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ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
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Д.В. Сокольский атындағы
«Жанармай, катализ және электрохимия институты» АҚ

Х А Б А Р Л А Р Ы

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
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NAS RK is pleased to announce that News of NAS RK. Series of chemistry and technologies scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of chemistry and technologies in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of chemical sciences to our community.

Қазақстан Республикасы Ұлттық ғылым академиясы «ҚР ҰҒА Хабарлары. Химия және технология сериясы» ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Web of Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия химии и технологий» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество в глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.

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**CHEMICAL EXPRESSION OF THE STRUCTURE OF HUMIC SUBSTANCES
IN TERMS OF COMPLEX COMPOUNDS**

Abstract. Currently, the requirements for the quality of new products are increasing. In this regard, there is a need to develop new scientific approaches, namely, process mechanisms for the development of technology and synthesis of chemical compounds necessary for various sectors of the national economy. In this regard, the task was to develop mechanisms for obtaining organomineral compounds from coal mining waste. Despite the obvious importance of the problem of obtaining organomineral humic compounds: firstly, local raw materials of industrial waste from the coal mining of the Lengher deposit of the Turkestan region are used as raw materials, secondly, the industries of the agricultural sector of the country are mainly based in the Southern region, but its solution is hindered by the absence of studies of the chemistry of formation, composition, structural features of the compounds obtained and sound technological developments. A literature review on the methods of obtaining, compositions and structures of synthesized organomineral compounds (chelate complexes) allows us to conclude that the mechanism of the process of obtaining humates from coal mining waste with treatment with an aqueous solution of sodium hydroxide remains a little-studied area in chemical technology. In addition, the synthesis of new organomineral compounds is impossible without studying the composition, properties and chemical structure of the complexes formed by them. The study of the mechanism of the complexation process (chelate complexes) of the composition of «humate - aqueous solutions of sodium hydroxide» leads to an understanding of the structure of the molecule and to the development of the theory of chemical structure. The development of the mechanism of the basics of complexation (complexones-chelates), in turn, are a prerequisite for the creation of the scientific foundations of the technology for the production of complex organomineral compounds used as feed additives from industrial waste of the Republic of Kazakhstan.

Key words: humic substances; humates; extraction; complex compounds; coal mining waste.

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**КЕШЕНДІ ҚОСЫЛЫСТАРДЫҢ ТЕОРИЯСЫ ТҮРҒЫСЫНАН ГУМИНДІ ЗАТТАРДЫҢ
ҚҰРЫЛЫМЫН ХИМИЯЛЫҚ ӨРНЕКТЕУ**

Аннотация. Қазіргі уақытта өндірілетін жаңа өнімдердің сапасына қойылатын талаптар артып келеді. Осыған байланысты жаңа ғылыми тәсілдерді, атап айтқанда, ұлттық экономиканың әртүрлі салаларына қажет химиялық қосылыстардың технологиясы мен оларды синтездеу үрдістерінің жаңа тетіктерін жасау қажет. Алдымызда көмір өндірісі қалдықтарынан органоминералды қосылыстар алу әдістерін әзірлеу міндеті тұр. Органоминералды гуминді қосылыстарды өндіру мәселесінің айқын маңыздылығына қарамастан: біріншіден, шикізат ретінде Түркістан облысының Ленгір кен орнындағы көмір өндірісі қалдықтары жергілікті шикізат ретінде пайдаланылады, екіншіден, еліміздің ауыл шаруашылығы саласының салалары негізінен оңтүстік өңірде орналасқан, алайда, гуминді қосылыстардың шекті түрде қолданылуы алынған қосылыстардың түзілу химизмі, құрамы, құрылымдық

