 \quad \frac{dU_c}{dr}+a_5\frac{U_c}{r}+a_6 j_n W_s=\{\omega_{1c},\omega_{2c}\},$$

$$\frac{dW_s}{dr}-j_n U_c=0, \qquad (6)$$

where $F_1(r,n)=\frac{d}{dr}\int_0^h\Theta(r,z)\cos(j_n z)\,dz,$

$$F_2(r,n)=\int_0^h\frac{\partial\Theta(r,z)}{\partial z}\sin(j_n z)\,dz,$$

$$\{\omega_{1c}(R,n),\omega_{2c}(1,n)\}=$$

$$=\frac{d}{dr}\int_0^h\{\omega_1(R,z),\omega_2(1,z)\}\cos(j_n z)\,dz.$$

The system (5) is reduced to a resolving equation with respect to the function $W_s$:

$$\nabla\frac{d}{dr}\nabla\frac{dW_s}{dr}+b_1\nabla\frac{dW_s}{dr}+a_3 j_n^4 W_s=F_H, \qquad (7)$$

the right-hand side of which admits the following factorization into commutative multipliers:

$$\left(\nabla\frac{d}{dr}-A^2\right)\left(\nabla\frac{d}{dr}-B^2\right)W_s=F_H, \qquad (8)$$

The general solution of the differential equation (9) has the form:

$$W_s(r,n)=D_{1n}I_0(A_n r)+D_{2n}K_0(A_n r)+$$
$$+D_{3n}I_0(B_n r)+D_{4n}K_0(B_n r)+$$
$$+\int_R^r\frac{F_H(\tau,n)}{\det\|B_{ms}(\tau)\|}\sum_{m=1}^{4}\det\|G_{ms}(r,\tau)\|d\tau, \qquad (9)$$

The expression for the function $U_c(r,n)$ is obtained by reducing the system (5) to (7) and has the form:

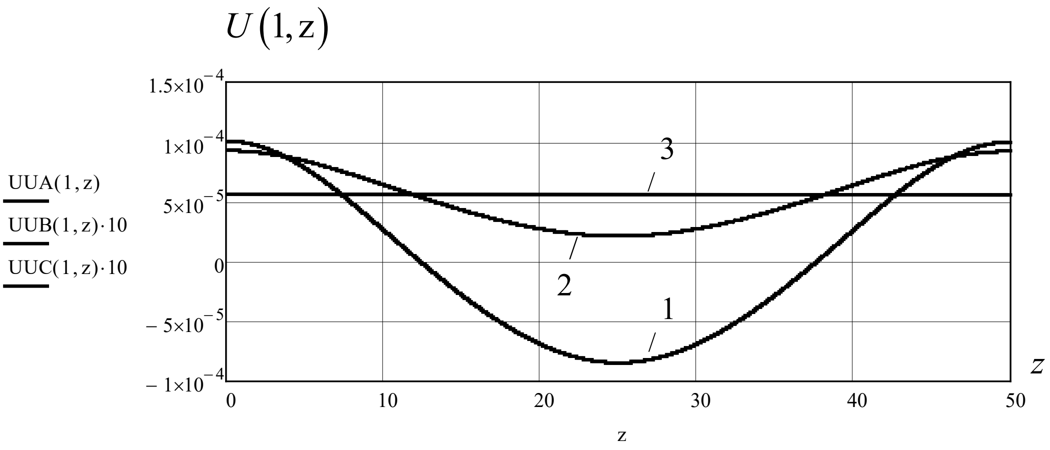
$$U_c(r,n)=\frac{1}{a_2 j_n^3}\frac{d}{dr}\nabla\frac{dW_s}{dr}+$$
$$+\frac{(a_2a_2-a_3)}{a_1a_2 j_n}\frac{dW_s}{dr}-\frac{1}{a_1 j_n^2}F_1-\frac{a_4}{a_1a_2 j_n^3}\frac{dF_2}{dr}. \qquad (10)$$

The final expressions for determining displacements are obtained as a result of substituting (9), (10) into (4).

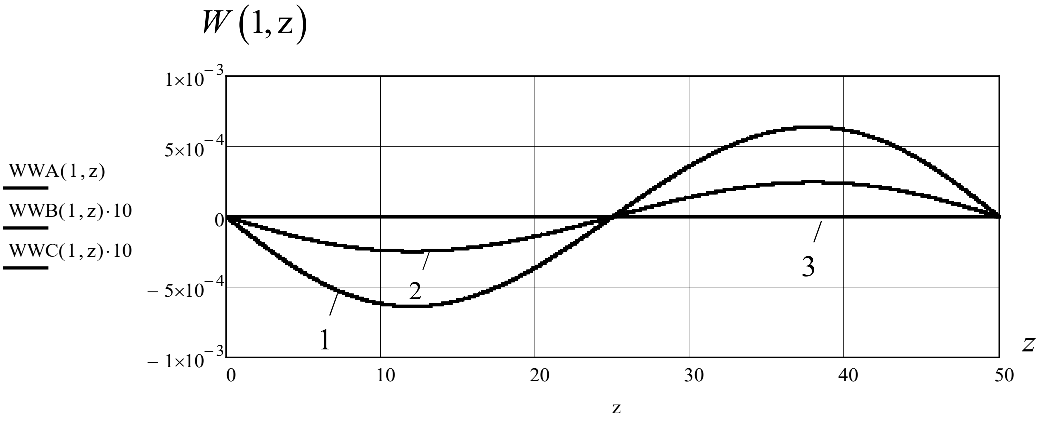
Analyzing the numerical dependences given, graphs of changes in height displacements of a piezoceramic cylinder with finite dimensions, without taking into account the electrical load, were obtained.

**Figure 1.** *Graphs of changes in cylinder height movements*

a)



b)



When the temperature load changes, the radius of the cylinder changes, associated at the first stage with a decrease in it, and then with an increase. When determining the movements along the height of the cylinder, we come to the conclusion that the values decrease (Fig. 1, b, line 1, 2). When exposed to a constant temperature load along the height of the piezocermal cylinder, a radial component arises that practically does not change in height, this follows from Graph 3, Fig. 1 b, and small the values determine the vertical movements.

It follows from the above that when studying a thick-walled cylinder with finite dimensions under the influence of a constant temperature load, the problem can be described using thermal conductivity equations that take into account radial and circumferential deformations (Kalmova M. A., 2023).

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## Section 3. Technical science in general

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# STUDY OF THE TECHNOLOGY OF FLOUR PRODUCTION FROM LOCAL VARIETIES OF THE WHEAT AND RYE AND IMPROVEMENT OF ITS BAKING QUALITIES

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**Abstract**

The article discusses the advantages of the production of wheat-rye bakery products by separating large fractions of intermediate grinding. A comparative analysis of experimental and control samples baked using traditional technology is given. Practical recommendations on the use of improvers for bread production are given.

