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Modern data analysis technologies used for geomechanical monitoring. Review

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ABSTRACT

The paper considers the possibilities of modern technologies and software that make it possible to create continuity of geomechanical monitoring of man-made objects from shooting in automatic mode, robotic surveillance systems, transmitting information over the Internet to cloud storage, to performing stability calculations, determining the parameters of displacement and deformation of slopes of ledges and sides of quarries. The development of modern technologies for collecting and processing information allows the use of artificial neural networks that are adapted for modeling geodetic deformations. Technogenic objects, which are very complex systems, have a huge number of external factors affecting the stability of the mountain range, so it becomes incredibly difficult to take into account and determine the amount of displacement and deformation. Due to the complexity and variety of influencing factors, it becomes necessary to use a new system for assessing the state of objects, called "neural networks". The training of such a system is based on the already available research results collected during the direct operation of industrial enterprises. Neural networks can become an alternative to various methods of describing deformation processes, especially in the continuous monitoring of man-made objects, where there is no a priori knowledge of the underlying deformation processes. For effective monitoring and forecasting of deformation processes at a mining enterprise, a multiparametric monitoring method is needed, which includes a comprehensive system based on GPS measurements, supplemented with data from sensors for changes in water level and changes in stresses and deformations of the array. The results of automated survey and data recording sent to the cloud storage are distributed using "Big Data" technology and analyzed by geoinformation systems. In turn, the adaptation of neural networks to model deformations allows specialists to obtain a good alternative to the description of structural deformations of the mountain range.

Keywords: The concept of the "Internet of Things", deformation monitoring, "Big Data", neural networks, analytical models, modeling of deformation processes.

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Introduction

Application of the concept of the "Internet of things" in the organization of observations of the object.

The automation of geodetic observations began in the 1980s, when personal computers and analog signal modems appeared, which were used by engineers to control electronic sensors and record sensor data on magnetic drives. At this time, time measurements were increasingly replaced by constant and continuous observations in time. The prerequisite for automatic measuring digital systems in geodesy was the availability of suitable software for touch control and data processing [1]. These strain monitoring systems have been developed as isolated applications for individual

personal or industrial computers with limited or no network functions.

Even today, more modern systems lack such functions as an open network and data interfaces for machine-to-machine interaction with third-party software, which makes it difficult to integrate them into sensor networks [2]. To coordinate measurements over large areas, monitoring systems must be integrated into sensor networks.

The possibilities of the "Internet of Things" concept and modern technologies make it possible to simplify access to the network in engineering geodesy and surveying without costs and technical problems [3].

The emergence of small single-board computers, such as the Raspberry Pi, made it possible in a short time to create inexpensive middleware for monitoring and controlling tasks using rapid prototyping methods. These embedded boards are full-featured computers with an ARM or MIPS processor, memory, and I/O interfaces. In addition, any application, for example, the monitoring software discussed, can be launched (Fig. 1). The concept of embedded sensor nodes is also applicable to portable Android devices such as smartphones and tablet computers. They can provide the necessary interfaces for connecting sensors, as well as mobile Internet access via 3G/4G. The reuse of such universal devices can reduce the effort and cost of implementing sensor networks [3].

At the moment, foreign research groups and universities are already working on the development of this direction. The result of such work is a German system called OpenADMS [4]

It consists of several components:

- Open ADMS Control is a software for personal computers designed to receive short–term data from sensors.

- Open ADMS Web is a system designed to work with a server, its main task is to work with a complex of sensors and devices in the long term.

This system allows real-time observations of the reference points of the observation stations of the geomonitoring system and reflects on the graph the displacement and changes in the coordinates of the observation point, shown in Fig. 2.

The advantage of using this system can be called the continuity of the work of the surveying department. While specialists are in the field, reproducing the shooting or it is performed automatically by robotic surveillance systems, the data obtained is immediately sent via the Internet (or an internal LAN) to cloud storage. The data from the repository is used to perform further calculations of stability, the volume of work performed, etc.

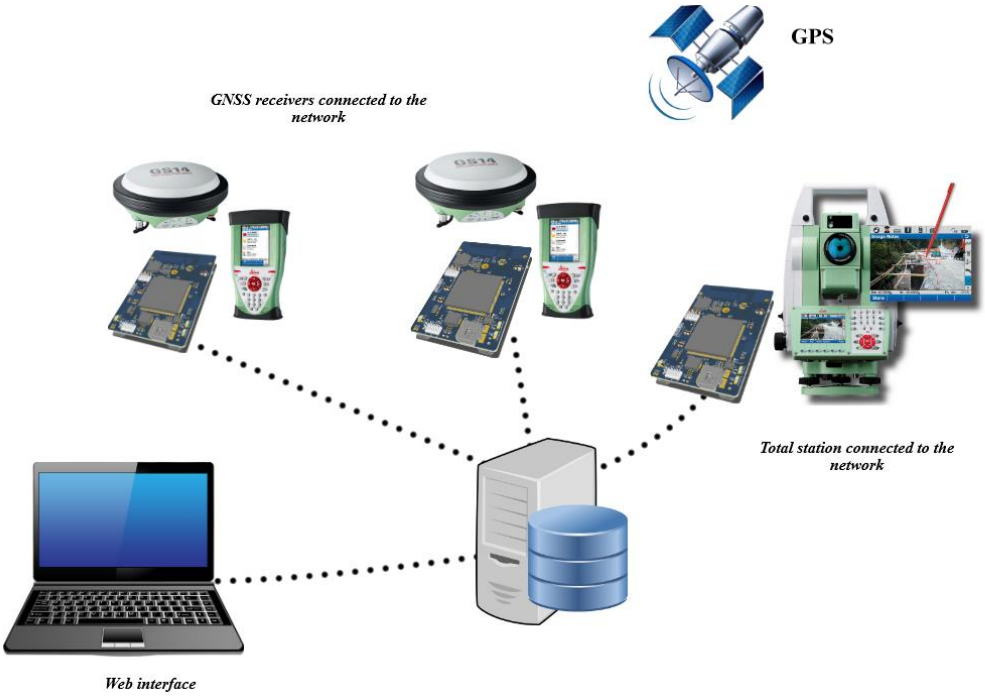

Figure 1 - The scheme of the "Internet of Things"

Figure 2 - Observation of the displacement of the point coordinate using OpenADMS

Big Data and prospects of application in desk work

The term "big data" (BD) was "officially" introduced by the Oxford English Dictionary in 2013. He associates the term BD with a large data set that is (almost) impossible to manage — process using "traditional" tools [5].

BD can carry "big errors", such as lack of consistency and reliability, "false" data, noise, lack of representativeness, incomplete information, etc.

Currently, most of the "big data" consist of spatial data, i.e. discrete representations of continuous phenomena. Spatial data is represented by the following basic models:

a) raster (grid): satellite images are good examples of raster data;

b) vector: consists of points, lines, polygons, and their combined (or multi-) analogs;

c) network: graphs consisting of spatial networks form another important data type used to represent road networks.

The problem of working with big data arises when shooting large objects with a high level of detail. The total amount of information about the object can reach huge volumes, and every year it will only grow exponentially. The main task of Big Data is to work with such a volume of data, their analysis, and processing. Within the framework of this concept, cloud storage, database software, machine learning, etc. are widely used.

Application of neural networks

The latest interesting developments in the direction of software development are the method of machine learning and the use of neural network technology. Technogenic objects are very complex systems that have a huge number of external factors, which makes it incredibly difficult to take them into account, so mathematical models have a discrepancy with the established forecasts of their condition [[6], [7]].

Due to the complexity of the influencing factors, when trying to take into account most of the conditions, a model is obtained that is difficult to fully describe with dependencies. For this reason, it becomes necessary to use new methods for assessing the state of objects, using a system called "neural networks" for this purpose. Though they are in fact artificial neural networks, they are often called neural networks or nets which are basically IT systems that mimic biological neural networks [8].

The training of such a system is based on the already available research results collected during the direct operation of industrial enterprises. Artificial neural networks are adapted for use in modeling geodetic deformations (Fig.3).

Surveyors and surveyors have long been faced with the problem of finding effective solutions to approximation functions that determine the

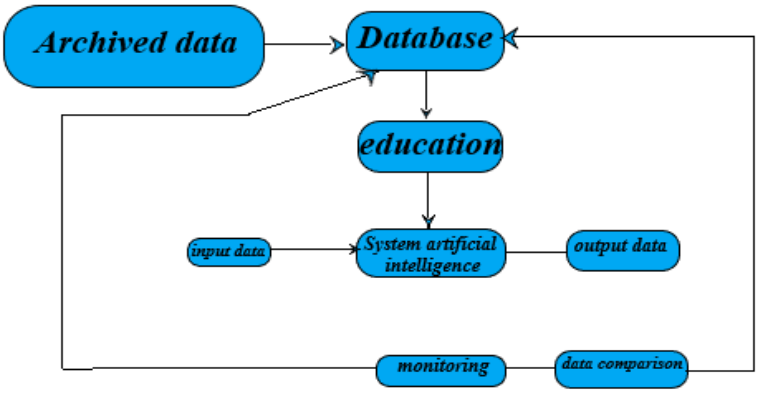

Figure 3 - Neural network operation diagram

amount of displacement and deformation, especially when working with continuously controlled processes. Most solutions are obtained in the time domain, since measurements are currently obtained online in the form of continuous or discrete time sequences.

This system has a number of features:

- The system constantly accumulates data and improves itself, which over time will improve the quality of creating an object model, and will allow analysis under changing operating conditions.

- It will be impossible to make clear dependencies within this system, since it is closed, and does not allow the user to see on the basis of which calculations such a calculation was obtained, however, the user can still make a comparison based on the data entered and the result obtained.

- The real physical model may not fully correspond to the one that was built by the neural network.

The adaptation of neural networks to model deformations allows specialists to obtain a good alternative to the description of structural deformations. Certain parameters, in this case, weights, inherently describe the mapping between input and output data, but cannot be used in any other way as a representation of a typical mathematical function for the deformation process. It is very important to note that the results of using neural networks strongly depend on the choice of both input and output data, as well as the architecture of the network used, since they are able to learn anything. Figure 4 shows graphs of real measurements and models built by a neural network trained by researchers from the Institute of Geodesy and Photogrammetry of the Braunschweig Technical University. In the left part of the graph, the parameters are adjusted, after which the neural network makes its own forecast regarding further changes in the coordinates of the geodetic point. On the graph, in the right part of it, you can track a large degree of convergence of real measurements and the model of changing the coordinates of a point built by a neural network.

Neural networks also have a disadvantage, which is that there is no single analogous solution for any given set of input-output data, since the parameters determined depend on various settings performed during the learning process, which rely solely on personal human judgment. However, neural networks can become an alternative to methods of describing deformation processes, especially in the continuous monitoring of man-made objects, where there is no a priori knowledge about the underlying deformation processes. Thus, they can serve as an addition to the existing methodology for modeling deformation processes.

Organization of a comprehensive monitoring system using the latest technologies

The issues of creating an automated geomechanical monitoring system are considered in order to study the state of stability of the Western and Eastern quarries of LLP "JV "Alaigyr" based on the use of GPS equipment and software.

The Alaigyr deposit is located in the eastern part of the Uspenskaya crumple zone. Devonian and carboniferous deposits, and subvolcanic and dike formations take part in the geological structure of the deposit [10]. Of the magmatic formations within the deposit, the subvolcanic body of ore-containing high-potassium liparite porphyries is of the greatest interest. A total of 20 dyke bodies have been identified at the deposit. The Alaigyr lead deposit is localized within the subvolcanic body of liparite porphyries. Since the ore mineralization along the strike has

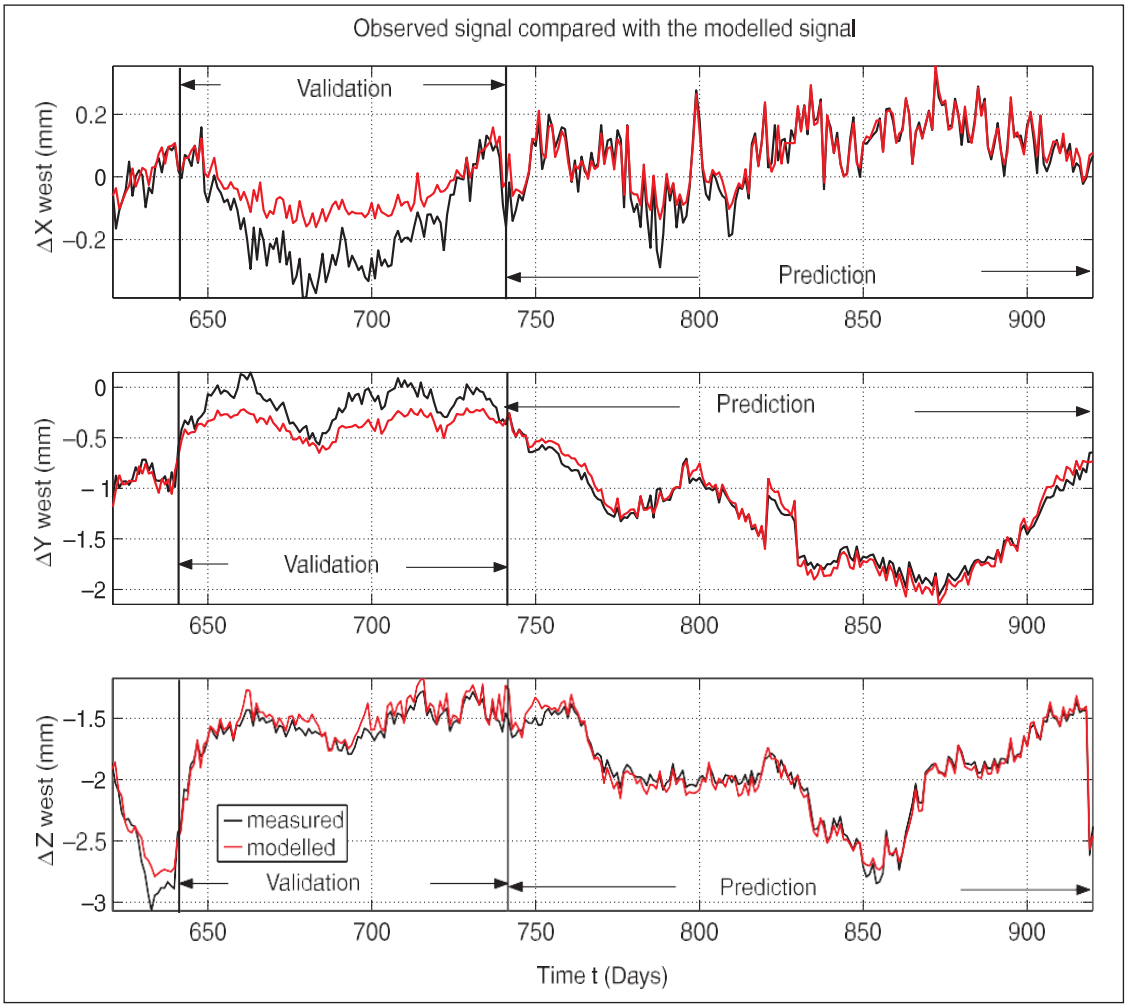

Figure 4 - Graph of comparison of neural network data with real measurements

Table 1 – Summary of the recommended parameters of the ledges of the Eastern quarry

Cut	Type of breed	Slope angle of the ledge, degree	Ledge height, m
I-I	Clay	40	12
	Weathering Crust	50	12
	Carbonaceous-siliceous shales	65	24
	Terrigenous siltstones	65	24
II-II	Clay	40	12
	Rhyolite porphyries	65	30
III-III	Weathering Crust	50	12
	Rhyolite porphyries	65	30
	Beresitized porphyry	65	30
	Tufopeschaniki	50	12
IV-IV	Weathering Crust	50	12
	Rhyolite porphyries	65	30
	Beresitized porphyry	65	30

interruptions, the deposit is conditionally divided into three sections: Western, Middle, and Eastern.