ерекшеліктері және осыған негізделген технологиялық әзірлемелер бойынша зерттеулердің жоқтығы кедергі келтіреді. Синтезделген органоминаралды қосылыстарды (комплекс-хелаттар) алу әдістері, құрамы мен құрылымы туралы әдеби шолу натрий гидроксидінің Сулы ерітіндісімен өңделген көмір қалдықтарынан гуматтарды алу процесінің механизмі химиялық технологияда аз зерттелген аймақ болып қала береді деген қорытындыға келдік. Сонымен қатар, жаңа органоминаралды қосылыстардың синтезі олар құратын комплекстердің құрамын, қасиеттерін және химиялық құрылымын зерттеуді қажет етеді. «Натрий гидроксиді гуматының сулы ерітінділері» құрамының кешенді түзілу процесінің механизмін (комплекс - хелаттар) зерттеу олардың молекулалық құрылымын түсінуге және химиялық құрылым теориясын жасауға ықпал етеді. Кешенді құру негіздерінің (хелат кешендері) тетігін әзірлеу, өз кезегінде, Қазақстан Республикасының өнеркәсіптік қалдықтарынан тыңайтқыш қоспалары ретінде пайдаланылатын кешенді органоминаралды қосылыстар алу технологиясының ғылыми негіздерін құру үшін алғышарт болып табылады.

Түйін сөздер: гуминді заттар; гуматтар; экстракция; кешенді қосылыстар; көмір өндірісінің қалдықтары.

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ХИМИЧЕСКОЕ ВЫРАЖЕНИЕ СТРУКТУРЫ ГУМИНОВЫХ ВЕЩЕСТВ С ТОЧКИ ЗРЕНИЯ ТЕОРИИ КОМПЛЕКСНЫХ СОЕДИНЕНИЙ

Аннотация. В настоящее время возрастают требования, предъявляемые к качеству получаемых новых продуктов. В связи с этим возникает необходимость в разработке новых научных подходов, а именно механизмов процессов для разработки технологии и синтеза химических соединений, необходимых для различных отраслей народного хозяйства. В связи с чем перед нами стояла задача разработать механизмы получения органоминаральных соединений из отходов угледобычи. Несмотря на очевидную важность проблемы получения органоминаральных гуминовых соединений: во-первых, к качеству исходного сырья используется местное сырье промышленных отходов угледобычи Ленгерского месторождения Туркестанской области, во-вторых, в Южном регионе в основном базированы промышленности аграрного сектора страны, однако решение ее тормозится отсутствием исследований химизма образования, состава, особенностей строения получаемых соединений и обоснованных технологических разработок. Литературный обзор по способам получения, составам и структурам синтезированных органоминаральных соединений (комплексон-хелатов) позволяет сделать заключение, что механизм процесса получения гуматов из отходов угледобычи (Ленгерского месторождения Туркестанской области) с обработкой водным раствором гидроксида натрия остаются малоизученной областью в химической технологии. Кроме того, синтез новых органоминаральных соединений невозможно без изучения состава, свойств и химического строения образованных ими комплексон-хелатов. Изучение механизма процесса комплексобразования (комплексон-хелаты) состава «гуматводный растворов гидроксида натрия» ведет к пониманию структуры молекулы и к разработке теории химического строения. Разработка механизма основ комплексобразования (комплексон-хелаты), в свою очередь, являются предпосылкой для создания научных основ технологии получения комплексных органоминаральных соединений используемых в качестве удобрительных композиций из промышленных отходов Республики Казахстан.

Ключевые слова: гуминовые вещества; гуматы; экстракция; комплексные соединения; отходы угледобычи.

Introduction. Currently, the use of environmentally friendly and safe substances in agriculture is becoming relevant. One of the directions of the future is the use of humic substances. Humic substances are high-molecular compounds of natural origin, which are formed as a result of oxidation of coal or changes in dead biomass and are shapeless formations with a chaotic structure of dark brown color, which can dissolve or swell in water [1]. These compounds do not have a single chemical formula, but it is known that their main structures are aromatic rings and functional groups (hydroxyl, carboxyl, carbonyl, alkyl and methoxyl) [2]. In addition to aromatic rings, the substance may contain polypeptide and polysaccharide fragments. Even simple compounds, such as fulvic acids, have a complex chemical structure.