**Keywords:** *technological process, acidity, porosity, baking properties, bread, pastries*

## Introduction

The technology of producing flour, bread and bakery products belongs to an exceptional place in human nutrition, since these products provide a significant part of the physiological need of the human body for food substances.

High taste advantages and increased nutritional value of rye-wheat bread ensure a fairly high demand for this product and its expanded range.

The usual technology for preparing rye-wheat bread varieties provides for separate processing of rye and wheat grains, followed by mixing rye and wheat flour at bakeries. Varietal grinding of wheat is characterized by complexity, significant energy and labor costs, and also requires a sufficiently high quality of processed grain (Butkovsky B.A., Merko A.I., Melnikov E.M., 1999; Egorov G.A., 2005; Anninkova T. Yu., 2001; Auerman L. Ya., 1984; Polandova R.D., Guseva L.I., Maslikova N.H., 1984; Kovbasa V.N., Kobylinskaya E.V., Kovalev A.V., etc., 1998).

Due to these advantages, research on improving the technology of production of rye-wheat flour from grain mixtures is relevant and has important theoretical and practical significance (Butkovsky V.A., 1989; Yakovleva O.V., 1995; Butkovsky B.A., Merko A.I., Melnikov E.M., 1999; Egorov G.A., 2005; Andreev A.N., 1999; Anninkova T. Yu., 2001).

In this regard, in this work, in addition to studies of certain stages of the technological process of processing rye-wheat mixtures, attention was paid to the problem of interaction of their components.

The rye and wheat standard provides for a restriction of the content of germinated grains to 3% (Kislukhina O.,1997; Yakovleva O.V.,1995; Auerman L. Ya., 1984; TU8–18–149–94; Matveeva I.V., Belyavskaya I.G., 1998).

In terms of grinding capacity, rye and wheat differ from each other due to the peculiarities of structural and mechanical properties. Also, the process of forming varietal rye-wheat flour from grain mixes by qualitative indices and percentage content of components is not studied, which is necessary for production of products with specified composition and properties (Butkovsky V.A.,1989; Kislukhina O., 1997; Butkovsky B.A., Merko A.I., Melnikov E.M., 1999; Egorov G.A., 2005; Kovbasa V.N., Kobylinskaya E.V., Kovalev A.V., etc., 1998).

However, the chemical composition of bakery wheat flour is characterized by a low content of essential amino acids, a low content of dietary fiber, which, in turn, requires enrichment of these products with micronutrients.

In this regard, rye grain processing products have an absolute advantage. Rye has worse flour-milling advantages compared to wheat grain, but it is significantly superior to wheat in amino acid composition (Kislukhina O.,1997; Egorov G.A.,2005; Bystrova A.I., Tokareva G.A.,1997; Matveeva I.V., Belyavskaya I.G.,1998; Poland R.D., Barkalova I.V., Podobedov A.V., etc. 1997).

To obtain rye flour with high yields and quality that meets the requirements and standards, it is necessary to improve grinding, but there are two problems:

1) rational use of grain mixture or flour;

2) obtaining high-quality finished products, a useful product for human health.

Due to the fact that rye grain has increased autolytic activity, the studies are devoted to the problem of improving the bakery advantages of rye grain, which are characterized by amylolytic activity.

An effective means of improving the bakery properties of flour, regulating the technological process and improving the quality of bread is the use of complex additives – improvers (Kislukhina O., 1997; Egorov G.A., 2005; Bystrova A.I., Tokareva G.A., 1997; Vorobyova I.S., 2002; TU8-18-149-94; Dremucheva G., Karchevskaya O., Polandova R., 2000; Matveeva I.V., Belyavskaya I.G., 1998; Polandova R.D., Guseva L.I., Maslikova N.H., 1984; Nechaev A.P., Dubtsova G.N., Dubtsov G.G., Bakulina O.N., 1990; Poland R.D., Barkalova I.V., Podobedov A.V., etc. 1997; Kovbasa V.N., Kobylinskaya E.V., Kovalev A.V., etc., 1998)

Currently, both in our country and abroad, the use of multi-component enhancers, the composition of which is specially selected taking into account the alleged focus of their action, has become the most widespread.

In this regard, research aimed at the development of bakery improvers from domestic, affordable and inexpensive raw materials is of particular relevance, since bread is a commodity of everyday demand and the increase in its cost will primarily affect the meager budget of socially unprotected segments of the population (Yakovleva O.V., Egorov G.A., 2005, Bystrova A.I., Tokareva G.A., 1997; 1995; TU8-18-149-94; Nechaev A.P., Dubtsova G.N., Dubtsov G.G., Bakulina O.N., 1990; Poland R.D., Barkalova I.V., Podobedov A.V., etc. 1997).

Aims and functions of scientific research.

– to development of technology for production of wheat-rye varieties of flour;

– to study of composite mixtures as improvers of wheat-rye bread varieties.

a study of the efficiency of using enzymes for wheat and rye flour;

– to make compositions of baking improvers for bread produced from wheat flour with "strong" or "weak" gluten;

– to determine the nutritional value of finished products and developed improvers;

– to conduct a comparative assessment of the effect of bakery improvers on the quality of bread and its preservation;

– to develop and approve technical documentation.

### Methods of the study:

Technical analysis of grain and flour was carried out in accordance with GST 10839-64 “ Grain. Test methods. “Nature was determined according to GST 10840-64; vitreous content – GST 10987-76; weight of 1000 grains – GST 10842-89; quantity and quality of gluten – GST 13586.1-68; ash content – GST 10847-74; content of weeds, grain impurities, fine grains and coarseness

– GST 13586.2–81; germination energy – GST 10968–88; acidity – GST 10844-74; humidity – GST 13586.5–85; protein – GST 10846-74; fall number – by the Hagberg-Perten method according to GST 27676-88. Autolytic activity of grain was determined by exspress baking. An organoleptic assessment of the appearance and state of the crumb of baked finished products was carried out.

Flour was analyzed for the following parameters: humidity determined according to GST 9404-88; ash content – GST 10847-74; acidity – GST 27493-87; size – GST 27560-87; whiteness – on RZ-BPL device as per GST 26361-84; number of fall – GST 27676-88.

### Porosity determination procedure (as per GST 5669–96):

$P = 100 \cdot (V - m/p)/V$, where

P – porosity,%;

V – total volume of bread cuts, $cm^3$;

m – mass of notches, g;

p – is the density of the non-porous mass of the crumb.