During the exploration of the deposit, 11 major tectonic disturbances were revealed [10]. The most ancient discontinuous disorders complicating the folded structure are consonant longitudinal (sublatitudinal) violations such as thrusts or interplastic disruptions. Violations of this type are characteristic of the entire Assumption crumple zone. A linear type weathering crust has been developed at the deposit, confined to crushing zones of the type of inter-plastic breakdowns between liparite porphyries and host rocks. Weathering crust rocks are represented by intensely fractured rocks, often decomposed to the state of structural and structureless clays.

The stability of the sides and ledges of the Alaigyr deposit is influenced by a huge number of factors, such as the presence of tectonic disturbances, a developed zone of weathering crust, intense fracturing of rocks, waterlogging of the deposit, physical and mechanical properties of rocks, technological features of mining, etc.

In 2018, Mining Research Group LLP carried out work on the geomechanical justification of the parameters of the quarries of the mining complex at the Alaigyr deposit. Analysis of the simulation results shows that the least stable areas are the upper ledges composed of clays (SRF = 0.63), which indicates the instability of the sides and ledges of the Eastern Quarry. The simulation results are summarized in Table 1.

Based on the Alaigyr field development project, a combined scheme for monitoring the stability of the sides of the quarry and ledges was chosen.

To monitor the stability of the sides, a GPS base station will be used, which is installed on the roof of the production and administrative building. Four observation profiles and eight scoring reference points are fixed on the sides of the quarries to install a GPS receiver. (Figure 5, 6)

Also, based on the geomechanical justification of the quarry parameters, the location of the robotic electronic total station is selected.

Geomechanical control at the quarries is carried out in order to obtain reliable information about the condition of the sides of the quarry at various stages of field development. One of the ways to ensure such control is to conduct instrumental observations, on the reliability of the implementation of which the adequacy and timeliness of decision-making on emerging deformations of the instrument array depends. Untimely response to emerging deformations can lead to the death or loss of the working capacity of people and increase production costs.

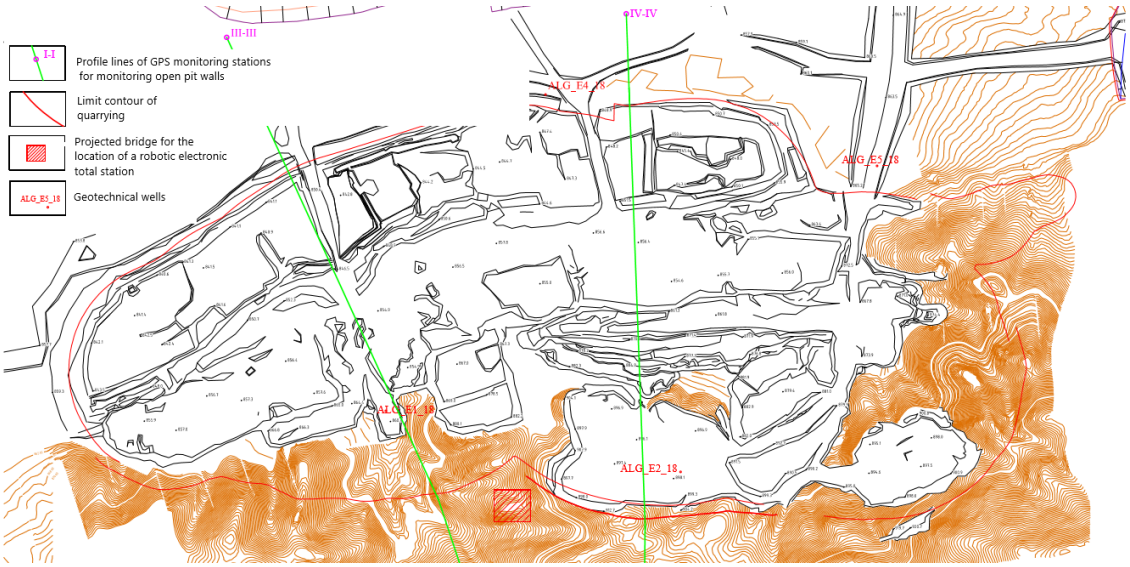

Figure 5 - Layout of profile lines for monitoring the stability of the eastern side of the quarry

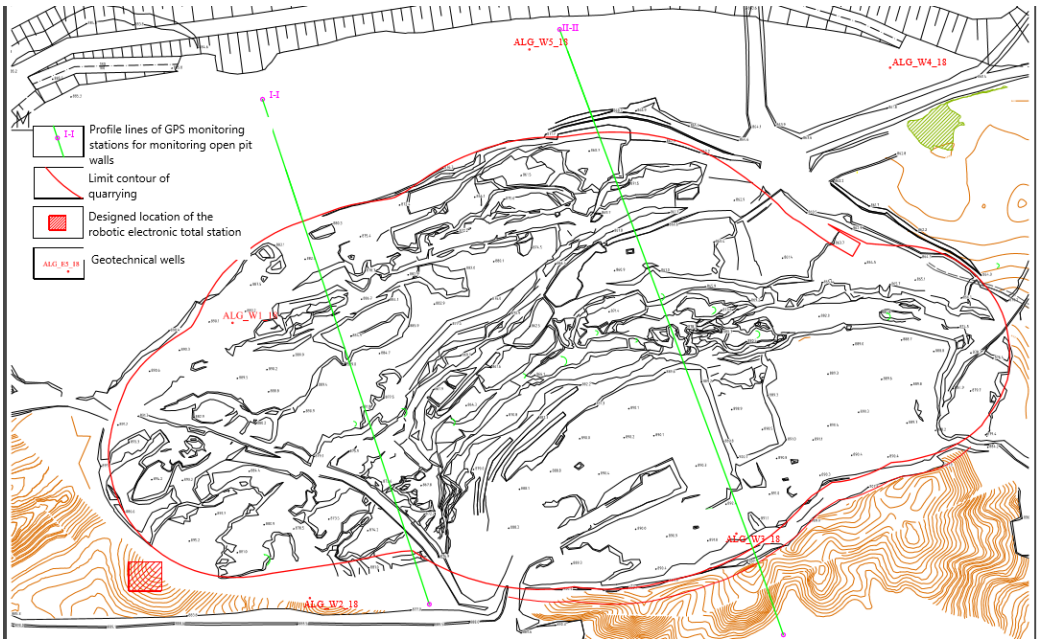

Figure 6 - Layout of profile lines for monitoring the stability of the western side of the quarry

Currently, there is a large amount of data, both in the field of hardware layout of observational surveying networks, and software products that provide analysis and processing of incoming information from deformation sensors. One of the main manufacturers whose GPS equipment is used in the creation of information collection and processing systems (GPS receivers and communication equipment) are Leica Geosystems, Trimble, Garmin, etc [9].

In the field of software that allows you to fully automate the control mode of deformation sensors, collect and archive statistical data, process data in real-time, as well as notify about exceeding permissible values of deformations, the following companies have achieved the greatest success: InteTrak (Orion Monitoring Systems, Inc); 3D Tracker (Condor Earth Technologies); GPS RTK software (Geodetic Research Laboratory (GRL) at UNB) [11].

Using high-precision equipment operating around the clock in real-time with an advanced warning system helps to secure mining operations (for example, to remove equipment and field workers from the zone of probable collapse). In view of the fact that the mountain range is usually heterogeneous, the function of the development of the deformation rate is often not linear in nature. In this regard, high-precision instrumental observations with short intervals between measurements are necessary to determine the transition point of ordinary displacements to the critical zone [1].

This multiparametric remote monitoring system can monitor various characteristics of an unstable landslide on a large scale before it collapses in various aspects, which gives the geomechanical service of the enterprise valuable time to prepare anti-landslide measures [12]. According to the different slope characteristics, different parameters can be combined and different types of sensors can be selected. Then the data is transmitted to the control room using wireless communication technology. In the control room, correct conclusions are given through the system's data analysis system and the expert's experience analysis, which can play a real role in predicting and predicting deformation processes.

To reproduce the real effect of monitoring and forecasting on an unstable slope, a multiparametric monitoring method is needed [[13], [14]]. Based on the ideas presented, a combination of several monitoring methods is proposed [15]. A comprehensive monitoring system based on GPS monitoring is being created, which is complemented by touch monitoring [16].

Firstly, the stability of the slope of the ledge is influenced by many factors. To collect information affecting the stability of the slope, a multiparametric and multi-device monitoring system based on the Internet of Things is proposed.

Secondly, the monitoring cycle of the slope monitoring project is long, so it requires little real-

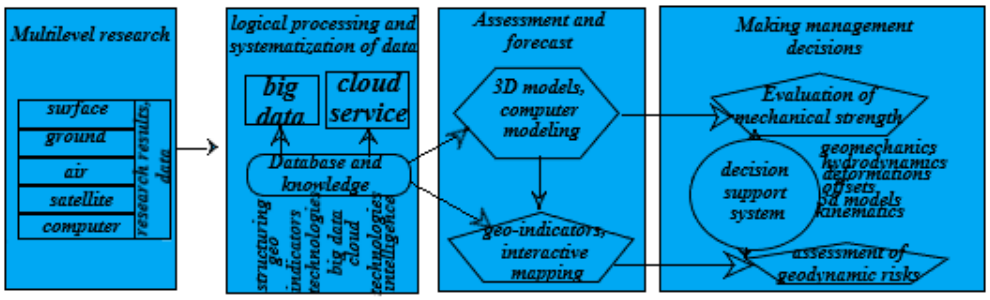

Figure 7 - Geomechanical monitoring system

time data. However, due to the specifics of the monitoring environment, it has high requirements for energy conservation, scalability, and reliability of the wireless transmission network [[6], [7]].

Thirdly, the efficiency and accuracy of monitoring data play an important role in predicting the stability of the slope of the ledge and the side of the quarry [17]. Therefore, the data must be properly processed and classified. The key to the data management process is to extract useful data from the array of monitoring data that are useful for the forecasting process [18]. Monitoring data is transmitted to the remote monitoring information management center using ZigBee technology. A large-scale information management system for slope monitoring is being created (Fig.7). They manage a database of ground and deep displacements, a database of attributes of water level changes, and a database of attributes of stress and strain changes. Through Internet technologies, geomechanics can view data in each database. To make it easier for users to manage the application, connection, device, and other contents of the intermediate service components of the Internet of Things, an Internet-based web services system is used [[19], [20], [21], [22]].

Wireless sensor network data transmission and middleware management are implemented respectively. The Access database stores information about hardware, information about applications, and so on. The Wireless Sensor Network Data Interface (API) packages wireless sensor data into an interface and provides a call to the user. It is economically feasible to use such a system at large facilities where there is a large amount of data and work performed. Due to the automation of the process with the help of robotic devices and sensors, data acquisition is possible in automatic mode [[23], [24]]. The results of the survey and data recording sent to the cloud storage will be distributed using the "Big Data" technology, after which further work with them is possible using geoinformation systems. Potentially, neural network technology can be included in this complex, but training the system will take time and require large computing power [25].

Currently, a database is being created in order to obtain reliable information about the condition of the sides of the quarry at various stages of field development, taking into account the complex mining and geological conditions of development and the results of instrumental observations of the developed integrated monitoring system based on GPS monitoring, which is supplemented by sensor monitoring.

A similar approach can be applied to large facilities such as the Ekibastuz basin coal pits, the Vasilskovskoye gold deposit, the deep pits of the Sokolovo-Sarbay Mining and Production Association, and other enterprises of the mining industry of the Republic of Kazakhstan [26]. In the end we should not forget that beyond the advantages big data has it is also like "(...) the new plutonium. In its natural state it leaks, contaminates, harms. Safely contained & harnessed it can power a city" [27].

Conclusions

Modern technologies and capabilities of the "Internet of Things" concept make it possible to simplify access to the network in engineering geodesy and surveying without costs and technical problems. The German OpenADMS system, based on working with a complex of sensors and devices, allows real-time observations of the reference points of the observation stations of the geomonitoring system and reflects on the graph the displacements and changes in the coordinates of the observation point.

The total amount of information about the object can reach huge volumes, and every year it will only grow exponentially. When shooting large

objects with a high level of detail, the main task of Big Data is to work with such a volume of data, their analysis, and processing.

Monitoring of man-made objects includes taking into account the complexity of influencing factors, while most of the conditions are difficult to fully describe with dependencies. For this reason, it becomes necessary to use new methods for assessing the state of objects, using a system called "neural networks" for this purpose. The adaptation of neural networks to model deformations allows specialists to obtain a good alternative to the description of structural deformations.

Neural networks can become an alternative to methods of describing deformation processes, especially in the continuous monitoring of man-made objects, where there is no a priori knowledge about the underlying deformation processes. Thus, they can serve as an addition to the existing methodology for modeling deformation processes

The technology of the "Internet of Things" and "Big Data" can really improve the situation, simplify the way of data collection, bringing desk work to almost automatic execution, but the use of these technologies does not yet have a sufficient practical basis.