Since the structure of the humic acid molecule cannot be quantified by traditional methods, researchers have developed a classification method based on solubility in alkalis and acids. Thus, humic substances are classified into three categories: humins - substances insoluble neither in acids nor in alkalis; humic acids - substances insoluble in acids, but soluble in alkalis; fulvic acids - substances soluble in acids and alkalis [3]. Humic acids are fractions of humic substances that are soluble in an alkaline medium, semi-soluble in water and insoluble in an acidic medium. This classification parameter may vary depending on the content of humic acids, pH and ionic bonds. Due to their amphiphilic nature, humic acids form micelle-like structures called false micelles in a neutral and acidic environment. This property is used for use in water treatment plants and to increase the solubility of hydrophobic preparations in water [4-5]. Humic substances contain various functional groups, the amount of which depends on the origin, seasons, climate and environmental conditions during the production of humic acids [6]. The various functions of humic substances mainly relate to the functional groups of phenols and carboxylic acid. These properties provide humic acids with many positive properties, such as improved plant growth, complex formation with heavy and variable metals, which means that they can remove heavy metals from the body and form chelated compounds. In addition, their antiviral and anti-inflammatory activity has been proven [7]. It has been proved that the presence of phenols, carboxylic acids and quinones in the structure of humic acids is associated with their antioxidant, fungicidal and bactericidal activity [8]. The chemical composition of humic substances can vary depending on geographical origin, timing, climate and biological conditions, which makes it difficult to determine its exact definition [9].

In the literature, a number of authors have a common opinion about the chemical composition of humic acids obtained from various sources [10-11]. Thus, substances prepared on the basis of humic acids contain 50% carbon, 35% oxygen and 5% hydrogen, and the remaining percentage is nitrogen and sulfur [12]. The largest amount of carbon is contained in coal and brown coal, and its content can reach 60-65%. The ability of humic acids to bind cationic metals and complexes makes them useful in various fields. Thus, they ensure the transfer of micronutrients from soil to plants and from feed additives to the body of farm animals [13]. In addition, humic acids reduce the content of heavy metals in soil, water and living organisms [14].

The main source of humic acids is brown coal. Its reserves in Kazakhstan amount to 34 billion tons [15]. It was found that brown coal differs in chemical composition compared to other sources of humic acids. It contains various trace elements that can be used in the production of animal feed additives [16].

The main methods of isolation of humic substances include alkaline extraction with ammonia solutions or potassium / sodium hydroxides. Such methods convert humic substances into water-soluble salts, that is, potassium or sodium humates with high biological activity. This method is practically waste-free, therefore it is widely used in many countries [17]. Analyzing the literature data on the chemical composition of humates, it can be noted that the most effective method of their application is the isolation of humic acids and their bases from brown coal, including humates. Humates obtained from brown coal allow them to be used in small quantities, everywhere and regardless of the time of year, due to the high concentration of nutrients and biologically active substances in them [18]. Most humic substances are chemically added to inorganic components (clay and oxides). An important feature of humic substances is that they can combine with metal ions, oxides, clay minerals and interact with organic compounds such as alkenes, fatty acids, capillary active substances and pesticides [19].

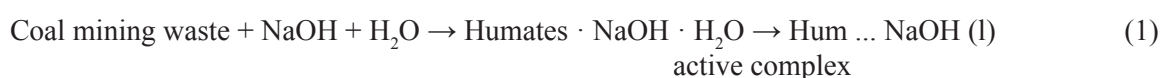
Methods and Materials. Extraction of humic substances was carried out according to the following method: weighed the bulk of coal mining waste from the Lengersky deposit of 1.0 g. The bulk is placed in a conical flask A with a capacity of 250 cm³, 100 cm³ of an alkaline solution of sodium pyrophosphate is poured and stirred for 1 hour using a mechanical shaker. The suspension is centrifuged for 15 minutes. The solution is decanted by collecting in a conical flask with a capacity of 1000 cm³. The insoluble residue is washed twice with 100 cm³ of sodium hydroxide solution. The suspension is centrifuged after each washing, collecting the washing solution into the flask B. The washed precipitate is transferred to flask A, 100 cm³ of sodium hydroxide solution is added and heated for 2 hours in a boiling water bath. After cooling to room temperature, the contents of the flask A are centrifuged for 15 minutes. The solution is decanted and collected into a flask B. The undissolved residue is washed twice with 100 cm³ of sodium hydroxide solution. The suspension is centrifuged after each washing, collecting the washing solution into the flask B.

Ultimately, sodium humate with the following chemical composition was used to chemically express the structural features of synthesized humic substances from coal mining waste from the point of view of the theory of complex compounds (Table 1).