### Determination of bread acidity (according to GST 5670–96):

Acidity of pulp X (deg) is calculated with accuracy to 0.5 deg. by the formula:

$X = 2V \cdot K$, where

X – acidity, deg;

V – volume of sodium hydroxide solution with molar concentration 0.1; $mol/dm_3$ used for titration of the test solution, cm3;

K – correction factor of the solution used sodium hydroxide to a 0.1 $mol/dm_3$ solution.

### Experimental part

This detail provides experimental learning of the baking properties and quality indicators of flour and bread made from wheat and rye.

**Table 1.** *Recipe of raw materials and baking process parameters*

| Description of raw materials and process parameters | Quantities of raw materials and bread preparation parameters | | | |
|---|---|---|---|---|
| | control | 1 sample | 2 sample | 3 sample |
| | Dough | Dough | Dough | Dough |
| First grade wheat flour, kg | 100 | 95 | 90 | 80 |
| Rye flour, kg | – | 5 | 10 | **20** |
| Fariman yeast, kg | 1 | | | |
| Table salt, kg | 1.5 | | | |
| Water | by calculation | | | |
| Humidity,% | 48 | 47 | 46 | 46 |
| Initial temperature,° C | 26–30 | | | |
| Fermentation duration | 25–30 | | | |
| Baking time, min | 20–25 | | | |
| Baking oven temperature, ° C | 220–240 | | | |

Bread baking was carried out in a laboratory electric oven of the company “MF GR Company” at a temperature of 230–240 °C for 35–40 minutes. The baked bread was stored in a laboratory cabinet. The analysis was carried out 12–18 h after baking.

As can be seen from table No. 2, the quality indicators of flour produced from wheat and rye grain utilise enzymes showed the best indicators on samples 3.

**Table 2.** *Quality indicators of obtained flour from wheat and rye grain (with addition of 0,5,10,20% of flour and 0,03 g of enzyme Alfa2 and LOU)*

| Indicators | 1 sample control grade 1 | 2 sample 5% | 3 sample 10% | 4 sample 20% |
|---|---|---|---|---|
| Humidity | 14.8 | 15 | 14.5 | 14.8 |
| Amount of raw gluten | 34 | 32.4 | 30 | 28 |
| Raw gluten quality (IDS) | 73 | 76 | 80 | 78 |
| Description of raw gluten (manual method) | Homogeneous, crumbles a little sticky | non-traditional, crumbles, sticky | unreported, uncooperative, sticky | non-traditional, crumbles, sticky |
| Number of falls per PChP-3 | 325 | 260 | 236 | 224 |
| Ash content,% | 0.77 | 0.86 | 1.10 | 1.21 |
| Whiteness, unit of the device | 48 | 46 | 46 | 44 |
| Damaged starch index: | 23.2<br>7.80 | | | |

**Table 3.** *Quality indices of wheat and rye flour by alveograph*

| Temperature | Parameters | Results are standard | Temperature | Parameters | Results are standard |
|---|---|---|---|---|---|
| Water: 19,7 °C<br>Kneader: 24.2 °C<br>Spacer chamber: 25,2 °C<br>Alveograph compartment: 20,7 °C | Air humidity: 53%<br>Humidity: 15.6<br>Hydration: 50%<br>Basic humidity: B15%$H_2O$<br>Quantity of water: 122.35ml<br>Mass of flour: 250g | P: 158 mm$H_2O$<br>L: 55mm<br>G: 16,5<br>W: 341 10–4J<br>P/L: 2,87<br>Ie: 54,8% | Water: 19,8 °C<br>Kneader: 23.8°C<br>Spacer chamber: 25,2 °C<br>Alveograph compartment: 20,6 °C | Air humidity: 64%<br>Humidity: 14.5<br>Hydration: 50%<br>Basic humidity: B15%$H_2O$<br>Quantity of water: 127.21ml<br>Mass of flour: 250g | P: 118 mm$H_2O$<br>L: 33mm<br>G: 12.8<br>W: 163 10–4J<br>P/L: 3,58<br>Ie: 0% |