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ТҮЙІНДЕМЕ

Жұмыста автоматты режимде түсіруден, роботтандырылған бақылау жүйелерімен техногендік объектілердің геомеханикалық мониторингінің үздіксіздігін құруға, интернет желісі бойынша ақпаратты бұлтты қоймаға беруге, орнықтылық есептеулерін орындауға, карьерлердің кемерлері мен ернеулері еңістерінің жылжу және деформация параметрлерін анықтауға мүмкіндік беретін қазіргі заманғы технологиялар мен бағдарламалық қамтамасыз етудің мүмкіндіктері қарастырылды. Ақпаратты жинау мен өңдеудің заманауи технологияларын дамыту геодезиялық деформацияларды модельдеуге бейімделген жасанды нейрондық желілерді пайдалануға мүмкіндік береді. Өте күрделі жүйелер болып табылатын техногендік объектілерде массивтің тұрақтылық жағдайына әсер ететін көптеген сыртқы факторлар бар, сондықтан қозғалыс пен деформацияның мөлшерін ескеру және анықтау өте қиын. Әсер етушілердің күрделілігі мен алуан түрлілігіне байланысты "нейрондық желілер" деп аталатын объектілердің жағдайын бағалаудың жаңа жүйесін қолдану қажет. Мұндай жүйені оқыту өнеркәсіптік кәсіпорындарды тікелей пайдалану кезінде жиналған қолда бар зерттеу нәтижелеріне негізделген. Нейрондық желілер деформациялық процестерді сипаттаудың әртүрлі әдістеріне балама бола алады, әсіресе олардың негізінде жатқан деформациялық процестер туралы априорлық білімі жоқ техногендік объектілерді үздіксіз бақылау кезінде. Тау-кен кәсіпорнында деформациялық процестерді тиімді бақылау және болжау үшін GPS-өлшеулер негізінде кешенді жүйені қамтитын мониторингтің көп параметрлі әдісі қажет, ол су деңгейінің өзгеру датчиктерінің деректерімен және массивтің кернеулері мен деформацияларының өзгеруімен толықтырылады. Бұлтты қоймаға жіберілген автоматтандырылған түсірілім және деректерді жазу нәтижелері "Big Data" технологиясының көмегімен таратылады және геоақпараттық жүйелермен талданады. Өз кезегінде нейрондық желілерді модельдердің деформацияларына бейімдеу мамандарға массивтің құрылымдық деформацияларын сипаттауға жақсы балама алуға мүмкіндік береді.

Түйін сөздер: «Заттардың интернеті» түсінігі, деформация мониторингі, «Үлкен деректер», нейрондық желілер, аналитикалық модельдер, деформация процестерін модельдеу.

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Современные технологии анализа данных применимые для выполнения геомеханического мониторинга. Обзор

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Аннотация

В работе рассмотрены возможности современных технологий и программного обеспечения, позволяющие создать неразрывность геомеханического мониторинга техногенных объектов от съемки в автоматическом режиме, роботизированными системами наблюдения, передачи информации по сети интернет в облачное хранилище, до выполнения расчетов устойчивости, определения параметров сдвижения и деформаций откосов уступов и бортов карьеров. Развитие современных технологий сбора и обработки информации позволяет использовать искусственные нейронные сети, которые адаптированы для моделирования геодезических деформаций. Техногенные объекты, представляющие собой очень сложные системы, обладают огромным количество внешних факторов, влияющих на состояние устойчивости горного массива, поэтому учесть и определить величину сдвижения и деформации становиться невероятно сложно. Из-за комплексности и разнообразия влияющих факторов, возникает необходимость использовать новую систему оценки состояния объектов, называемую «нейронными сетями». Обучение подобной системы, основывается на уже имеющихся результатах исследований, собранных при непосредственной эксплуатации промышленных предприятий. Нейронные сети могут стать альтернативой разнообразным методам описания деформационных процессов, особенно при непрерывном мониторинге техногенных объектов, где нет априорных знаний о лежащих в их основе деформационных процессах. Для эффективного мониторинга и прогнозирования деформационных процессов на горном предприятии необходим многопараметрический метод мониторинга, который включает в себя комплексную систему на основе GPS-измерений, дополняется данными датчиков изменения уровня воды и изменений напряжений и деформаций массива. Результаты автоматизированной съемки и записи данных, отправленные в облачное хранилище, распределяются с помощью технологии «Big Data», и анализируются геоинформационными системами. В свою очередь адаптация нейронных сетей к деформациям моделей позволяет специалистам получить хорошую альтернативу описанию структурных деформаций горного массива.

Ключевые слова. Концепция «Интернета вещей», мониторинг деформации, «Большие данные», нейронные сети, аналитические модели, моделирование деформационных процессов.

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Engineering and Technology

Mismatch problem of the model and topology of oil pumping facilities

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ABSTRACT

The mismatch of the model and the topology of real objects is important in modeling technological processes, which is the purpose of this paper. The problem is considered when modeling hot oil pumping in the "Kasymov–Bolshoy Chagan" oil pipeline. In this problem, the topology of objects consists of the linear part of the pipeline and technological equipment (pumps and heating furnaces) of the stations. The accuracy of the simulation results is determined by the calculations of pressure and temperature in the oil pipeline. The pressure in the pipeline is created by pumps at the stations and is determined by the dependence of the pressure and efficiency of the pump on the oil flow rate. These characteristics change depending on the service life of the pump. The identification of the actual dependences of the pressure and efficiency of the pump on the oil flow rate was carried out by the regression analysis of experimental data. The pressure in the linear part is determined by the hydraulic resistance of the pipeline. The actual dependence of the hydraulic resistance coefficient on the Reynolds number and wall roughness was obtained by regression analysis of experimental data. The temperature in the oil pipeline is created at the stations by heating furnaces. The identification of the actual characteristics of the heating furnace was also found by regression analysis of the experimental data. The temperature distribution in the linear part is determined by the heat transfer of oil with the surrounding environment. An undefined parameter for calculating heat transfer is the soil thermal conductivity, which depends on the type of rock and the degree of soil moisture. The soil thermal conductivity is determined in such a way that at a given oil flow rate, oil temperatures at the beginning of the section and soil at the section, the calculated oil temperature at the end of the section has the smallest discrepancy with the actual one. Thus, the determination of the actual dependencies of the objects makes it possible to increase the accuracy of the results of hot pumping modeling and eliminates the mismatches of the model and the topology of the objects.

Keywords: regression analysis, mismatches of the model and topology of oil pumping facilities, the actual data of pressure, temperature and flow rate sensors.

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Introduction

Machine learning is becoming a tool for researching technological processes in various fields of technology and production. In particular, machine learning finds application in industry, such as rolling, sheet metal forming, oil production, and transportation, well logging in uranium deposits, etc. The application of machine learning methods

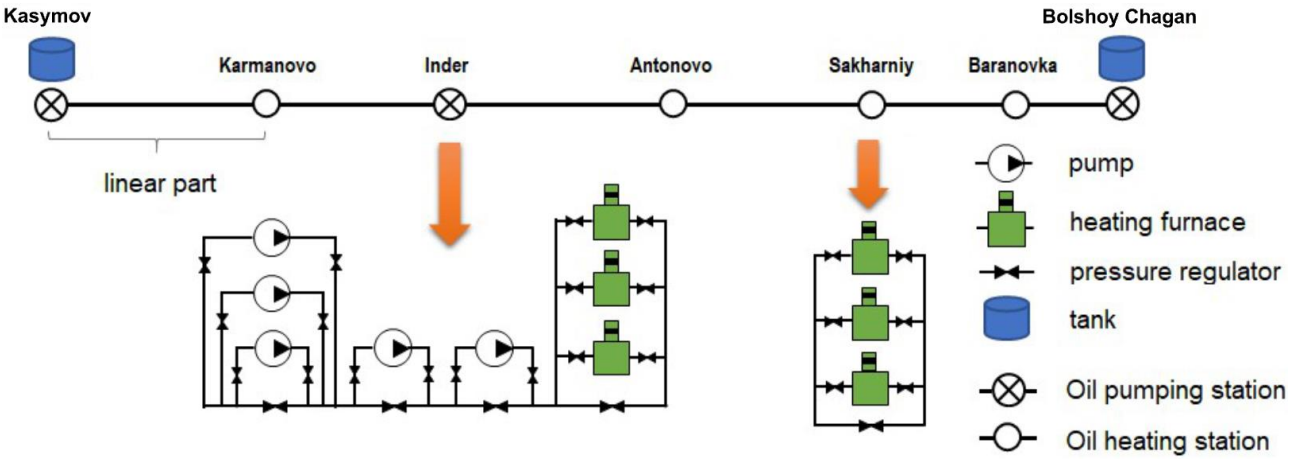


Figure 1 - Pipeline structural diagram: tanks, linear part, stations, heating furnaces, pumps, and pressure

allows analyzing and generalizing actual data to describe the patterns of processes. However, this will require a large amount of reliable empirical data. The presence of fuzzy data among them can have an impact on the results of machine learning. For the first time, Zadeh [1] introduced the concept of data oddness. Currently, the theories, models, and methods of decision-making based on fuzzy data have been developed [[2], [3], [4], [5], [6]]. The mismatch of the model and the topology of real objects is important in modeling and optimizing technological processes [[7], [8], [9], [10], [11], [12], [13], [14], [15], [16], [17], [18], [19], [20], [21]]. In this paper, this problem is considered when modeling the technological process of oil pumping. The accuracy of the pumping simulation results eliminates the mismatches of the model and the topology of real objects and expresses the novelty of this work. Below are the results of a study on achieving accuracy in calculating the pressure and temperature of hot oil transfer.

Nomenclature

OPS	Oil pumping station
OHS	Oil heating station

Problem statement

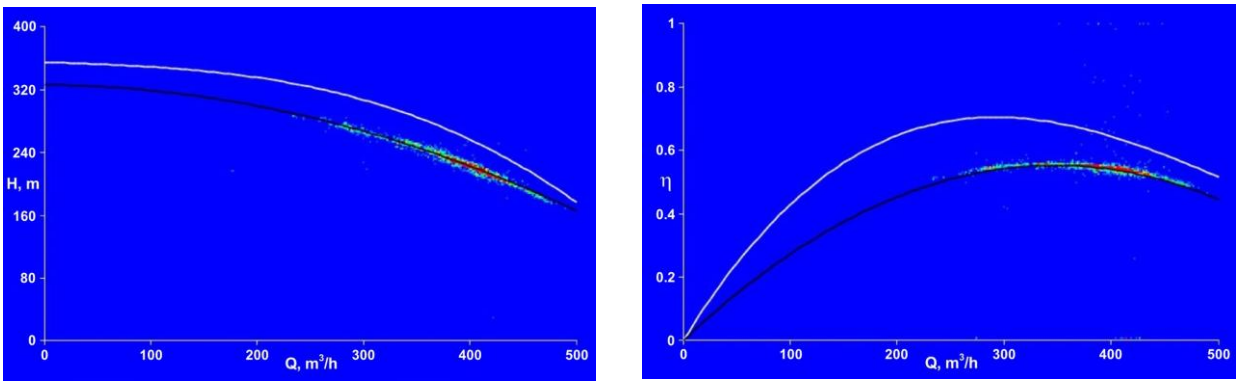
Oil is pumped in the technological sections of the "Kasymov–Bolshoy Chagan" main oil pipeline (see Figure 1). This main oil pipeline has only one starting point for the beginning of the flow (the "Kasymov" OPS) and only one endpoint for the end of the flow (the "Bolshoy Chagan" station). Oil flows through the pipeline in exactly one direction from the start station to the end station. There are intermediate stations in the pipeline. The topology of the object consists of the topology of the linear part of the pipeline and the topology of the stations. The accuracy of the simulation results of hot oil pumping is determined by the calculations of pressure and temperature in the oil pipeline [[22], [23]].

The pressure in the pipeline is created by pumps at the stations and is determined by the dependence of the pressure and efficiency of the pump on the oil flow rate. These characteristics change depending on the service life of the pump (see Fig. 2). The pressure in the linear part is determined by the hydraulic resistance of the pipeline. This value varies depending on the Reynolds number and the roughness of the pipeline wall (see Fig. 3).

The temperature in the oil pipeline is created at the stations by heating furnaces and the energy costs of the furnaces depend on the efficiency of the furnace. The temperature distribution in the linear part is determined by the heat transfer of oil to the environment and depends on the accuracy of determining the thermal conductivity of the soil (see Fig. 4).

Machine learning regression analyzes the accuracy of determining the characteristics of pumps and heating furnaces, as well as the hydraulic and thermal characteristics of the pipeline. For this, the actual readings of sensors (pressure, temperature, and flow) in the "Kasymov–Bolshoy Chagan" oil pipeline are used.

The accuracy of the simulation results depends on the accuracy of determining the characteristics of objects at the station (pumps and heating furnaces), as well as the hydraulics and heat transfer of the linear section of the oil pipeline.



a) Head curves b) Efficiency curves

Figure 2 - Identification of the characteristics of the pump at the "Kasymov" OPS by the regression analysis of experimental data.

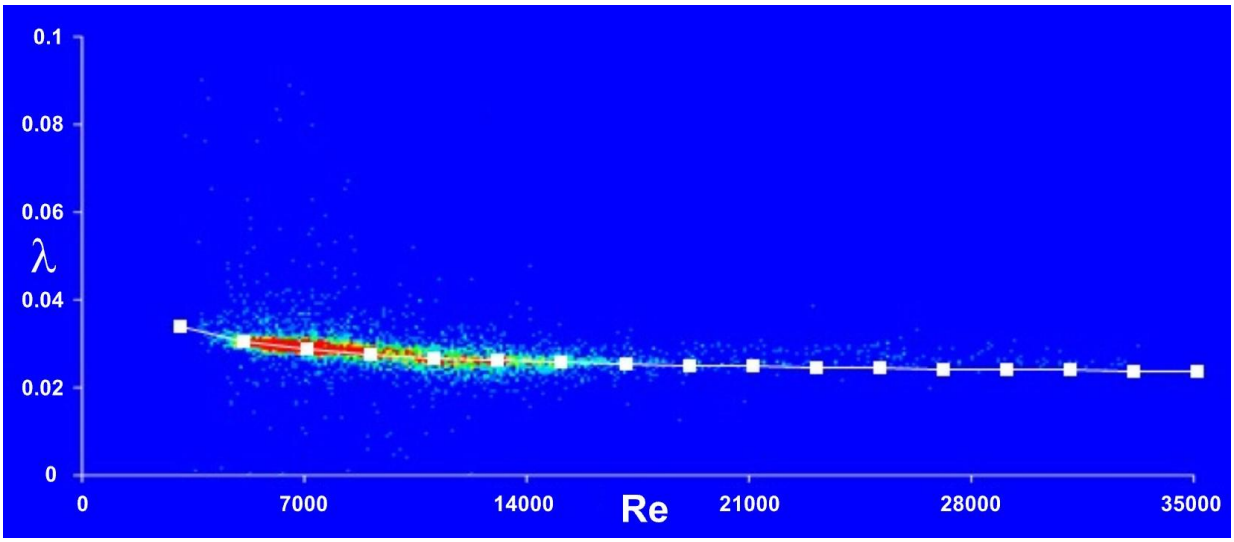


Figure 3 - Identification of the hydraulic resistance coefficient of the "Kasymov–Bolshoy Chagan" oil pipeline by the regression analysis of experimental data

Pressure distribution calculation along the pipeline

The pressure in the oil pipeline can vary at the stations and at the linear part. In the stations, the pressure in the pipe can increase due to the operation of pumps.

It is known that the pressure and efficiency of the pump functionally depend on the flow rate of pumped oil. This dependence, as a rule, is revealed as a result of factory tests and is indicated in the pump passport as the pressure and efficiency characteristics of the pump. During operation, the pressure and efficiency characteristics of the pump change. Practice shows that using the correct actual sensor data, for each pump, it is possible to obtain a clear actual dependence of head and efficiency on the flow rate of oil.