Table 1 – Elemental composition of sodium humate synthesized from coal mining waste

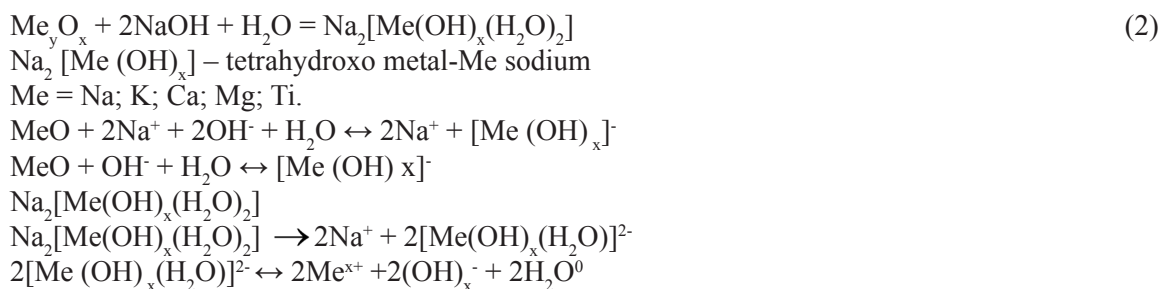
Element	Weight, %	In terms of oxides, %
C	42,72	-
O	38,17	-
Na	17,14	23,10
Al	0,34	0,64
Si	0,79	1,68
Ca	0,23	0,32
Fe	0,49	0,70
Ti	0,12	0,20

Results and Discussion. To describe the experimental data obtained and the established regularities, the mechanism of chemical reactions under selected optimal conditions is proposed. At the first stage, a mechanism is proposed for the process of interaction of coal mining waste according to their experimentally established composition, with an aqueous solution of sodium hydroxide in order to obtain Humates (Hum.), where the process can be described by the following general chemical equation:



Agreement with experimental data can be obtained if we assume that the process of formation of humates is carried out by reactions (1), under the condition of formation of an active complex Hum ... NaOH (I), the chemical structure and composition of which is the formation of intra-complex compounds (chelates). Experimental data can be explained that some nonmetals containing double-charged metal ions (Ca^{2+} , Mg^{2+} , ...) in coal mining waste: Si, S, N... and p-Al..., d-Ti, Fe.... - elements, as well as containing several organic functional groups form strong complexes that are of great practical importance, i.e. complexons are polydentant ligands.

The proposed mechanism at the first stage corresponds to the formation of an active complex (1), which are intra-complex organic compounds (chelates). According to the established composition of coal mining waste, their following possible chemical reactions with an aqueous solution of sodium hydroxide are proposed in order to obtain humates, which are characterized by the following equations:



where, the stability of complex compounds is determined by the instability constant, according to the theory of complex compounds [20], the smaller this constant, the more stable the complex compound. A detailed scheme of the formation of a complex compound is presented in the Figure 1.

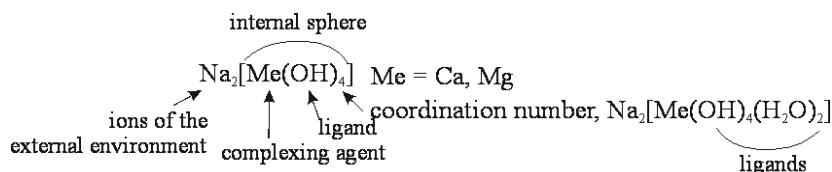


Figure 1. The structure of the complex compound

According to the theory of complex compounds, the coordination number (c.n.) of the complexing agent reflects the number of bonds that the complexing agent forms with ligands. The coordination number can vary from 2 to 12 [21]. The most common c.c. are 4 and 6. In most cases, the rule is fulfilled: the coordination number is twice its charge.

In this case: $Me^{2+}(Ca, Mg) = c.n. - 4, 6$; $Me^{3+}(Al, Fe) = c.n. - 6$; $Me^{4+}(Ti) = c.n. - 8$.