**Figure 1.** *Alveogaph curve*
a)

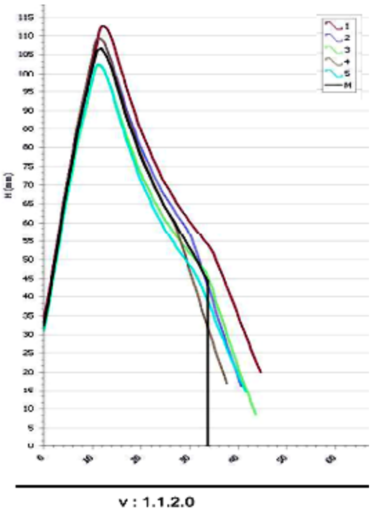

**Figure 2.** *Alveogaph curve*
b)

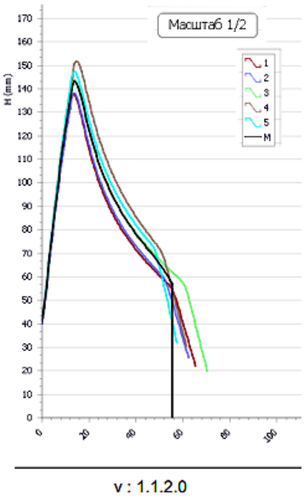

**Figure 3.** *Qualitative indices of porosity and acidity of bread from wheat and rye flour*

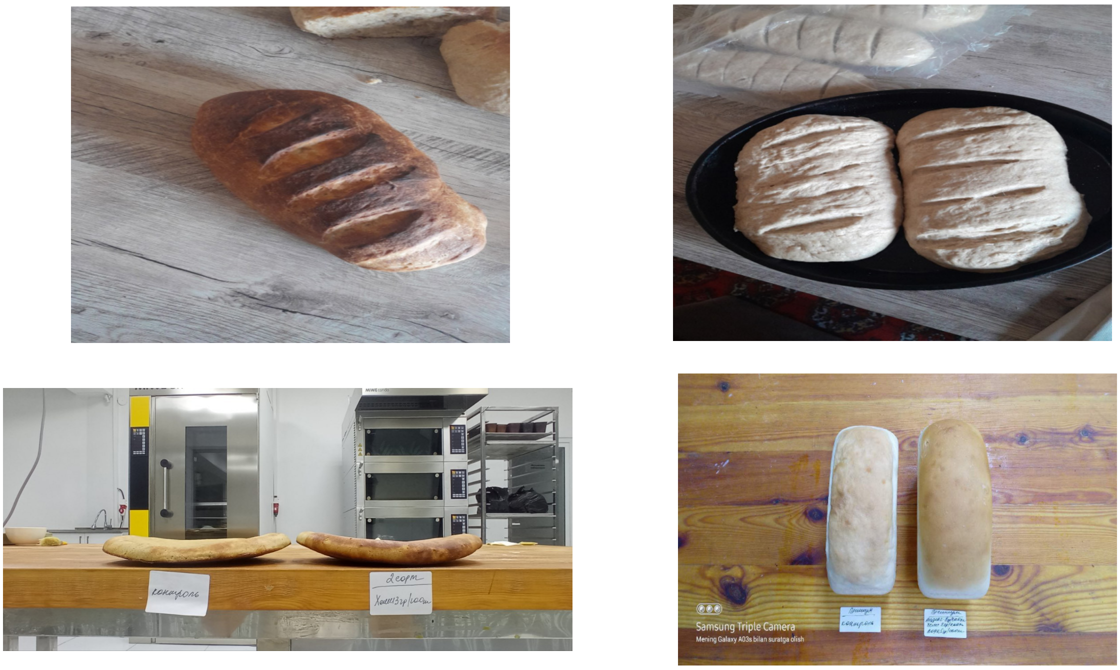

**Table 4.** *Organoleptic, physicochemical and technological properties of products*

| Organoleptic qualities | Properties of indicators | |
|---|---|---|
| | 1-grade | 1-grade Wheat-rye flour mixture |
| Color | White and cream | Dark brown light |
| Taste | Non-cooked flavors, without hot | |
| Smell | Characteristic of the taste of wheat grains and rye | |
| Mineral mixtures | Absent | |
| Consistence | Same with dark shades | |
| Screening No. 38: | 5 / 60 | 2 / 70 |

**Table 5.** *Physical and chemical qualities and technological properties*

| Indicators | Quantities | | | |
|---|---|---|---|---|
| | From 1-grade wheat flour | Wheat and rye flour mixture (5%) | Wheat and rye flour mixture (10%) | Wheat and rye flour mixture (20%) |
| Humidity,% | 14.6 | 14.9 | 14.92 | 14.89 |
| Density, ρn, kg/m$_3$ | 704 | 706 | 708 | 707 |
| Natural angle of inclination | 38 | 35 | 36 | 33 |
| Floatability | well | good | Very good | Satisfactorily speckled |
| Water pollution rate,% | 62.0 | 60.2 | 58.1 | 57.8 |
| Specific volume | 2.4 | 2.5 | 2.76 | 2.71 |
| Prowl | 67 | 68 | 72 | 71 |

**Table 6.** *Indicators of bread quality and study of the effect of flour on baked goods*

| Flour indicators | Bread Control | Bread (5%) | Bread (10%) | Bread (20%) |
|---|---|---|---|---|
| Humidity,% | 48.4 | 46.4 | 44.2 | 45.1 |
| Gaseous force, ml | 1510 | 1480 | 1530 | 1490 |
| Weight of bread, g | 540 | 545 | 560 | 550 |
| Crude gluten composition,% | 26.6 | 28.0 | 24.4 | 21.5 |
| Gluten elasticity, IDK-2 | 74 | 76 | 74 | 72 |
| Prowl | 66 | 68 | 72 | 74 |
| Acidity, deg | 6.2 | 5.8 | 5.4 | 5.6 |

## Conclusions:

– Improvement and investigation of the possibility of separation of coarse fractions of intermediate wheat and rye grain grinding products by Q-factor.

– Comparative analysis of bread quality showed that physicochemical and organoleptic indices of test and control samples were beaten by close ones according to different criteria. In appearance, ground, specific volume, acidity, the test samples were better than the control.

– recommendations on application of improvers for bread production depending on its formulation and baking properties of used wheat and rye flour are given.

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# ANALYSIS OF DEMULSIFIER PRODUCTION FROM SULFURIZED COTTONSEED OIL AND BREAKDOWN OF WATER-OIL EMULSIONS

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## Abstract

This article presents the synthesis of a demulsifier based on the secondary raw materials of the domestic oil industry for breaking down water-oil emulsions of high-viscosity heavy oils. Demulsifier is obtained by sulphuring cotton oil obtained by extraction method with sulfuric acid and treating the obtained component with sodium hydroxide. The process of breaking up emulsions of high-viscosity oils with synthesized demulsifier and previously known demulsifiers was analyzed.

**Keywords:** *water-oil emulsion, complex emulsion, dehydration, desalination, deemulgator, heavy oil, high-viscosity oil, composition*

## Introduction

The decomposition of water-oil emulsions (WOE) of high-sulfur heavy oils involves the implementation of processes such as water drop approaches and their coalescences, which cause the interphasic protective shells of water globules to decompose. In the coalescence of water droplets, their dimensions increase sufficiently to sink to the bottom of the structure.

In existing technologies, a number of processes of dehydration and desalination of oil are used. The choice of one or another method of oil dehydration will depend on the productivity of operation of the devices, the quality and quantity of layer waters, the effectiveness of deemulging.

Most often, the layer water is contained in petroleum raw materials, and in some cases it is in a free state, that is, it is not dispersed. Such water is separated from oil by immersion.

## Research method

Modern deemulgators, as a rule, are chemicals obtained with a complex multi-stage synthesis. Approximately one-third of deemulgators are designed to break down the following types of WOE:”oil in water” and “water in oil”, which is formed in the method of pumping water into oil fields and under the conditions of applying various methods of affecting the layer. While deemulgators decompose WOE, requirements related to the ingestion of emulsions of corrosion in metals are also imposed.

In order to obtain surfactants with the greatest deemulgating activity, optimal proportions of polyoxyethylene chain and hydrophobic radical must be achieved. In the synthesis of noionogenic surfactants, the number of groups of ethylene oxide and propylene oxide changes in the form of blocksopolymers, and thus, based on their properties, the ratio between the hydrophobic and hydrophilic parts of the deemulgator is easily regulated.

For the synthesis of oxyethylated fatty acids with a number of carbon atoms of more than 20, fatty acids and gudrons in cubic residues of oil and oil enterprises are used. The decomposition efficiency and physical properties of these deemulgators (solid temperature, viscosity, density) will depend on the oxyethylenated group of each fatty acid with carbon number $C_{14}$– $C_{25}$.

Effective deemulgators are synthesized with $C_{25}$ based on fatty acid with 65–67% ethylene oxide. In terms of deemulgirating efficiency of stationary WOE, it is not inferior to imported Dissolvan.

Oxytylated alkylphenols are known to be the oxytylation products of mono-and dialkylphenols.