Figure 2 shows an example of the obtained actual characteristics of the pump (main pump No. 1 at the "Kasymov" OPS), using data on flow rate, oil density, pressure at the inlet and outlet of the pump, and electricity consumption. The colored dots are the actual pairs of head and flow rate values (see Figure 2, a), as well as the pairs of efficiency and flow values (Figure 2, b). The colors of the dots indicate the concentration of data in the area: from blue (no data) to red (maximum data concentration). The white lines show the passport head and efficiency curve, the black lines show the actual curve.

Thus, for each pump, it is possible to accurately calculate its generated pressure (by a head) and power consumption (based on efficiency) at a given

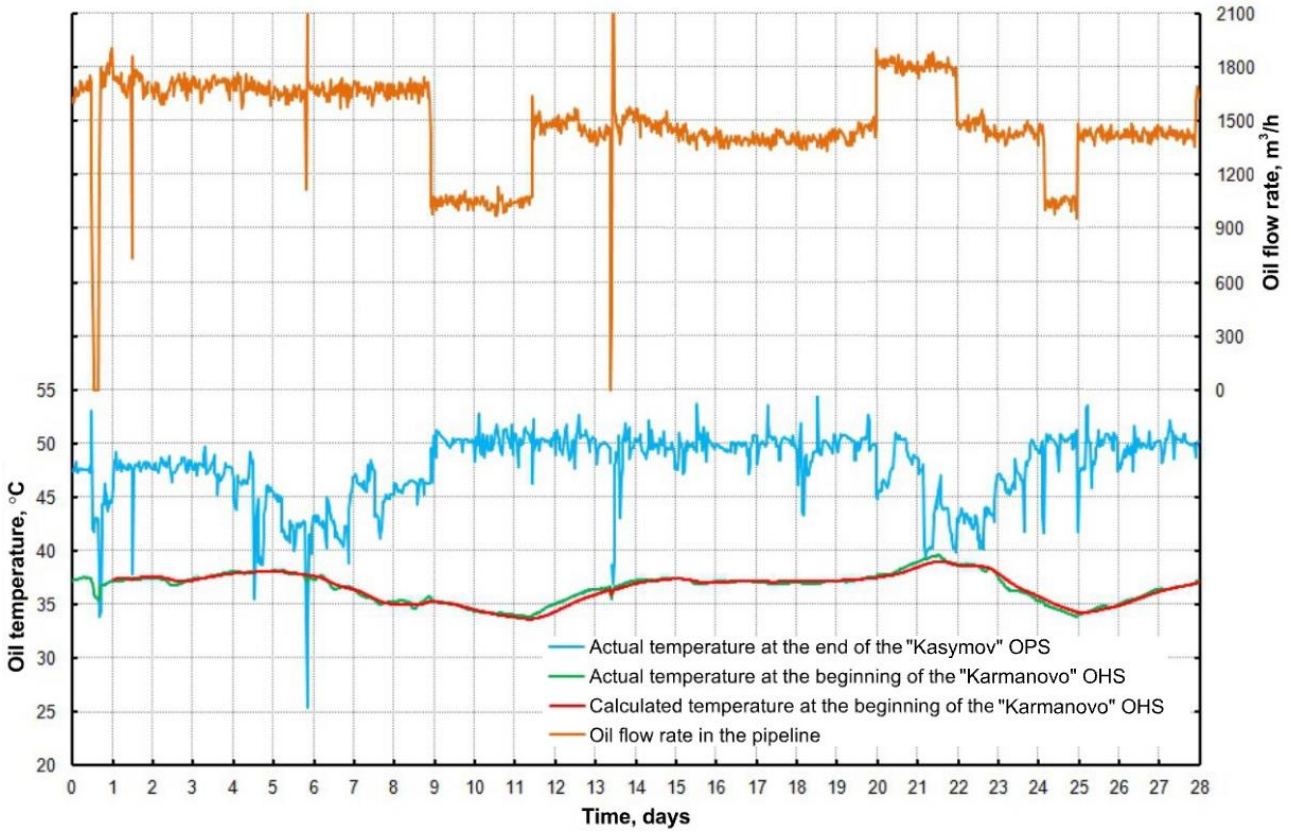


Figure 4 - Comparison of the actual and calculated temperature at the end of the section with the thermal conductivity value in the period of the month of February 2021 at the section of the "Kasymov" OPS–the"Karmanovo" OHS.

oil consumption by using the machine learning regression analysis.

At the linear part, the pressure changes due to the loss of the resistance of the oil flow through the pipeline. In the "Kasymov–Bolshoy Chagan" oil pipeline, the oil flow regime is turbulent. Hydraulic losses in the pipeline are calculated according to the well-known Darcy-Weisbach formula [24]. The hydraulic resistance coefficient of the Darcy-Weisbach formula depends on the Reynolds number and the wall roughness, which is determined by the empirical formulas of the following works [[25], [26], [27], [28], [29]].

The roughness coefficient changes during pipeline operation. As well as the pipe roughness, it can be non-uniform along the pipeline length, for example, as a result of repairs, the pipe sections can be replaced in the pipeline. It follows that without actual data on the condition of the pipes, the calculated values of total hydraulic losses in the pipe may differ significantly from the actual ones. Using the actual pressure and temperature data by the machine learning regression analysis, it is possible to construct the pipe hydraulic resistance coefficient dependence and determine the wall roughness effect. Figure 3 shows the found actual dependence of the hydraulic resistance coefficient of the Darcy-Weisbach formula on the Reynolds number in the turbulent flow regime in the "Kasymov–Bolshoy Chagan" oil pipeline.

Temperature distribution calculation along the pipeline

The temperature in the pipeline may vary in the stations and the pipeline linear part. In the stations, the temperature can rise due to heating in the furnaces. Using the actual data from the data of heating furnace sensors, it is possible to build actual dependences for the efficiency of heating furnaces, as in the case of the pump (see Figure 2, b).

In the linear part, the oil temperature decreases due to heat exchange with the surrounding soil of the pipeline, and there are various models for calculating the heat exchange of the oil flow with the surrounding soil [[22], [23]].

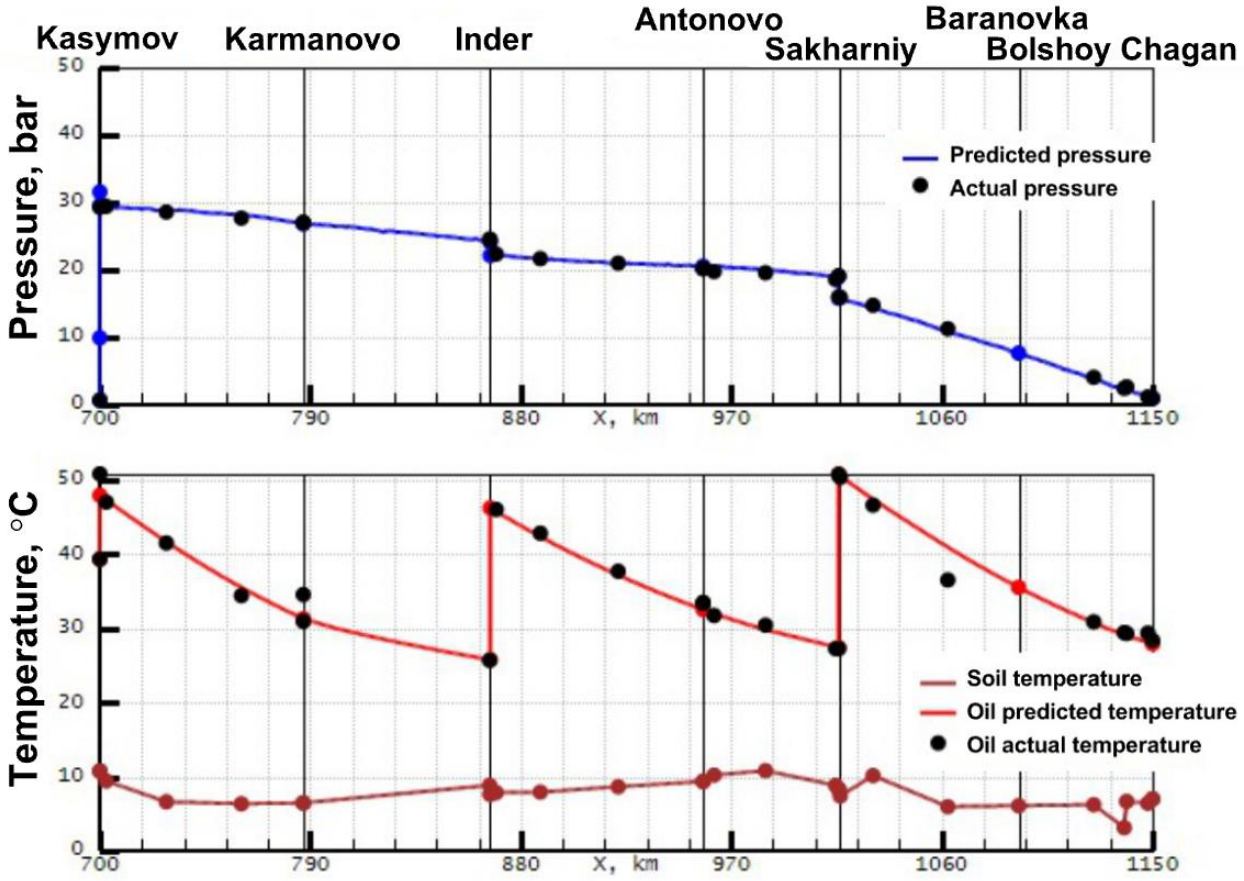


Figure 5 - Comparison of calculated and experimental data on the distribution of pressure (the blue line on the top diagram) and temperature (the red line on the bottom diagram) along the length of the "Kasymov–Bolshoi Chagan" oil pipeline at an oil flow rate of 1143 m^3/hour. In the diagrams: the lines are the calculated data, and the dots are the experimental data.

Some calculation parameters are known: flow rate, insulation, soil temperature, and the properties of oil and metal pipes.

The undefined parameter is the soil thermal conductivity. It is known that the soil thermal conductivity depends on the type of soil rock (sand, sandy loam, loam, clay, etc.) and on the degree of soil moisture.

It is obvious that the soil thermal conductivity value is not constant along the pipeline length and over time (it depends on the snowmelt season and the frequency of rains for a given area). Therefore, without investigating the actual data, the calculated oil temperature distributions along the pipeline may differ significantly from the actual one. When finding the calculated thermal conductivity, its value is averaged over the length and time. Averaging over the length depends on the distribution density of temperature sensors along the pipeline. In the "Kasymov–Bolshoy Chagan" main oil pipeline, temperature sensors are located quite often (at a distance of 5-15 km). Averaging over time is sufficient to carry out by month or by 10-15 days (for some months). The determination of the soil thermal conductivity is carried out in such a way that for a given oil flow rate in the pipe, oil temperatures at the beginning of the section and soil temperatures at the section, the calculated oil temperature at the end of the section has the least discrepancy with the actual one. Figure 4 shows the comparison of the actual and calculated temperature (see the red and green lines in the figure) at the end of the section with the found value of the soil thermal conductivity in the section of the "Kasymov" OPS–the"Karmanovo" OHS.

Thus, by the regression analysis of the actual data, the dependencies of the topology of oil pumping facilities are determined and used for the accuracy of thermal-hydraulic calculations. As can be seen from Figure 5, the calculated pressure and temperature distributions agree with the actual data along the length of the "Kasymov–Bolshoy Chagan" oil pipeline.

Conclusions

The mismatches of the model and topology of objects are determined by the accuracy of pressure and temperature calculations along the "Kasymov–Bolshoy Chagan" oil pipeline length. The results of pressure and temperature calculations depend on the actual dependencies of the pumps and heating furnaces, as well as the hydraulic and thermal characteristics of the pipeline.

The machine learning regression analysis makes it possible to analyze the accuracy of determining the actual dependencies of pumps and heating furnaces, as well as the hydraulic and thermal characteristics of the pipeline.

By the regression analysis of the actual data, the following were obtained:

1) the dependences of the pressure characteristics and efficiency of pumps, as well as the efficiency of heating furnaces, considering their operating resources;

2) the pipeline hydraulic resistance coefficient dependence on the Reynolds number;

3) the dependences of the soil thermal conductivity to determine the oil flow heat transfer coefficient in the pipe with the environment.

Conflict of interest. On behalf of all the authors, the correspondent author declares that there is no conflict of interest.

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Мұнай айдау объектілерінің моделі мен топологиясының сәйкессіздігінің проблемасы

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ТҮЙІНДЕМЕ

Нақты нысандардың моделі мен топологиясының арасындағы сәйкессіздік технологиялық процестерді модельдеген кезде маңызды және осы мақаланың мақсаты болып табылады. Қасымов-Большой Шаған мұнай құбырында ыстық мұнай айдауды модельдеу мәселесі қарастырылды. Бұл мәселеде нысандардың топологиясы құбырдың сызықтық бөлігінен және станциялардың технологиялық жабдықтарынан (сорғылар мен жылыту пештері) тұрады. Модельдеу нәтижелерінің дәлдігі мұнай құбырындағы қысым мен температураның есептеулерімен анықталады. Құбырдағы қысым станциялардағы сорғылармен жасалады және сораптың қысымы мен ПӘК-нің мұнай ағынына тәуелділігімен анықталады. Бұл сипаттамалар сорғының қызмет ету мерзіміне қарай өзгереді. Эксперименттік мәліметтерді регрессиялық талдау арқылы қысым мен сорғы тиімділігінің мұнай шығынына нақты тәуелділігі анықталды. Сызықтық бөліктегі қысым құбырдың гидравликалық кедергісі арқылы анықталады. Гидравликалық кедергі коэффициентінің Рейнольдс санына және қабырға кедір-бұдырлығына нақты тәуелділігі тәжірибелік мәліметтерді регрессиялық талдау арқылы алынды. Мұнай құбырындағы температура станцияларда қыздыру пештері арқылы жасалады. Жылыту пешінің нақты сипаттамаларын анықтау тәжірибелік мәліметтерді регрессиялық талдау арқылы да жүргізілді. Сызықтық бөліктегі температураның таралуы мұнайдың қоршаған ортамен жылу алмасуымен анықталады. Жылу беруді есептеудің анықталмаған параметрі топырақтың жылу өткізгіштігі болып табылады, ол тау жыныстарының түріне және топырақтың ылғалдылық дәрежесіне байланысты. Топырақтың жылу өткізгіштігі мынадай түрде анықталады. Мұнайдың шығыны, учаскенің басында оның температурасы және участкедегі топырақтың температурасы беріледі. Осындай жағдайларда участкенің соңында мұнайдың есептелген температурасы фактімен сәйкес келуі керек. Осылайша, объектілердің нақты тәуелділіктерін анықтау ыстық айдау модельдеу нәтижелерінің дәлдігін арттыруға мүмкіндік береді және модель мен объектілер топологиясы арасындағы сәйкессіздіктерді жояды.