It is known [22] that metals in the composition of humic substances indicate the formation of simple or complex salts with the participation of various organic groups in their composition:

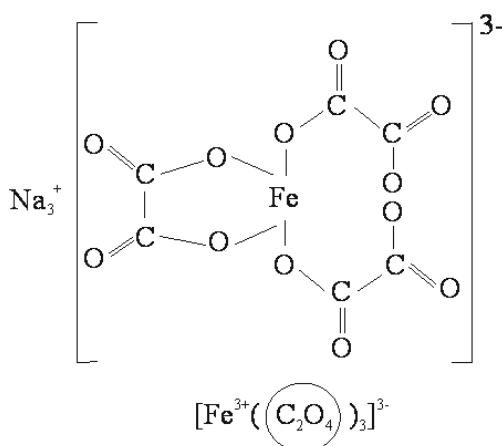
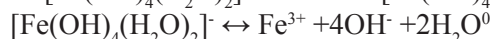
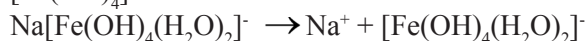
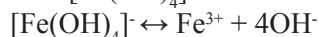
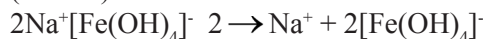
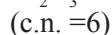
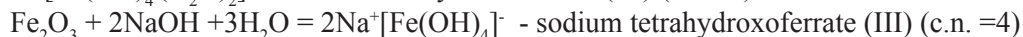
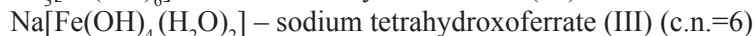
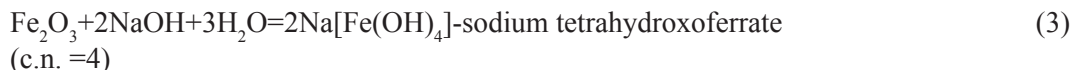


Figure 2. The structure of the sodium tetrahydroferrate

The ability of individual chemical elements in the composition of synthesized humic substances to form complex compounds is determined by the structure of the electron shell of their atoms, ionic radii and valence. For example, one of the compounds in the composition of humic substances is silicon dioxide. Below is the mechanism of formation of a complex compound - sodium disilicatodiaquanaty:

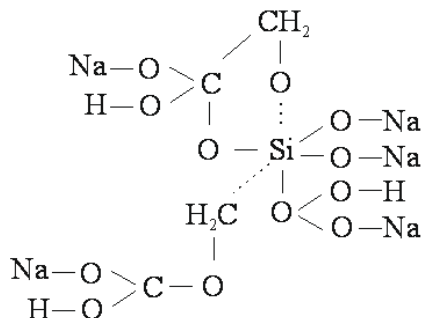
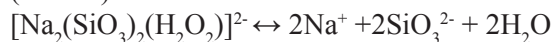
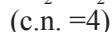
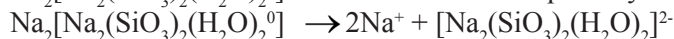
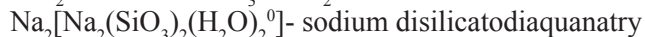
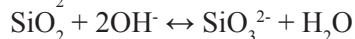
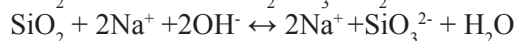


Figure 3. The structure of $Na_2[Na_2(SiO_3)_2(H_2O)_2^0]$

Polydentant ligands - they consist of several donor atoms and have several positions in the coordination sphere. Polydentant ligands often form chelates in which the ligand and the central atom form cycles (Figure 4).



Figure 4. Structure of polydentant ligands

When considering the ability of complexation of humic substances, it is necessary to take into account the interaction of the organic part of humates with various metal cations can be carried out under conditions of hydrogen substitution of carboxyl groups, adsorption and sedimentation.

The substitution of hydrogen ions in the interaction with metals can occur with the cationic and anionic part of humic substances. If the metal enters the anionic part into the structure of humates, then it is connected by means of coordination chemical bonds and leads to the formation of chelate-containing compounds [23].