According to polyoxyalkylene block-sopolimers, the most effective and universal deemulgator and their high deemulgating activity are related to appearance, the hydrophobic part of the surfactants is not directed to the depth of the oil phase, like typical deemulgators, and the partial emulsion phases are spread over the surface. This contributes to the low consumption of deemulgators (30 g/t).

## Results

The history of the synthesis of deemulgators consists of several stages: as deemulgators, inorganic products (iron powder, sodium carbonate, mineral acids, etc.), alkaline salts of carboxylic and naphthenic acids, neutralization products began to use oxidized paraffin or gazoyl. In addition, deemulgators containing sulfogroup–$SO_2OH$ or sulfate group–$OSO_2OH$ were developed.

The use of the sodium salt of sulfurized castor oil as a deemulgator the results of the deemulgating properties of sulfurized surfactants have provided the impetus for extensive research. For these purposes, various high – molecular unsaturated fatty acids and oxycislotes, as well as their derivatives-esters or alkylated amides, as well as natural acid glycerides, undergo sulfiration. In addition, many deemulgators were obtained by sulfirating alkylated aromatic hydrocarbons, with subsequent elimination or etherification of the sulfoic acids obtained.

Demulsifiers, in addition to high surface activity, should have a wetting and dispersing effect, especially for asphalt-resin substances. The "effective" concentration of demulsifiers, depending on the ratio of their polar and non-polar parts, determines their hydrophilic-lipophilic balance (HLB). The maximum demulsifying properties of solutions of nonionic surfactants are manifested in the area of their micelle-forming concentrations, in which aqueous solutions of surfactants have high washing and wetting properties.

Fatty amines react with the active groups of the emulsifier on the surface of the phases, and fatty alcohols of medium chain length destabilize the emulsion by diffusion and redistribution of their molecules in the boundary layers. Therefore, a mixture of fatty Amine and fatty alcohol indicates the maximum deemulgirating ability, since "acid – Amine" charged complexes are formed on the surface of the compartment, the film is hydrophilic and cannot stabilize the water oil emulsion.

Authors some specific laws of interphasic distribution of noionogenic surfactants have been identified.

After the decomposition of the emulsion in the aqueous phase, regardless of the total amount of surfactants included in the emulsion (release rate), the stability of the transition share in the initial emulsion over a wide range of dispersed water content was determined.

In research work when the structure of surfactants is studied its effect on the ability to deemulge, the effect of deemulge water oil emulsions will not only depend on the structure, but also depend on the position of the functional groups and the value of the GLM. For example, the ability to deemulge nonionogenic surfactants synthesized on the basis of fatty acids directly depends on their molecular weight and the length of the ethylene oxide chain. Polyglycerides of fatty acids and the above-mentioned ionogenic surfactants are inferior to polyglycol esters of fatty acids, alcohols and alkylamides in their ability to deemulge. In addition, demulsifiers with a high conditional group number of GLM will have the greatest effectiveness in attenuating stable WOEs.

In practice, import deemulgator Reapon-4V designed at a temperature of no less than 15–17 °C is used to deeply dehydrate and desalinate oils. It contains noionogenic surfactants and 50% active substances, and its consumption will be 30–100 g / t compared to oil (Abdirakhimov I. E., 2021; B. 44).

To dehydrate and desalinate stagnant WOE and oil sludge emulsions (OSE), a composite of 5002–2b-40 simple polyephyr, m-2 high boiling fraction, flotoreagent-oxal t-66 and ethylene glycol and water in a certain structural ratio is proposed.

More than 25 large oil and oil enterprises are successfully operating in Uzbekistan, where cottonseed oil is extracted for technical purposes by the method of extraction with a hydrocarbon solvent. Technical cottonseed oil, along with triacylglyceride, contains gossipol, chlorophyll and their derivatives with certain surfactant properties.

For example, the general formula of gossipol is as follows: $C_{30}H_{30}O_8$ acts as a strong dibasic acid, i.e. polyphenol, in chemical reactions. Gossipol interacts with hydroxide, e.g., NaOH forms "phenolates", which means that sodium gossipolates dissolved in water show the surfactant properties of heavy oils in deemulgating water emulsions.

**Figure 1.** *The gossipol and alkali reaction is carried out according to the following scheme:*

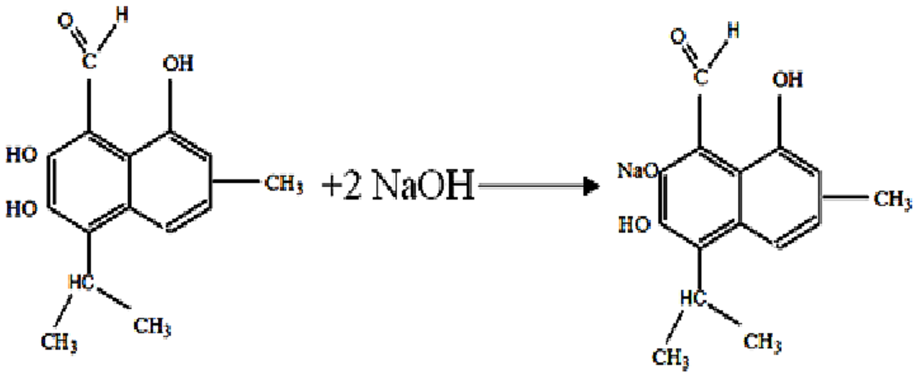
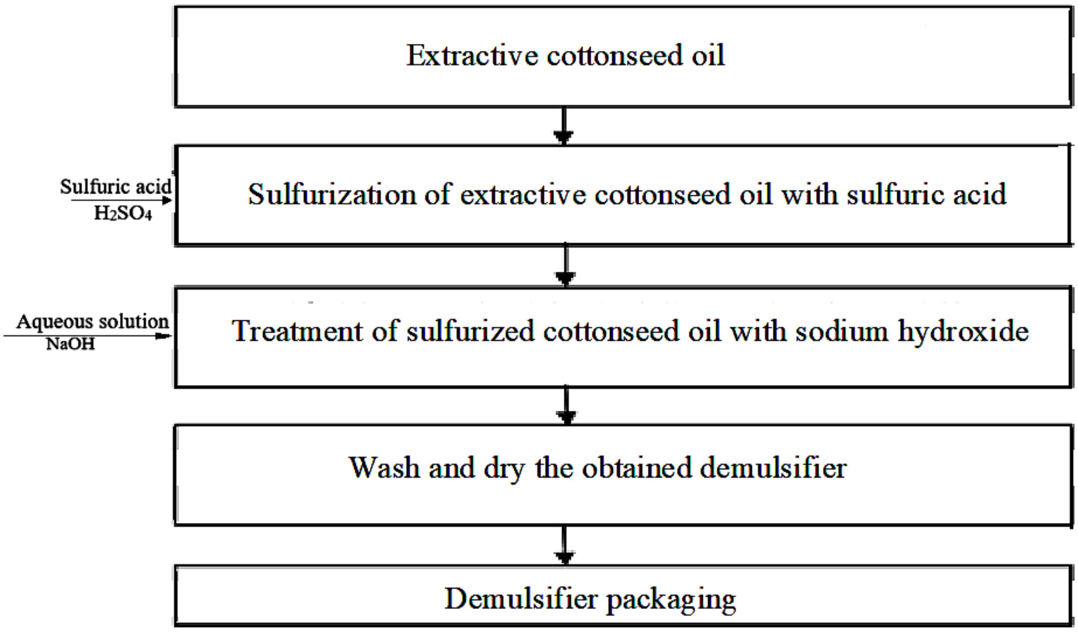