Түйін сөздер: регрессиялық талдау, айдау объектілерінің моделі мен топологиясының сәйкессіздігі, қысым, температура және шығын датчиктерінің нақты деректері

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Проблема несоответствия модели и топологии объектов перекачки нефти

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АННОТАЦИЯ

Несоответствие модели и топологии реальных объектов важно при моделировании технологических процессов, что и является целью данной статьи. Задача рассмотрена при моделировании перекачки горячей нефти в нефтепроводе «Касымов–Большой Чаган». В этой задаче топология объектов состоит из линейной части трубопровода и технологического оборудования (насосов и нагревательных печей) станций. Точность результатов моделирования определяется расчетами давления и температуры в нефтепроводе. Давление в трубопроводе создается насосами на станциях и определяется зависимостью напора и КПД насоса от расхода нефти. Эти характеристики изменяются в зависимости от срока службы насоса. Выявление реальных зависимостей напора и КПД насоса от расхода нефти осуществлялось путем регрессионного анализа экспериментальных данных. Давление в линейной части определяется гидравлическим сопротивлением трубопровода. Фактическая зависимость коэффициента гидравлического сопротивления от числа Рейнольдса и шероховатости стенки была получена путем регрессионного анализа экспериментальных данных. Температура в нефтепроводе создается на станциях нагревательными печами. Выявление реальных характеристик нагревательной печи также было установлено путем регрессионного анализа экспериментальных данных. Распределение температуры в линейной части определяется теплообменом нефти с окружающей средой. Неопределяемым параметром для расчета теплоотдачи является теплопроводность грунта, которая зависит от типа породы и степени влажности грунта. Теплопроводность грунта определяют таким образом, чтобы при заданных расходе нефти, температурах нефти в начале участка и грунта на участке расчетная температура нефти в конце участка имела наименьшее расхождение с фактической. Таким образом, определение реальных зависимостей объектов позволяет повысить точность результатов моделирования горячей откачки и устраняет несоответствия модели и топологии объектов.

Ключевые слова: регрессионный анализ, несоответствия модели и топологии объектов перекачки, фактические данные датчиков давления, температуры и расхода.

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Engineering and Technology

ADME Webtool for Analysis of Selected Apple Phytochemical Constituents: A Comprehensive Integrated Online Platform

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ABSTRACT

ADME-Tox qualities should be considered while designing/engineering a novel medicine because they are the primary cause of failures for candidate molecules in drug design development. Early examination of these features during medication creation might save time and money. ADME has played an important part in the drug engineering/design process throughout the last five decades. The ADME characteristics of apple constituents were determined using SwissADME web servers. The ADME profiles of the compounds were assessed, and most of them were deemed to be appropriate for further research. *In-silico* ADMET analysis has been shown to be an effective approach in drug engineering/design development. As a result, all compounds were tested for ADMET prediction, and the phytochemical constituents were shown to be acceptable drug-like molecules. More *in vitro* and *in vivo* research with our possible phytochemical compounds will be conducted in the near future to find a solution to cure different diseases.

Keywords: SwissADME, ChemDraw, *in silico* prediction, ADME-Tox, Design, Medicine.

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Introduction

ADME stands for absorption, distribution, metabolism, and excretion. It describes the pharmacokinetics of a pharmacological molecule. There have been multiple reports of drug development efforts being abandoned due to poor ADME profiles [1]. ADME profiling was shown to be more effective prior to synthesis and *in-vivo* research. The determination of ADME characteristics of substances necessitates a slew of time-consuming and costly experimental techniques. As a result, *in sillco* ADME models have been created. SWISS ADME predictor [2] was used for the ADME investigation. The latter is considered a free tool (web) that is used for estimating the drug similarity, pharmacokinetics, as well as medicinal chemistry of small compounds. As previously stated, emphasis was placed on designing molecules that adhered to the criterion of drug-likeness [[3], [4]].

Log P is a measure used to estimate a molecule's lipophilicity and can be defined as the logarithm of the ratio of drug concentration among two unionized solvents [5]. The Lipinski rule establishes a maximum limit of 5 for druggable substances. It is well known that the lower log P values indicate the higher lipophilicity of the drug. A compound's water solubility has a large impact on its absorption as well as distribution properties. On the other hand, low water solubility relates to weak absorption, therefore the overall goal is to avoid weakly soluble substances. Log S is defined as a unit to represent solubility which is the 10-based logarithm of solubility and measured in unit mol/L. The Log S distribution in traded medications indicates a value ranging from -1 to -4, which will be ideal for improved drug absorption and distribution in the body [[3], [4]].

Food offers not only the necessary nutrients needed for a living but also additional chemicals for promoting health and preventing disease since it is a

complex mixture of a broad range of components, many of which are biologically active. Nutrients are a group of previously identified substances that are crucial for the body's development, maintenance, and repair. Previous epidemiologic studies have repeatedly demonstrated the critical impact that nutrition plays in preventing chronic illnesses [6]. Remarkably, according to research, a diet high in fruits and vegetables may reduce the risk of chronic illnesses such as cancer and cardiovascular disease.

The bioactive components in these natural products—dietary fiber and antioxidants, namely phenolic compounds, flavonoids, phenolic acids, etc.—are thought to be what give them their beneficial effects [7]. Therefore, there is presently a lot of interest in the numerous bioactive substances that may be found in food, especially food that comes from plants. These plant-based bioactive substances are usually referred to as phytochemicals [8]. Although it is considered that more than 5000 phytochemicals have been found, a significant portion is still unknown and needs to be discovered before their health advantages can be properly comprehended.

The advantages of phytochemicals in fruit and vegetables may, however, be much higher than currently believed since oxidative stress brought on by free radicals is thought to be a contributing factor in the development of a wide range of chronic illnesses [9]. Due to their content of phytochemicals, several frequently eaten foods and drinks have been identified as particularly advantageous in the diet. Although there has been continuing study into the health benefits of these foods, there are now available evaluations of this work for all save apples [10].

Because of a variety of variables, including market accessibility and cultivar variation, apples make up a significant amount of the fruit supply throughout the year in most nations [11]. The adage "one apple a day, keeps the doctor away" refers to the widespread consumption of apples and their overall health benefits. What is less generally recognized, though, is that apples are a good source of strong plant components such as phytochemical constituents (Figure 1), and epidemiological findings have connected apple consumption to a minor risk of some malignancies, cardiovascular disease, diabetes, and asthma [10].

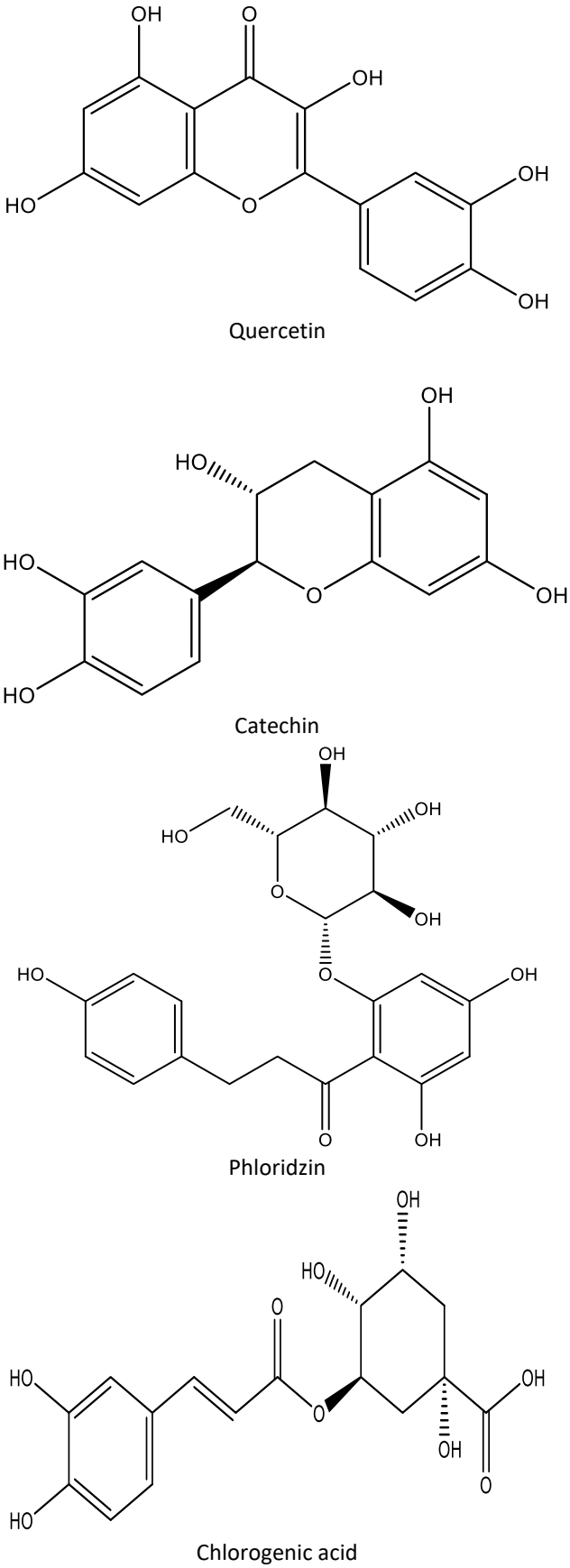

Figure 1 - Chemical structures of some phytochemical constituents in apple

ADME properties calculation

The structure was created in Chemsketch, and the SMILES of each chemical were translated into molfiles using an online SMILES translator and structure file generator available in the online program SwissADME. Furthermore, pharmacokinetics such as gastrointestinal absorption, skin permeability, blood-brain barrier, as well as a drug-likeness estimate for instance bioavailability score.

BBB penetration is a parameter used to determine if a substance passes via the blood-brain barrier. Numerous drugs must not cross over the blood-brain barrier if the aim is unrelated to the nervous system [12].

The skin permeability of the substance is a significant feature regarding the adverse reaction of the drug in the case of pharmaceuticals taken orally to identify of accidental contact with skin and the skin permeability of drugs to be administered transdermally where skin penetration is a significant aspect. The result value of a compound's skin permeability is provided such as log Kp. Kp [cm/hour] is described as Kp= Km*D/h, where Km is called the distribution coefficient between the vehicle and the stratum corneum, D is defined as the average diffusion coefficient [cm^2/h], and h is the skin thickness [cm] [4].

The knowledge of chemicals that are considered either a substrate or a non-substrate of the permeability glycoprotein. It refers to the most significant ABC transporter, which is vital for evaluating active efflux through biological membranes, for example from the wall of the gastrointestinal to the lumen or even from the brain. P-gp shields the central nervous system against xenobiotics. Additionally, P-gp is overexpressed in specific cancer cells, which leads to tumors in multidrug-resistant [13].

Drug-likeness is considered a qualitative evaluation of a molecule's chances of becoming an oral drug by means of bioavailability. Drug-likeness resulting from physicochemical or structural examinations of research compounds progressed sufficiently to be considered oral drug candidates. This concept is commonly used in the filtering of chemical libraries to refuse compounds with characteristics that are most probable incompatible with an appropriate pharmacokinetics profile [[4], [14]]. The current SwissADME section offers full access to five alternative rule-based filters together with changing ranges of attributes within which the molecule is regarded as drug-like. These filters are frequently produced by pharmaceutical corporations to enhance the quality of their chemical collections. The Lipinski (Pfizer) filter was the first to include the Ghose (Amgen), Veber (GSK), Egan (Pharmacia), and Muegge (Bayer) technologies [15].

A soluble molecule significantly simplifies several drug development procedures, particularly the simplicity of processing and formulation. Solubility is an important property that influences absorption in drug discovery plans aimed at oral administration, and drugs aimed for parenteral administration must be very water soluble to deliver an adequate amount of active ingredient in the minor volume of pharmaceutical dosage. SwissADME includes two topological approaches for predicting solubility in water.

The first model is called ESOL model implementation, whereas the second one is adapted from Ali et al. modification. Both differ from the fundamental universal solubility equation by omitting the difficult-to-estimate melting point parameter. They show a significant linear connection between anticipated and observed values (R^2=0.69 and 0.81, respectively). SILICOS-IT created the SwissADME third predictor for solubility. This fragmental method's linear correlation coefficient adjusted for molecular weight is R^2 = 0.75. To calculate all expected values (log S), the decimal logarithm of the molar solubility in water is used. Additionally, SwissADME offers solubility in mol/l and mg/ml units, as well as solubility classes [[4], [16], [17], [18], [19], [20], [21] [22], [23]].

Results and Discussion

Table 1 shows the IUPAC name and the SMILES code of some phytochemical constituents of apples. SwissADME online version was used to estimate the pharmacokinetic characteristics and drug-likeness of apple phytochemical ingredients, and the results are displayed in table 2 and table 3, respectively. All test substances demonstrated pharmacokinetic characteristics. The bioavailability score predicted medication similarity in moderately soluble and soluble gastrointestinal absorption.

Table 1 - The IUPAC name and the SMILES code of some phytochemical constituents of apple

No.	Phytochemical Constituents	IUPAC Name	Canonical SMILES
1	Quercetin	2-(3,4-dihydroxyphenyl)-3,5,7trihydroxychromen-4-one	CC1CCC2C(C(=O)OC3C24C1CCC(O3)(OO4)C)C
2	Catechin	(2S,3R)-2-(3,4-dihydroxyphenyl)-3,4-dihydro-2H-chromene-3,5,7-triol	C1C(C(OC2=CC(=CC(=C21)O)O)C3=CC(=C(C=C3)O)O)O
3	Phloridzin	1-[2,4-dihydroxy-6-[(2S,3R,4S,5S,6R)-3,4,5-trihydroxy-6-(hydroxymethyl)oxan-2-yl]oxyphenyl]-3-(4-hydroxyphenyl)propan-1-one	C1=CC(=CC=C1CCC(=O)C2=C(C=C(C=C2OC3C(C(C(C(O3)CO)O)O)O)O)O)O
4	Chlorogenic acid	(1S,3R,4R,5R)-3-[(E)-3-(3,4-dihydroxyphenyl)prop-2-enoyl]oxy-1,4,5-trihydroxycyclohexane-1-carboxylic acid	C1C(C(C(CC1(C(=O)O)O)O)OC(=O)C=CC2=CC(=C(C=C2)O)O)O)O

Table 2 - Pharmacokinetics and drug-likeness prediction of some Phytochemical constituents of apple

No.	Phytochemical Constituents	Pharmacokinetics			Drug-likeness
		GI Absorption	BBB Permeability	Log Kp (skin Permeation) cm/s	Bioavailability Score
1	Quercetin	High	Yes	-5.96	0.55
2	Catechin	High	No	-7.82	0.55
3	Phloridzin	Low	No	-8.58	0.55
4	Chlorogenic acid	Low	No	-8.76	0.11

Table 3 - Water solubility prediction for some Phytochemical constituents of apple

No.	Phytochemical constituents	LogP	Water Solubility		
		(Consensus LogP)	LogS (ESOL)	LogS (Ali)	LogS (SILICOS-IT)
1	Quercetin	2.50	-3.42	-3.69	-2.03
2	Catechin	0.85	-2.22	-2.24	-2.14
3	Phloridzin	0.08	-2.71	-3.83	-1.66
4	Chlorogenic acid	-0.57	-1.62	-2.58	0.40

Conclusions

According to Lipinski's rule-of-five (RO5), all phytochemical elements of apple displayed strong drug-likeness and were projected to be BBB non-permeant (blood-brain barrier), implying no expected neurological adverse effects. It has been demonstrated that it has a high bioavailability, implying that the molecules could be absorbed and transported throughout the body if used as a

medication. As a result, all compounds were examined for ADMET prediction, and the Phytochemical ingredients were found to be acceptable drug-like molecules.