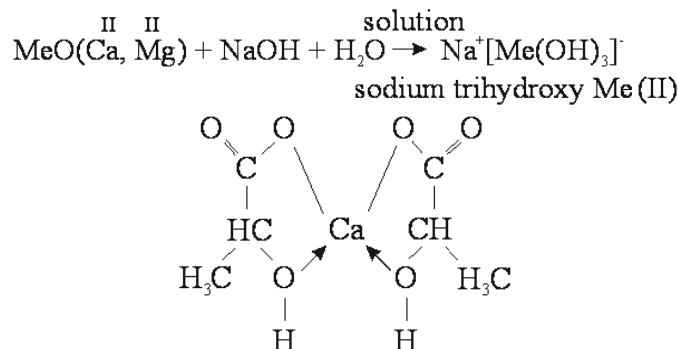
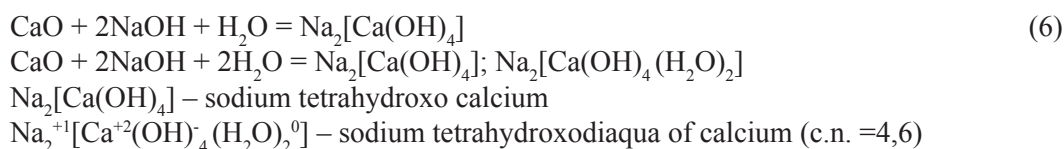
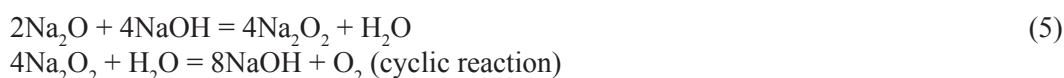


Figure 5. Calcium-lactate - chelates complex

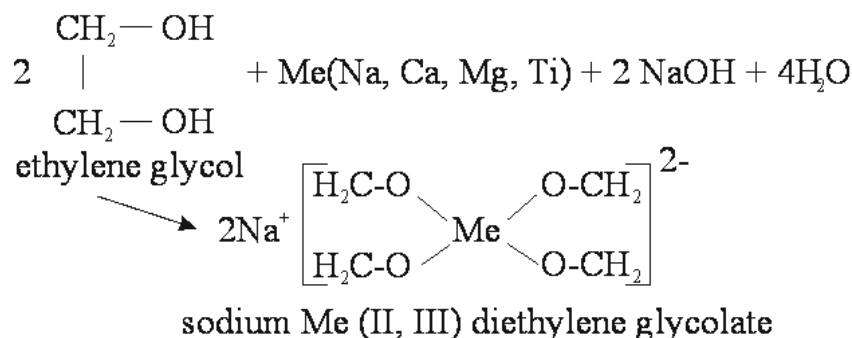
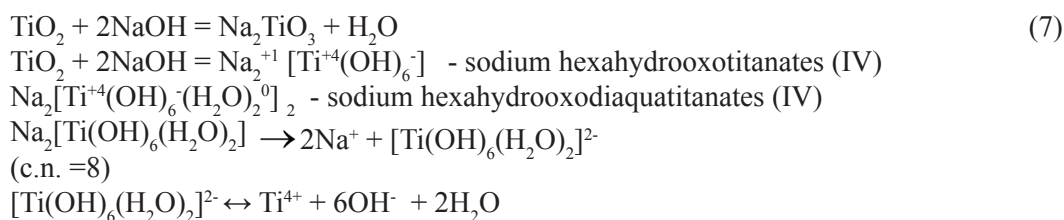


Figure 6. Intra-complex salt $\text{Me}(\text{Na}^I, \text{Ca}^{II}, \text{M}^{II}, \text{Ti}^{IV})$ with ethylene glycol

The chemical structure of the reaction product - the active complex Hum ... NaOH (I) can be represented in the following form:

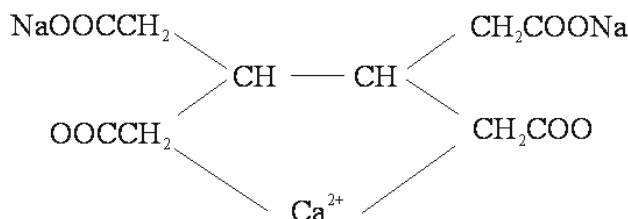


Figure 7. The chemical structure of Hum ... NaOH (I)

Each ethanol molecule is a functional organic compound, an intra-complex compound that forms 2 bonds with Me^{2+} - one by exchange, the other by donor-acceptor mechanism [24].