**Figure 2.** *Scheme for obtaining a deemulgator based on sulfurized cottonseed oil*



As you can see, washing with an alkaline aqueous solution of gossipol, for example, NaOH, allows you to get ionogenic surfactants with good moisturizing and foaming bility.

As you can see, washing with an alkaline aqueous solution of gossipol, for example, NaOH, allows you to get ionogenic surfactants with good moisturizing and foaming bility.

The development of the production of deemulgators is focused on the production of surfactants, which today include sulfoguruh ($SO_2OH$) or sulfate group – $OSO_2OH$. Taking this into account, the deemulgators containing the sulfur compounds mentioned above were synthesized on the basis of technical extraction cotton oil. In (Figure 2), a flow scheme is provided to obtain a deemulgator containing a sulfo group.

## Conclusion

The extraction of turbidity of cottonseed oil was carried out by treating it with sulfuric acid. Surfactants with sulfide after alkaline treatment have good deemulgatory properties. This high molecular weight can be obtained on the basis of unsaturated fatty acids or oxide acids, as well as using ether or alkylated amides. Basically, many deemulgators are obtained by sulfirating alkylated aromatic hydrocarbons, followed by elimination or esterification of the sulfonic acids obtained.

**Table 1.** *Indicators of residual water content in the emulsion when using deemulgators K-1 and SD-1, SD-2*

| Deemulgator designation | Deemulgator consumption, g/t | Starting water composition,% | Residual water content in the emulsion,% |
|---|---|---|---|
| K-1 (control) | 60 | 31,0 | 1,5 |
| СД-1 | 40 | 30,5 | 1,0 |
| СД-2 | 50 | 30,8 | 0,9 |

From Table 1, sulfur-containing deemulgators based on cottonseed oil remove water more deeply than the imported K-1 deemulgator in use. The specific consumption of deemulgator is the main technical and economic indicators of the processes of dehydration and desalination of water emulsions, especially heavy oils.

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# INTELLIGENT SYSTEM FOR PROTECTING THE PUMPING UNIT FROM EXCESSIVE VIBRATION

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## Abstract

Vibration is one of the main threats to electric motors, which can lead to long-term failure and premature wear. To prevent such problems, automated and/or intelligent vibration protection systems for electric motors are widely used. This article provides an analysis of the causes of the occurrence of a water pumping unit, substantiates the importance of using intelligent protection against excessive vibration, solution methods and conclusions.

**Keywords:** *automated and/or intelligent protection, electric motor, vibration, reliability, equipment, maintenance*

## Introduction

In modern industrial processes, liquid pumping and gas pumping units play an important role. However, increased vibration can negatively affect the operation of pumping equipment, leading to wear, damage and reduced performance (Smith, J., 2017). Electric motors play a key role in various industrial processes, and their smooth operation is essential for efficient production operations. However, electric motors are subject to vibration, which can be caused by various factors such as load unevenness, imbalance of rotating parts, bearing wear and others (Rao, J.S., 2016). When analyzing the causes of vibration of the pumping unit, it turned out that these factors are connected and interact with each other. Basically, the following aspects of the reasons can be cited:

– ***Electrical aspects.*** The engine is the main equipment of the unit. Imbalances in the magnetic force within the motor and imbalances in the electrical systems often cause vibration and noise.

– ***Mechanical aspects.*** The quality of the rotating parts of the engine and water pump is unbalanced, and the installation quality is poor, the axis of the unit is asymmetrical, the swing exceeds the permissible value, the mechanical strength and rigidity of the parts is poor, the bearing and sealing parts are worn and damaged, and the critical speed of the pump is related to the unit. The resonance caused by the natural frequency will generate strong vibration and noise all the time.

– ***Hydraulic aspects.*** Uneven flow and pressure distribution at the pump inlet, pressure pulsation of the working fluid at the inlet and outlet of the pump, displacement of flow and outlet, poor operating conditions and pump cavitation caused by various reasons are common causes of vibration of the pump unit.

– ***Other aspects.*** Unreasonable design of the unit's water intake channel or incompatibility with the unit, incorrect immersion depth of the pump, as well as unreasonable sequence of starting and stopping the unit will worsen the conditions for draining water, create vortices, cause cavitation or aggravate the vibration of the unit and the pump room.

All these impacts can lead to significant damage and premature failure of electric motors. To prevent such situations, it is important to use automated and/or intelligent protection systems.

## Formulation of the problem

The main task of automated motor vibration protection is to identify abnormal vibrations and take appropriate measures to prevent equipment damage (ANSI/EASA Standard AR100–2015, 2015). The automated control system for protecting the pump unit from increased vibration exposure sets itself the following tasks:

1. Determine abnormal fluctuations that exceed permissible standards.
2. Automatically shut down the motor or implement corrective measures when dangerous levels of vibration are detected.
3. Detection and measurement of increased vibration:
   - constant monitoring of vibration parameters of the pumping unit in real time;
   - analysis of the received data and determination of exceeding the limit values.
4. Response to increased vibration:
   - warning operators about dangerous vibration levels and possible damage to the pumping unit;
   - automatically taking measures to reduce vibration to prevent equipment damage and ensure safe operation.