Conflict of interest

The correspondent author declares that there is no conflict of interest on behalf of all authors.

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Алманың таңдалған фитохимиялық құрамдас бөліктерін талдауға арналған ADME веб-құралы: кешенді біріктірілген онлайн платформа

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ТҮЙІНДЕМЕ

ADME-Tox қасиеттерін жаңа препараттарды жасағанда, өндіргенде ескеру қажет, себебі бұлар дәрілік заттарды әзірлеу кезінде кандидат-молекулалар үшін болатын сәтсіздіктерінің негізгі себебі болып табылады. Дәрі-дәрмектерді дайындағанда бұл сипаттамаларды мерзімінен бұрын зерттеу - уақыт пен ақшаны үнемдеуге мүмкіндік береді. ADME соңғы бес онжылдықта дәрілерді жасағанда маңызды рөл атқарады. Алмалардың ADME сипаттамалары SwissADME серверлерінің көмегімен анықталды. ADME қосылыстарының профильдері бағаланды және олардың көпшілігі қосымша зерттеулер үшін жарамды деп саналды. In-silico ADMET талдауы дәрілік инженерия/дизайн әзірлеуде тиімді тәсіл екені көрсетілді. Нәтижесінде барлық қосылыстар ADMET болжамы үшін сыналған және фитохимиялық құрамдастардың қолайлы дәрілік молекулалар екендігі көрсетілген. Жақын арада әртүрлі ауруларды емдеудің шешімін табу үшін біздің ықтимал фитохимиялық қосылыстарымызбен in vitro және in vivo зерттеулері жүргізіледі.

Түйін сөздер: SwissADME, ChemDraw, *in silico* болжау, ADME-Tox, дизайн, медицина.

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Веб-инструмент ADME для анализа выбранных фитохимических компонентов яблока: комплексная интегрированная онлайн-платформа

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АННОТАЦИЯ

Свойства ADME-Tox следует учитывать при разработке/производстве новых лекарств, поскольку они являются основной причиной неудач для молекул-кандидатов при разработке лекарств. Преждевременное изучение данных характеристик дает воможность сэкономить время и деньги при создании лекарственных препаратов. ADME играет важную роль в процессе разработки лекарств на протяжении последних пяти десятилетий. Характеристики ADME яблок были определены с использованием серверов SwissADME. Были оценены профили ADME соединений, и большинство из них было сочтено

	подходящими для дальнейших исследований. Было доказано, что анализ *In-silico* ADMET является эффективным подходом при производстве/разработке лекарств. В результате всех соединений были протестированы на предсказание ADMET, и было показано, что фитохимические соединения являются приемлемыми лекарственными молекулами. В ближайшем будущем будут проведены дополнительные исследования in vitro и in vivo с учетом возможных фитохимических соединений, чтобы найти решение для лечения различных заболеваний.
	Ключевые слова: SwissADME, ChemDraw, *in silico* прогнозирование, ADME-Tox, дизайн, медицина.
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Fine-grained fiber concrete using polypropylene and basalt fibers

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ABSTRACT

The purpose of the study is to evaluate the effect of fibers on the bending strength of fine-grained concrete samples. The results of experimental studies of polypropylene and basalt fibers for dispersion reinforcement of concrete are considered. The strength characteristics of fiber concrete of different compositions have been determined. The regularities of the influence of fiber type and concentration on the strength characteristics of fiber concrete are revealed. The results of determining the bending strength of fine-grained fiber concrete without adding fiber (control composition) and with the addition of polypropylene fiber 0.1, 0.5, 1.5, 2.5% of the weight of cement and basalt 0.05, 0.1, 0.2, 0.5% of the weight of cement are presented. It is shown that the optimal limits of the introduction of polypropylene fiber in the mixture of fine-grained concrete can be considered 0.5 % by weight of cement. The introduction of basalt fiber in the mixture of fine-grained concrete in an amount of 0.1 % of the weight of cement can increase the bending tensile strength.

Keywords: fiber concrete, polypropylene fiber, basalt fiber, strength.

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Introduction

One of the promising ways to increase the performance characteristics of concrete and mortars is considered the introduction of microfiber in their formulation [[1], [2]]. The use of this material makes it possible to increase the strength of the cement stone, which provides higher compressive and bending tensile strength, as well as increases other characteristics of cement-based construction materials.

It is recommended to use polypropylene fiber in the technology of fiber concrete, plaster, masonry and installation mortars, hydraulic and cellular concrete to reduce the delamination of mixtures, increasing water resistance, frost resistance, corrosion resistance, impact strength, abrasion resistance [[3], [4], [5]].

To produce fiber concrete with the best characteristics it is necessary to achieve technological compatibility of concrete-matrix and fiber, maximum anchorage of fiber in concrete. It is categorically not allowed to get lumps of fiber, it must be very carefully mixed [6].

The undoubted advantages of fiber concrete include its high performance characteristics. Concrete with fiber in its composition is much superior to conventional concrete in quality, strength and durability. Products made of it become resistant to abrasion and chemical attack, are not deformed during operation and have high tensile and breaking strength. The use of fiber as a

reinforcing material can significantly reduce the labor intensity of concrete products. Such structures do not require additional reinforcement by means of metal frames and meshes. This factor significantly speeds up the construction process and eliminates labor-intensive costs [[7], [8]].

Among the most promising types of polymer fibers are polypropylene fibers. Polypropylene fibers belong to the class of fibers of organic origin. This type of fibers is characterized by low cost, low elastic modulus, high elongation coefficient and corrosion resistance [[9], [10], [11], [12]]. High corrosion resistance allows the use of fibers under the influence of acids and alkalis, which is especially important for concrete, hardening of which is activated by alkaline solutions [13]. Polypropylene fibers are produced by continuous method from pure polypropylene pellets by extrusion as well as by drawing when heated. When the fibers reach a certain temperature, an oiling compound is applied to their surface. It is this composition that promotes surface adhesion and dispersion of polypropylene fibers in concrete mortar. Fibers have the form of thin white polypropylene fibers of different sizes, which is an inert substance, resistant to alkalis and various chemicals [[14], [15], [16]].

The use of basalt fiber leads to the creation of the simplest aggregate formations, while viscoplastic properties are changed, as evidenced by the increase in the plastic strength of the system. The fibers improve the microstructure, reduce internal stresses and shrinkage of the cement stone [[17], [18]]. The spindle-shaped structure of basalt fiber chemisorptionally interacts with the cement system, creating conditions for the growth of newly formed low-base calcium hydrosilicates in the contact zone. The structure is strengthened due to adhesion, or fusion of fiber fibers with new formations of the curing material.

Basalt fiber is produced from basalt rock melts. The advantages of basalt fiber for disperse reinforcement are that along with high strength it does not pull under load, has chemical, corrosion and thermal resistance to the environment, temperature fluctuations and intense alternating loads, and also has a low cost. Basalt fiber (chopped basalt thread) is pieces of basalt fiber and is an effective reinforcing additive for various types of concrete [[19], [20]]. Basalt rock fibers have high natural initial strength, resistance to corrosive medium, durability, electrical insulation properties, and are produced from natural, environmentally friendly raw materials [21]. Therefore, basalt fibers have prospects of application in industry, construction, power engineering.

Purpose of the study: evaluation of the effect of fiber on the strength of standard concrete specimens.

To achieve the goal the following tasks were solved:

1. Preparation of samples;
2. Strength at bending;
3. Strength in compression;
4. Analysis of the results.

Experimental technique

For the experimental work as a binder used Portland cement PC 400 D0 without additive, true density - 3100kg/m^3, bulk density - 1100-1600 kg/m^3.

As a fine fraction of the aggregate used natural quartz sand with a fineness modulus of 2.23, which meets the requirements of GOST 8736-2014 «Sand for construction works».

Polypropylene and basalt fibers were selected for testing the mechanical properties depending on the degree of reinforcement of fine-grained fiber concrete.

The quantitative ratio of fibers in the composition of the samples was selected on the basis of their density ratios:

$$\frac{P\ pol.}{P\ baz.} = x \qquad (1)$$

$$a_1 \ldots a_n,\ b_1 \ldots b_n$$

$$a_1 / b_1 = x \ldots\ldots\ldots a_n / b_n = x$$

Thus we obtain the necessary amount of each type of fiber by weight, corresponding to their equal volume in the sample. The variability of the concrete mixture with the addition of polypropylene fibers is characterized by the following variables: $a_1 \ldots a_n$ - the limits of adding polypropylene fibers to the concrete mixture. Accordingly, for the concrete mixture with the addition of basalt fiber, the variability of the composition is characterized by the variables $b_1 \ldots b_n$.

Polypropylene fiber was introduced into dry mixture for fine-grained concrete in the amount of 0.1, 0.5, 1.5, 2.5% of the mass of cement, followed by thorough mixing and mixing with water. From equation (1) you can find ratio for basalt fiber: 0.05, 0.1, 0.2, 0.5% of cement mass. The chosen range of fiber dosage corresponds to the requirements of

regulatory documents and manufacturer's recommendations. The physical and mechanical characteristics of the fibers are given in Table 1.

Table 1 - Characteristics of fibers

Properties	***Polypropylene***	***Basalt***
Density (kg/m^3)	620	3100
Length (mm)	10	12
Dia. (mkm)	22	18
Tensile strength (MPa)	170 – 260	3000-4840
Elongation to break, %	150 – 250	3.1-6.0

Tap water as mixing water for concrete mix that meets the requirements of GOST 23732-2011 «Water for concretes and mortars».

Tensile bending strength of concrete were determined on specimens of bars 40x40x160 mm in size at the age of 3, 7 and 28 days of normal curing. The procedure of testing the bending strength of concrete bars was carried out in accordance with GOST 310.4-81 «Cements. Methods for Determining the Flexural and Compressive Strength».

Consumption of raw materials of cement-sand mortar samples is presented in Table 2.

Table 2 - Cement mortar composition

Type of sample	**Cement, g**	**Quartz Sand, g**	**Fiber, g**	**Water, g**
Type 1 Reference sample	450	1350	-	180
Polypropylene				
Type 2	450	1350	0.45	180.18
Type 3	450	1350	2.25	180.9
Type 4	450	1350	6.75	182.7
Type 5	450	1350	11.25	184.5
Bazalt				
Type 6	450	1350	0.225	180.09
Type 7	450	1350	0.45	180.18
Type 8	450	1350	0.9	180.36
Type 9	450	1350	2.25	180.9

The effect of micro-reinforcing fibers on the properties of fine-grained concrete with a cement to aggregate ratio of 1:3 and a water-cement ratio of 0.4 was investigated.

Mixtures were prepared manually in a mixing bowl in accordance with GOST 310.3-76. Preliminary prepared mixture of cement and sand was mixed with water for 2 minutes, after which fibers were evenly introduced into it for 4 minutes under constant stirring. After preparation of the mixture its consistency was determined by its melt on the shaking table in accordance with the method of GOST 310.4-81. There were investigated compositions with the consumption of polypropylene fiber 0.1, 0.5, 1.5, 2.5% and basalt fiber 0.05, 0.1, 0.2, 0.5% of cement mass, as well as control composition without additions, which were curing for 28 days in air-humid conditions.

Tap water as mixing water for concrete mix that meets the requirements of GOST 23732-2011 «Water for concretes and mortars».

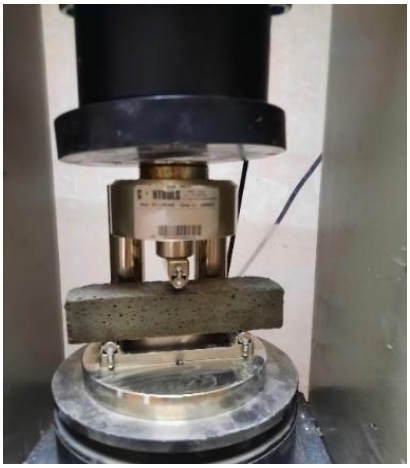

Figure 1 – Before the start of the test

Figure 2 – After completing the test

Tests were conducted at 3, 7 and 28 days of normal curing on an Automatic Pilot 500kN press (Figure 1-2).

After testing by destructive method, samples were inspected to visually assess the uniformity of

the distribution of fiber concrete along the shear fracture of the beam (Figure 3-4).

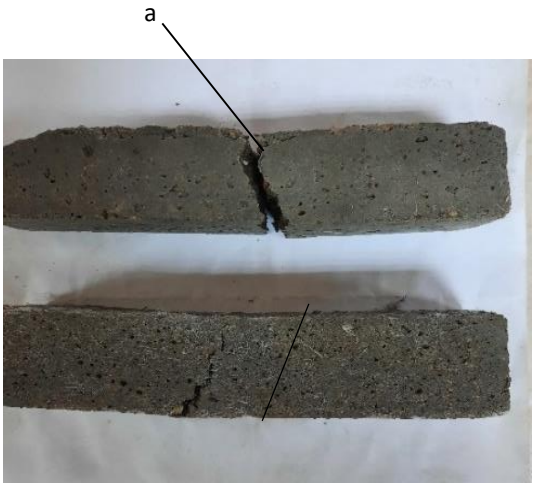

Figure 3 – Samples, after completion of the test. Side view.
a – reference sample; b - reinforced with fiber.

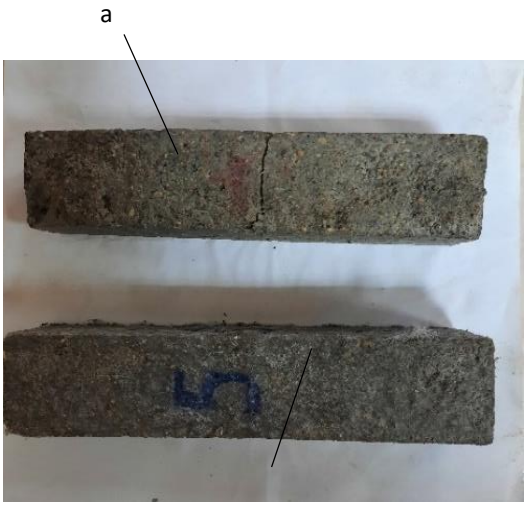

Figure 4 – Samples, after completion of the test. Top view.
a - reference sample; b - reinforced with fiber.