In the system under study, trace elements are reactants contained in coal mining waste, i.e. starting substances, and the chelating agent is a 1% aqueous solution of sodium hydroxide. The mechanism of interaction: coal mining waste and NaOH (aqueous solution) can be expressed in the following summary expression:

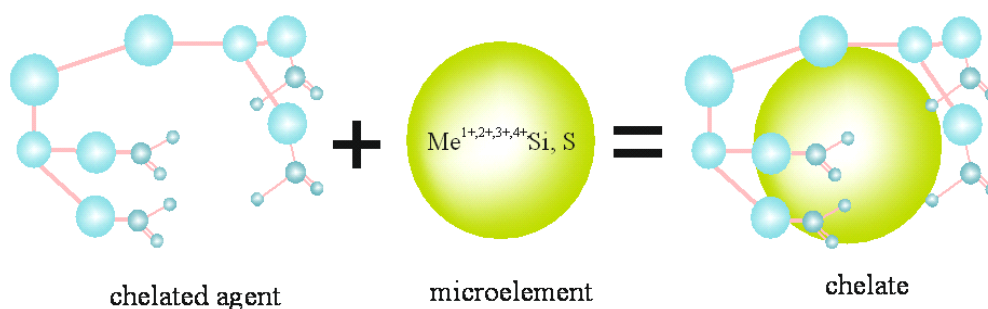


Figure 8. Formation mechanism of chelate substances

where, the formation of a complex ion can be explained by the presence of vacant orbitals in metal cations at the s-, p-, d- and f-sublevels, which participate in the donor-acceptor mechanism of bond formation in complex compounds. The ability to complex formation decreases in the series: $f > d > p > s$, and some non-metals can also be complexing agents: Si, S, N,....

According to the proposed mechanism, when the studied reagents “Coal mining waste + NaOH + H₂O” are used, cyclic (chelated): stable complex compounds of metals and nonmetals with polydentant ligands in which the central atom is a component of the cyclic structure are formed.

Coal mining waste and a 1% aqueous solution of sodium hydroxide form chelated (wedge-shaped) complex compounds – these are cyclic compounds in which the central atom in our case is two-charged metal ions (Ca^{2+} , Mg^{2+} , ...) some nonmetals: Si, S, N ... and p-Al..., d-Ti, Fe-.... elements, and also containing several organic functional groups is part of one or more cycles of covalent bonds according to the donor-acceptor or exchange mechanism. Polydentant - ligands with a coordination capacity of 3, 4, 6 [25]. they are attached to the complexing agent by means of several atoms and functional organic compounds. Based on the developed chemical reactions, the type of hybridization, spatial orientation and geometric configuration of the obtained complex compounds in the studied transformations were determined (Table 2).

Table 2 - Spatial configuration of the obtained complex compound

Size	Orientation	Type
$Na_2[Me(OH)_4]^{2-} \rightarrow sp^3, sd^3$ where $Me(Ca^{2+}; Mg^{2+})$ $Na_2[Na_2(SiO_3)_2(H_2O)_2]^{2-}$ $Na_2[Na_2(SO_3)_2(H_2O)_2]^{2-}$	4	tetrahedron
$Na_3[Fe(OH)_6]^{3-} \rightarrow sp^3, d^2$ d^2, sp^6 $Na_3[Al(OH)_6]^{3-}$	6	octahedron
$Na_2[Ti(OH)_6(H_2O)_2]^{2-} \rightarrow sp^3, d^2$ d^2, sp^3	8	octahedron

Conclusion. For the first time it was proposed that during the chemical interaction of coal mining waste (Lenger deposit, Turkestan region) and 1% aqueous solution of sodium hydroxide at the first stage of the formation of humates occurs by the mechanism of formation of an active complex, which in composition and chemical structure is an internal complex organic compounds - complex chelates. In accordance with the proposed mechanisms, the central atom - double-charged metal ions (Ca^{2+} , Mg^{2+} , et al.) enriched with additives contained in the formed sodium humate form a cyclic (chelated) complex compound that is part of one or more cycles of covalent bonds by donor-acceptor/exchange mechanisms Si, S, Al, Ti, Fe, organic functional groups and are an integral part of the central atomic cyclic structure. The internal complex organic compound contains polydentant ligands. In the cyclic (chelated) structure, metals and nonmetals form the main product, entering into a chemical bond with the complexing agent by means of stable complex compounds with polydentate ligands, several atoms with a coordination number of 3, 4, 6 and functional organic compounds.

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