## Results

In rotating equipment and flowing media, low-intensity mechanical vibrations are inevitable. Therefore, the manufacturing and installation process of the unit should avoid interference caused by vibration as much as possible during the design, operation and control of the unit, and vibration damage should be kept to a minimum. When the pump room or unit vibrates, the reasons that may cause the vibration should be analyzed according to the specific situation, and after discovering the nature of the problem, effective technical measures should be taken to eliminate it. In this case, to solve problems in the control system for protecting the pump unit from increased vibration effects, it is proposed to use automated and intelligent protection using specialized systems and devices. To do this you need to do the following:

1. Installation of vibration sensors:
   - Placement of vibration sensors on strategically important parts of the pumping unit, such as pumps, motors and main mechanisms.
   - Connecting sensors to the monitoring system.
2. Monitoring and data analysis:
   - Continuous collection of vibration data using sensors.
   - Analysis of the obtained data to determine whether vibration limits are exceeded.
3. Warning and response:
   - Issuing warning signals to operators when vibration limits are exceeded.
   - Automatic response system to certain levels of vibration, including procedures for reducing the load on the unit, reducing the speed, adjusting operating parameters and/or emergency shutdown of the pumping unit (Shearer, J., 2018).

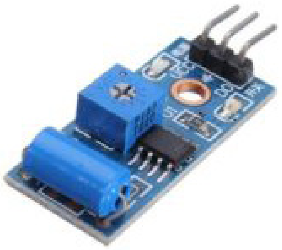
To implement the proposed approach, an 801S vibration sensor was selected (Figure 1), which has three outputs:

- Earth;
- Nutrition;
- Analogue signal output A0.

Technical parameters of vibration sensors for ARDUINO:

- Supply voltage from 3 to 5 V;
- Current consumption 4–5mA;
- With or without a digital output;
- With or without sensitivity adjustment.

**Figure 1.** *Vibration sensor 801S*



In Figure 2 shows a functional and technological diagram of the operation of the electric motor of a pumping unit for pumping liquids and gases. In particular, this article discusses a pumping unit used in the process of treating municipal and domestic wastewater.