Results and Discussion

Figure 5 shows the results of the reference sample (without the addition of fiber) in bending at the age of 3, 7 and 28 days of normal curing.

Figures 6-9 show the results of determining the bending strength of fine-grained fiber concrete with the addition of polypropylene fiber 0.1, 0.5, 1.5, 2.5% of the weight of the cement.

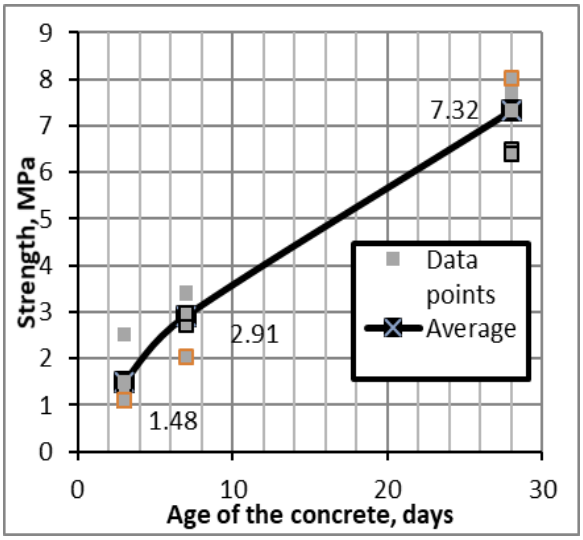

Figure 5 – Reference sample, Fiber content 0%

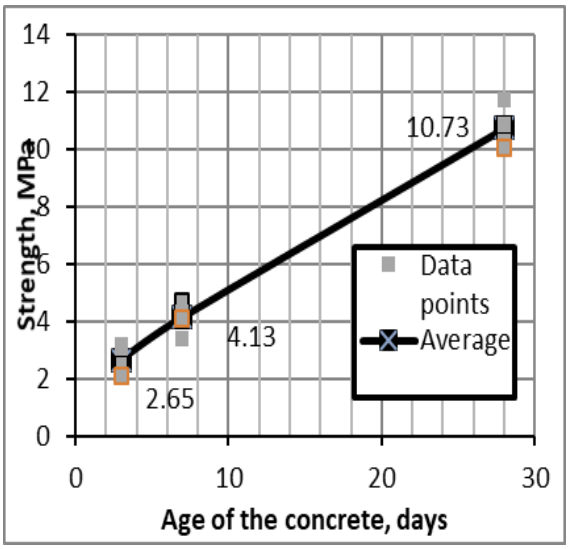

Figure 6 – Polypropylene Fiber content 0.1%

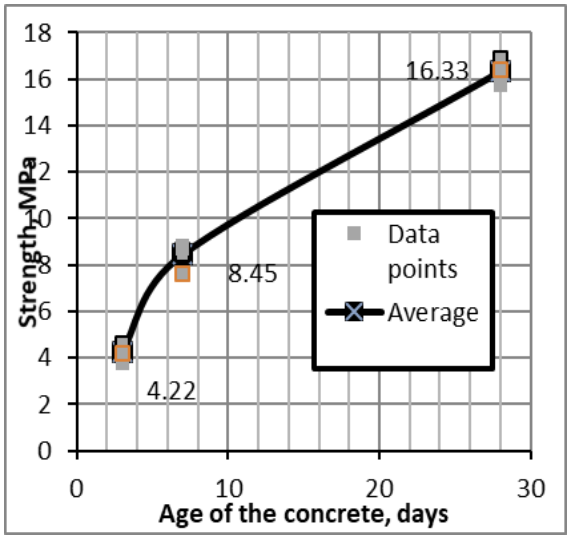

Figure 7 – Polypropylene Fiber content 0.5%

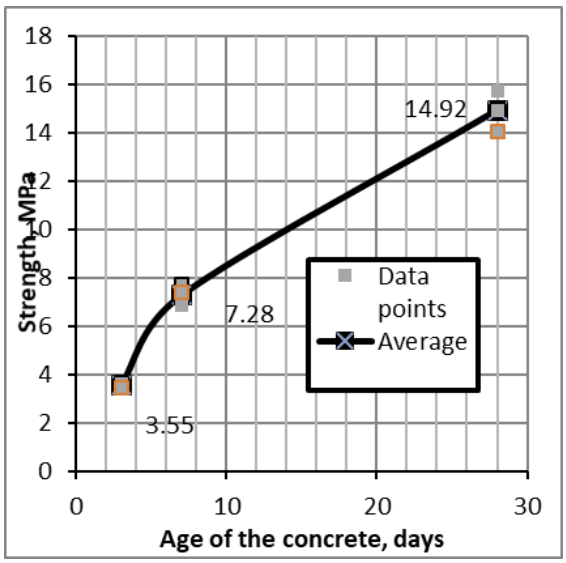

Figure 8 – Polypropylene Fiber content 1.5%

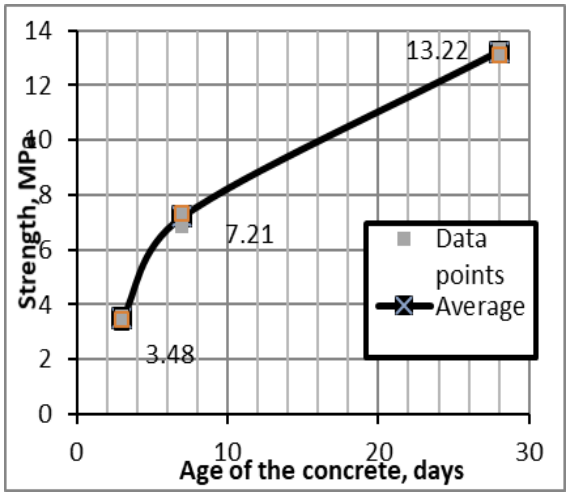

Figure 9 – Polypropylene Fiber content 2.5%

The test results show a positive trend in the dependence of the bending strength on the dosage of polypropylene fiber. According to Figure 6, an increase of 46.5 % in bending strength on 28 days is already observed with the addition of 0.1 % fiber volume compared to the reference sample (Figure 5).

Analysis of the data shows that with the introduction of 0.5% (Figure 7) polypropylene fibers, the ultimate tensile strength of concrete at flexure is in the range 16.33 MPa, and for the reference type 7.32 MPa, that is, this index is 2 times higher than for the reference sample (Figure 5).

A further increase in polypropylene fiber content of 1.5 and 2.5% in fine-grained fiber concrete leads to a gradual decrease in bending strength (Figure 8-9).

Figures 10-13 show the diagram of changes in bending strength with the addition of basalt fiber 0.05, 0.1, 0.2, 0.5% of the weight of cement as a function of curing time.

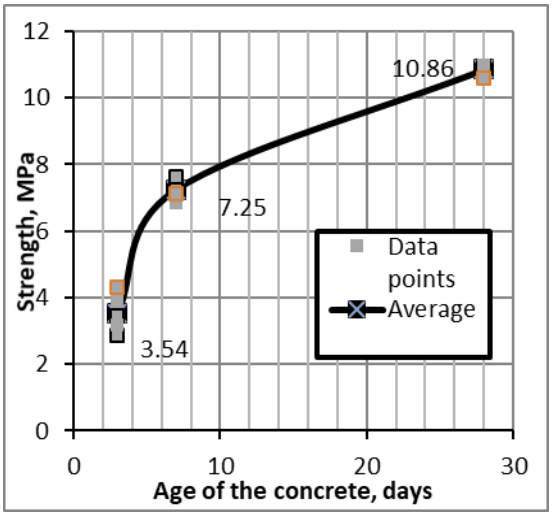

Figure 10 – Basalt Fiber content 0.05%

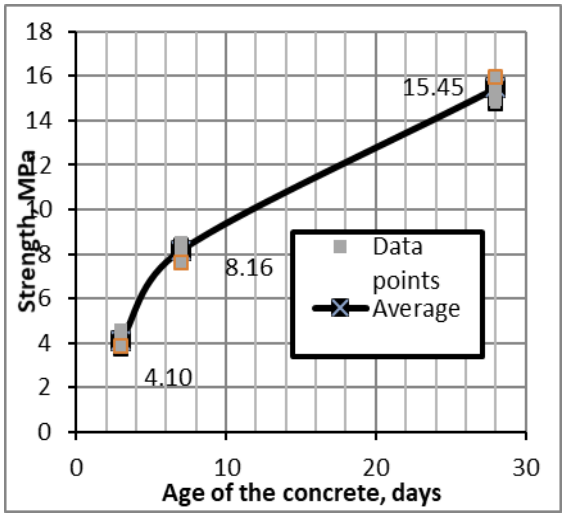

Figure 11 – Basalt Fiber content 0.1%

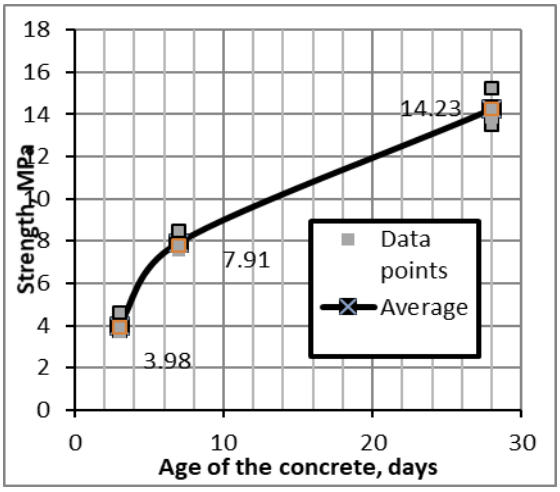

Figure 12 – Basalt Fiber content 0.2%

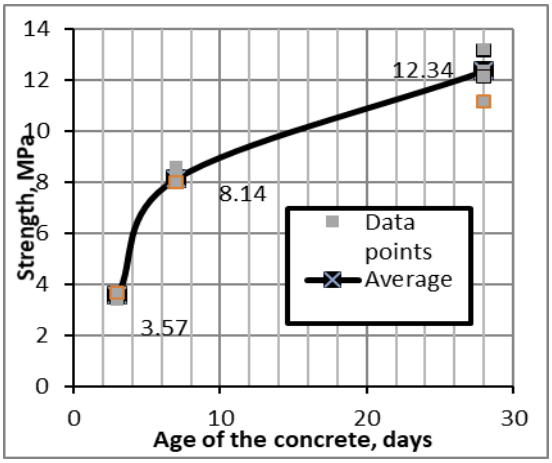

Figure 13 – Basalt Fiber content 0.5%

The introduction of basalt fiber in the concrete mix contributes to an increase in the compressive strength, tensile strength at bending by 5-14%, 24-39% respectively [[22], [23], [24]].

Figure 9 shows that the addition of basalt fiber in an amount of 0.05% bending strength of concrete increases slightly compared to the reference composition.

The most significant increase was observed with the introduction of basalt fiber in an amount of 0.1% (Figure 11) of the bending strength of concrete - an increase of almost 2 times compared with the reference composition.

The 0.2% content of basalt fiber in the concrete also increases the bending strength, but compared with the addition of basalt fiber at 0.1% there is a slight decrease of about 3% (Figure 12).

Figure 14 shows comparative bending tensile results with and without the addition of fibers.

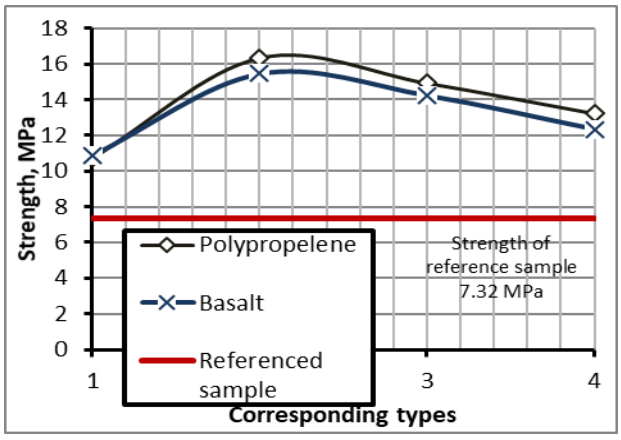

Figure 14 – Comparison of the results of the reference sample with the addition of polypropylene and basalt fiber

The introduction of basalt fiber in the amount of 0.5% also increases the bending strength of concrete, but there is a decline of 28% compared with the addition of fiber 0.1% of the weight of cement (Figure 13).

Figure 14 shows that the bending tensile strength with the addition of various fibers is a slightly higher than that without the addition. The addition of fibers in optimal amounts (polypropylene fibers 0.5% and basalt fibers 0.1%) has the most effective effect to increase the bending tensile strength of concrete.

With the addition of polypropylene and basalt fibers the crack resistance of concrete can be increased. Based on studies, the introduction of fiber in the mixture of fine-grained concrete reduces water separation, as well as increases resistance to cracking and impact [25].

If you visually compare two samples (a - reference sample, b - sample reinforced with fibers) on Figure 4-5, you can see that the reference sample, without fibers, after the load broke into two separate parts, the sample with polypropylene fibers on its surface has no cracks, on the side there are minor cracks. This means that the addition of fibers not only increases the strength of concrete samples, but also increases crack resistance. In general, visual inspection of the samples, after their destruction showed that the distribution of fibers is quite homogeneous. Throughout the cut (fracture) area, elements of fibers were observed.

Conclusions

Accordingly, the addition of polypropylene fiber in an amount of 0.5% of the weight of cement in the concrete mixture of fine-grained concrete allows increasing the flexural strength by 2 times more as compared with the reference composition. This consumption is optimal for the bending tensile strength, as a further increase in the consumption of polypropylene fiber leads to a decrease in the bending tensile strength.

The content of basalt fiber in the concrete mixture of fine-grained concrete in an amount of 0.1% of the weight of cement can increase the bending tensile strength by an average of 40%.

Conflict of interest

On behalf of all the authors, the correspondent author states that there is no conflict of interest.

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Полипропилен және базальт талшықтары қолданылған ұсақ түйіршікті фибробетон

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Қабылданды: *23 қараша 2022*

ТҮЙІНДЕМЕ

Фибробетон үлгілері әртүрлі талшық түрлерімен сыналды. Бетондарды дисперсті армирлеуге арналған полипропилен және базальт талшықтарын эксперименттік зерттеулердің нәтижелері қарастырылады. Фибробетонның беріктік сипаттамаларына талшықтың түрі мен концентрациясының әсер ету заңдылықтары анықталды. Талшықты қоспай (бақылау үлгісі) және цемент массасының 0,1, 0,5, 1,5, 2,5% құрайтын полипропилен талшығын және цемент массасының 0,05, 0,1, 0,2, 0,5% құрайтын базальт талшығын қоса отырып, ұсақ түйіршікті фибробетонның иілу кезіндегі созылу беріктігін анықтау нәтижелері ұсынылған. Полипропилен талшығын ұсақ түйіршікті бетон қоспасына қосудың оңтайлы шегі цемент массасының 0,5% деп санауға болатындығы көрсетілген. Цемент массасының 0,1% мөлшерінде ұсақ түйіршікті базальт талшықты бетон қоспасына қосу иілу кезінде созылу беріктігін арттыруға мүмкіндік береді.