**Figure 2.** *Functional and technological diagram of the electric motor*

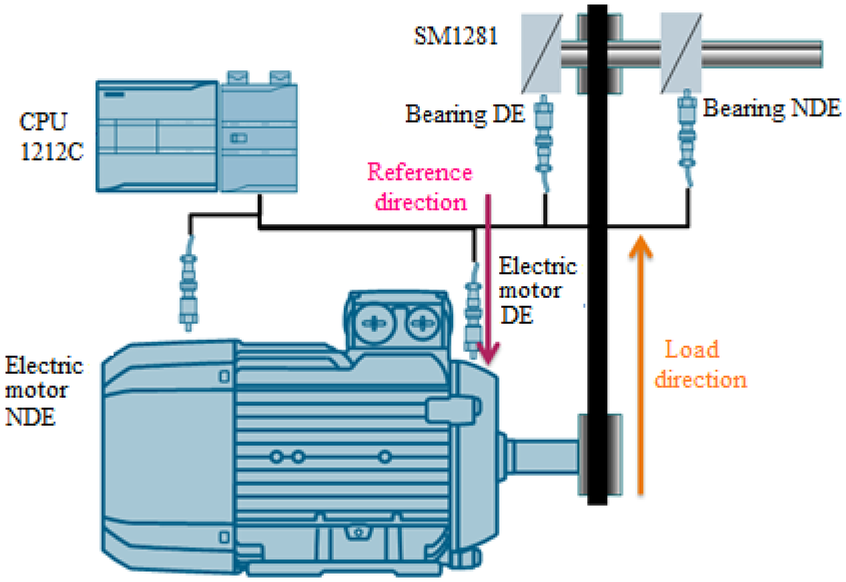
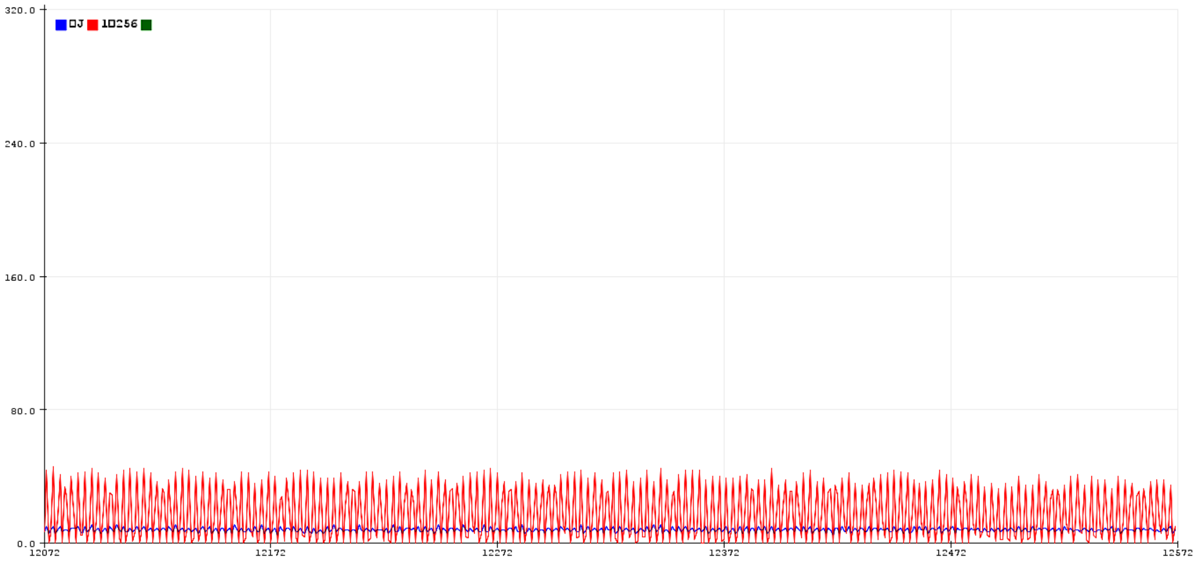
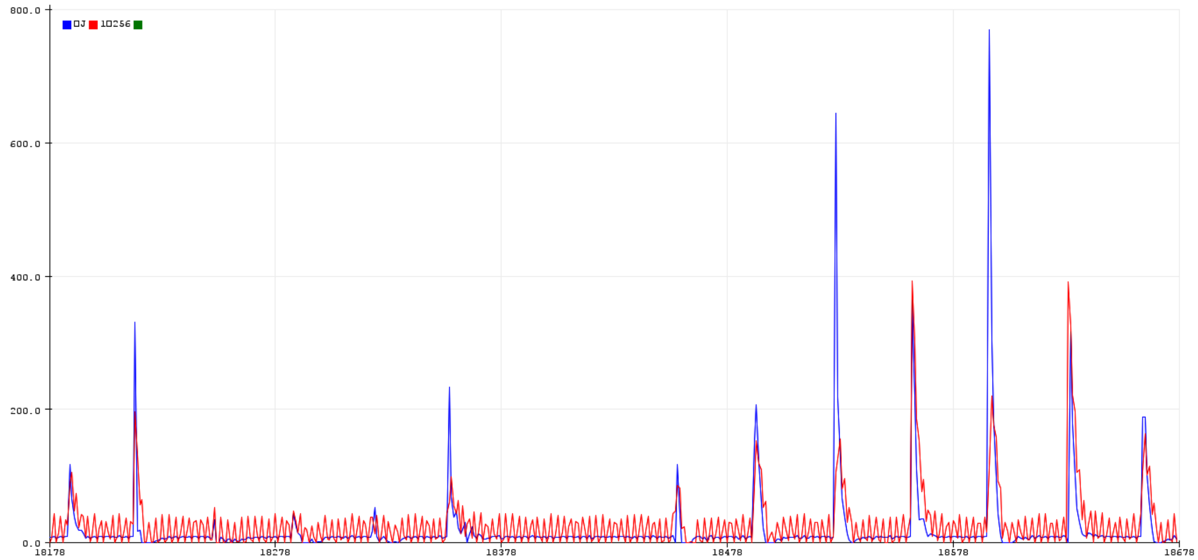
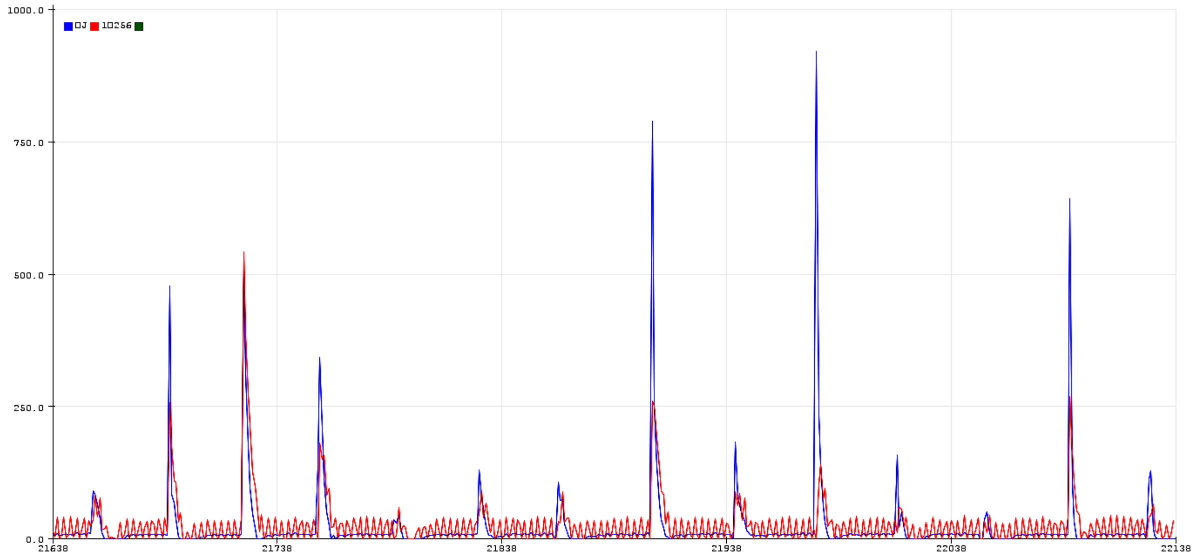
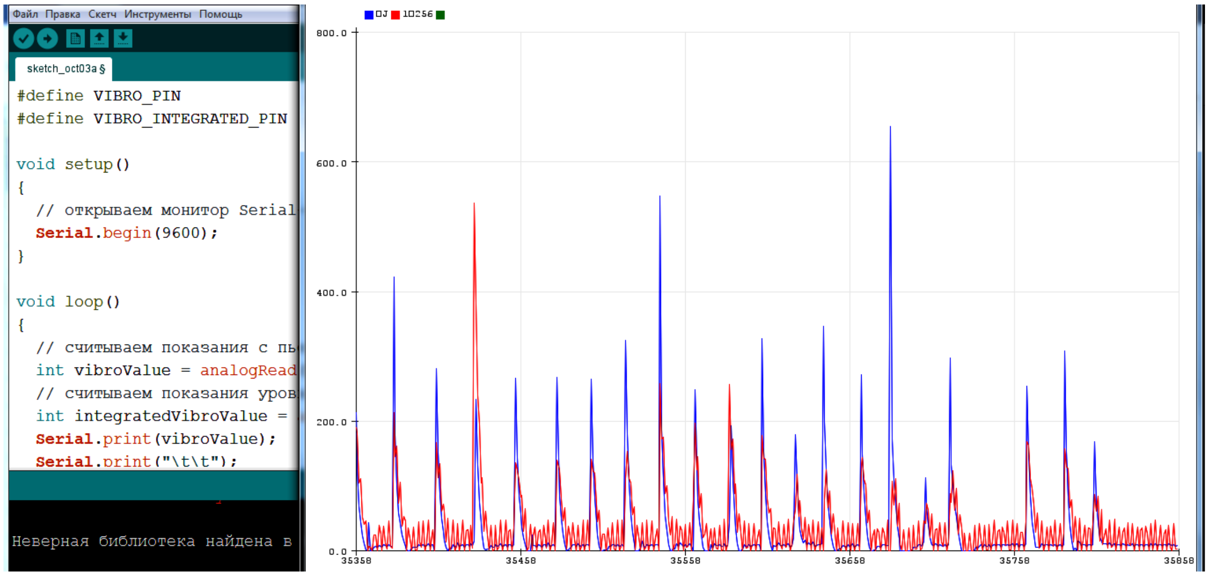


**Figure 3.** *Results of experiments of a pumping unit during vibration*









```
#define VIBRO_PIN
#define VIBRO_INTEGRATED_PIN

void setup()
{
  // открываем монитор Serial
  Serial.begin(9600);
}

void loop()
{
  // считываем показания с пь
  int vibroValue = analogRead
  // считываем показания уров
  int integratedVibroValue = 
  Serial.print(vibroValue);
  Serial.print("\t\t");
```

```
Неверная библиотека найдена в
```

In Figure 3 shows the results of experiments on the pumping unit during vibration. For this purpose, a code program was compiled to create a vibration sensor with a relay module in C⁺⁺.

## Discussion

An automated control system for protecting a pumping unit from increased vibration is an integral part of modern industry. It ensures reliable and safe operation of pumping equipment, prevents damage and loss of performance. By installing vibration sensors, monitoring and analyzing data, and alerting and responding to dangerous levels of vibration, the system provides effective protection for the pumping unit.

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# Contents

## Section 1. Biology

## Section 2. Chemistry

## Section 3. Mechanic engineering

## Section 3. Technical science in general