Түйін сөздер: фибробетон, полипропилен талшығы, базальт талшығы, беріктік.

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Мелкозернистый фибробетон с использованием полипропиленовых и базальтовых волокон

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АННОТАЦИЯ

Проведены испытания образцов фибробетона с различными типами фибры. Рассмотрены результаты экспериментальных исследований полипропиленовой и базальтовой фибры для дисперсного армирования бетонов. Определены прочностные характеристики фибробетонов различных составов. Выявлены закономерности влияния типа и концентрации фибры на прочностные характеристики фибробетона. Представлены результаты определения предела прочности на растяжение при изгибе мелкозернистого фибробетона без добавления фибры (контрольный состав) и с добавлением полипропиленовой фибры 0,1, 0,5, 1,5, 2,5% от массы цемента и базальтовой 0,05, 0,1, 0,2, 0,5% от массы цемента. Показано, что оптимальными пределами введения

	полипропиленовой фибры в смесь мелкозернистого бетона могут считаться 0.5 % от массы цемента. Введение в бетонную смесь мелкозернистого бетона базальтовой фибры в количестве 0,1-0,2% от массы цемента позволяет повысить прочность на растяжение при изгибе.
	Ключевые слова: фибробетон, полипропиленовая фибра, базальтовая фибра, прочность.
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Engineering and technology

An advanced method for the development of highly reliable asphalt concrete mixture

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ABSTRACT
This paper introduces the new technology of road construction pavement Superpave. From the beginning of the technology, the method of calculation of road pavement temperature has been taken as an example on the Shymkent city road in Kazakhstan. The material calculation for high quality was conducted with the new climate data of the exact city. A new methodological approach will determine the most accurate selection of bitumen binder grades using a specifically developed PG Grade calculation based on the meteorological data for the period from 2000 to 2020 (20 years) for the specific city. This will be intended to establish requirements for bitumen binders testing by the traditional method for both original and modified bitumen, such as penetration, softening point, and flash and fire point tests, taking into account the climatic characteristics of the republic. Today, have to be accounted that the most common bitumen binder is a 70-100 penetration rating, which means that quite incorrect to use at the highest temperature in Shymkent at +41.3°C and with the lowest temperature at -17.8°C. The results will help to decide on the use of polymer modification of binders, taking into account the design temperatures and operating conditions of asphalt concrete surfaces.
Keywords: bitumen grade, bitumen binder, Superpave, PG Grade calculations, Penetration grading, softening point, flash point.

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Introduction

Over the last few years, traffic on the roads of Kazakhstan and the world as a whole has increased significantly. At the same time, the number of vehicles and the traffic load on the roadway has increased, but with this load, road construction technologies themselves are growing much more slowly than necessary. To solve this problem, the government has decided to commission new roads and repair old ones. According to the road projects being implemented under the state programme "Nurly Zhol", almost all roads built will be provided with both asphalt and cement surfacing. Since the territory of Kazakhstan is subject to a sharply continental climate and the design of the pavement design must take into account for the climatic conditions of the construction area and the appropriate pavement material [[1], [2]].

As road maintenance practice shows, the durability of asphalt pavement is determined by its high plasticity, adhesion, and low-temperature properties as well as by its resistance to thermal oxidation aging.

Compared to closely related cement concrete pavements, they are also characterized by high evenness, good wheel grip, and the absence of expansion joints. All this ensures that not only individual vehicles but also all traffic can travel at the specified speed of up to 150 km/h and more [3].

For its part, the advantages of such pavements include dustlessness and quietness in vehicular traffic, low wear and tear (up to 1 mm per year), and easy maintenance and repair, although there are disadvantages such as increased slipperiness when wet and often short service life due to wave formation, shear due to lack of strength or excessive plasticity, cracking due to excessive brittleness and flaking due to insufficient water resistance. Considering the financial part, which is always perceived as a stimulus to domestic demand for the realization of economic growth, stable development of regions, and urban and rural settlements, the country still needs to carry out further scientific research.

The purpose of the review information is to provide a theoretical basis for an introduction to the scientific and technical developments regarding the improvement of pavement quality in Kazakhstan that has recently been published in the open press.

According to the World Economic Forum's 2017 Global Competitiveness Index, Kazakhstan ranked 115th out of 137 countries in terms of road infrastructure, between Russia and Zimbabwe. According to the information Kazakhstan needs this methodology [4].

Currently, according to the Road Committee of the MTC RK, the total length of Kazakhstan's roads is around 148,000 km, of which more than 93,000 km are public roads, divided into 23,495 km of national roads (including 12,992 km of international roads) and 70,116 km of local roads [4]. This indicates that most of the pavement is asphalt concrete, with the above-mentioned range of benefits, and needs to be improved in order to operate for a long period of time. For this purpose, our scientists such as B.B. Teltaev, E.D. Amirbayev, K.D. Sakanov, B.A. Asmatulaev, B.S. Murtazin etc. are engaged in research on the use of various new materials in construction. For the optimal solution in 2014, they stopped at the material warm asphalt concrete, which significantly reduces energy consumption, and gas emissions into the atmosphere and helps to prolong the construction season for high-performance asphalt concrete surface, also reminded to test polymeric additives in asphalt concrete that improve the properties of bitumen and bituminous emulsions.

The materials used for road construction must also be differentiated according to the characteristics of each road - both in terms of operating conditions and pavement design. For example, some roads are predominantly used by passenger cars, others by large vehicles. On some roads, the volume of traffic rarely changes throughout the year, in others, it depends on seasonal activities such as harvesting and transporting crops from agricultural enterprises [[5], [6], [7]]. For this reason, for each specific road, road builders develop individual asphalt mixes.

To ensure high-quality asphalt mixtures for highways, to make the best possible use of the effort and money invested in their construction - helps the US- Superpave methodology for creating best-performing asphalt pavements [8].

This methodology addresses problems such as classical deformation as well as resistance to fatigue and low-temperature cracking in Figures 1 and 2 [9].

Figure 1 - Classical deformation (rutting)

Figure 2 - Fatigue and low-temperature cracking

The Superpave system incorporates 3 interrelated components, successively updating the AASHTO and ASTM regulatory framework:

- SHARP - specifications and test methods for bitumen;

- Superpave - specifications and methods for the design of asphalt mixtures with mandatory determination of the pore characteristics of asphalt concrete samples at the different compaction stages;

- Test methods and a system for analyzing the rheological properties of asphalt concrete, focusing on

the use of mathematical models of performance and computer software [10].

1. The specifications for bitumen binders and the corresponding rheological test methods are the most complete part of the implemented research programme. Not only the standard test methods have changed dramatically, but also the approach to the standardization of quality indicators of bitumen binders for asphalt mixtures. It is customary to characterize a bitumen binder grade by a temperature performance interval (PG Grade) which corresponds to the minimum and maximum design temperatures of the asphalt pavement in the construction region in question [11].

Experimental technique

Calculation of the pavement temperature. PG grade bitumen binder is determined on the basis of climate change for the last 20 years counting every 7-day average maximum design temperature (instead of the softening point temperature with ring and ball) and the minimum design temperature (instead of the Fraas embrittlement temperature). The maximum temperature which describes the heat resistance of bitumen binder can take values with a graduation of 6℃ in the range from plus 46℃ to plus 82℃. The minimum temperature for the low temperature properties of bitumen can take values in the range from minus 10 ℃ to minus 46 ℃ with the same gradation of 6℃. When selecting a binder grade, the requirements for deformability and viscoelastic properties remain unchanged, but the temperature range in which these requirements have to be fulfilled changes [[12], [13], [14], [15]].

Thus, when designing asphalt concrete mixtures, the bitumen binder grade is selected based on the climatic conditions and the purpose of the asphalt concrete pavement. With respect to the working temperature range for the 4 climatic zones of the USA, the following binder grades were taken as the basic ones: PG 52-28, PG 58-22, PG 58-16 and PG 64-10 [16].

The higher reliability indicated in the figure of 97.5 % means a correspondingly lower risk of selecting an unsatisfactory binder grade. In order to increase the reliability of selecting a satisfactory bitumen grade for the region in question from 50 % to 98 % it is necessary to reduce the low design pavement temperature by 6 ℃ and to increase the maximum design temperature by 4 ℃. The assumptions that remain unchanged are the meteorological data recorded at the nearest weather station in previous years over a period of at least 20 years [17].

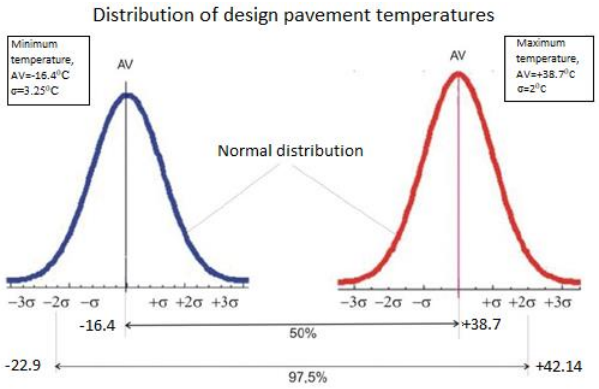


Figure 3 - Example of determining design pavement temperatures for selecting bitumen binder grades (AV-average temperature, σ- standard deviation)

To reduce the risk of plastic deformation in asphalt pavements in case of increased traffic loads as well as in braking areas, it is recommended to use bituminous binders of a higher heat-resistance grade.

Conventional Bitumen Test. Penetration, Ring and Ball Softening Point, Flash and Fire Point tests were used in order to investigate base bitumen and PMB respectively. A standardized needle with a weight of one hundred grams is used in the penetration test. The needle is given five seconds to make its way through the material while the load is being measured. The penetration of the needle into the substance being tested may be determined by the depth of penetration, which is represented in units of 1/10 millimeter. According to ASTM standard D5-13, the penetration grade of the bitumen that was tested is determined by the measurement taken at 25°C.

According to ASTM: D36-12, SP test method consists of determining the temperature at which bitumen, poured and cooled inside rings of given dimensions, under test conditions softens and, moving under the weight of a steel ball, touches the bottom plate.

As the temperature at which a steel ball lying on a bitumen film causes a deformation of 25,4 mm on bitumen.

The bitumen Flash and Fire Point tests provided by AASHTO Designation: T 48-06 or ASTM Designation: D 92-05a. The present test method consists of heating a sample of bitumen in an open cup at a prescribed rate until bitumen vapours flash over the surface of the cup from an ignition device.

Research Results and Discussion

Calculation of the pavement temperature. The city of Shymkent was considered as an example. Initially, the estimated air temperature at a certain reliability is

found. Data from the nearest weather station is processed (Figure 3) The average shade air temperature for the 7 consecutive hottest days of the year over a 20-year period is 38.7℃. There is a 50% probability that it will either be higher or lower than 38.7℃ in any given year. It would not be correct to assume an estimated summer temperature of 38.7℃ because, for example, with a lifetime of 14 years there will be approximately 7 hotter years. Therefore, taking its probability distribution as normal data for 20 years, we find that the standard deviation of the air temperature of the hottest week in a given year is 1.71℃. Using the probability integral table, we find that a two-fold standard deviation corresponds to a reliability of 0.9772, i.e. approximately 98%. Then the calculated summer air temperature will be (38.7℃ +2℃*1.71)= 42.14℃ with a 98% reliability. This means that there may be a hotter summer about once every 50 years.

Similarly, we process observational data from the same weather station on the minimum daily winter temperatures, but here its variation is characterized by a standard deviation of 3.25℃ for a reliability of 98%. Thus, the low temperature is (-16.4℃-3.25℃*2)=-22.9℃. (Fig. 3)

Based on these air temperatures, it is possible to find the calculated pavement temperatures. Then the calculated summer temperature in the pavement at a depth of 2 cm from the surface is expressed by the formula:

$$T_{20mm} = 0.9545(T_{air} - 0.00618Lat^2 + 0.2289Lat + 42.2) - 17.78 \quad (1)$$

where, $T_{air}-$ air temperature in shade, ℃; Lat −northern latitude in degrees, with solar radiation absorption coefficient 0.9; solar radiation transmittance coefficient 0.81; atmospheric radiation 0.7 and wind speed 4.5 m/s taken into account when deriving this formula.

$Lat - 42.18$ degrees north latitude of Shymkent and our found summer air temperature of 42.14℃, we obtain the calculated temperature of pavement in summer $T_{20mm} = 61{,}44$℃.

Taking the estimated winter temperature as the same as the air temperature creates an excessive reserve, according to Canadian researchers, and they have proposed a formula

$$T_{min} = 0.856T_{air} + 1.7 \quad (2)$$

By substituting it with the calculated minimum one-day air temperature of minus 22.9℃, we obtain the calculated winter cover temperature T_{min} =-17.9℃.

According to Table 1 in 6℃ increments we mark the binder, i.e. in our example we have to choose instead of 61.44℃ the nearest high temperature grade PG 64 and low temperature grade PG -22, i.e. for conditions of Shymkent city the binder grade PG 64-22. It turns out that in both cases the reliability is more than 99% as "rounding off" in 6℃ increments gave an additional margin. In view of this, however, our design air temperatures must be revised by reducing the required reliability and the design reliability of the resulting binder grade must be checked after the design pavement temperatures have been determined. As a rule, when the absolute difference between high and low design pavement temperatures is greater than 90°C, a polymer-bitumen binder must be used instead of bitumen.

Thus, the pavement temperature has to be calculated for each region separately, taking into account the individual climatic conditions. This is because, according to studies, temperatures have changed considerably over the last 10-20 years. In fact, all the media are reporting on the negative effects of its rise on society.

In our example of PG 64-22 this difference is (64-(-22))=86 and you can probably get by without modifying the polymer. But for PG 58-34, for example, it would be (58-(-34))=92, and the binder would probably cost about twice as much. According to Figure 4, which is taken from the Kazakhstan Highway Research Institute recommendation document, 80% of the republic needs a modification to bitumen which is up to 90. After recalculating the data, the percentage of regional modification bitumen might change, because there are differences in Shymkent city between our calculation with new weather data PG 64-22 and their calculation with 40 years past data PG 64-28 (purple region).

For each bitumen, the requirements for climatic operating conditions are determined, i.e. for resistance to rutting, fatigue failure, and low-temperature cracking at design operating temperatures.

Before testing, the binder is first subjected to artificial thin-film aging in an RTFO oven to simulate the short-term aging process during asphalt mix preparation, transportation and road laying and then to a high temperature and pressure (PAV) chamber to simulate the long-term oxidative aging process under years of pavement use. The dynamic shear rheometer test is then used to calculate the resistance to permanent deformation accumulation (rutting) and flexural fatigue of the pavement. It is intended to

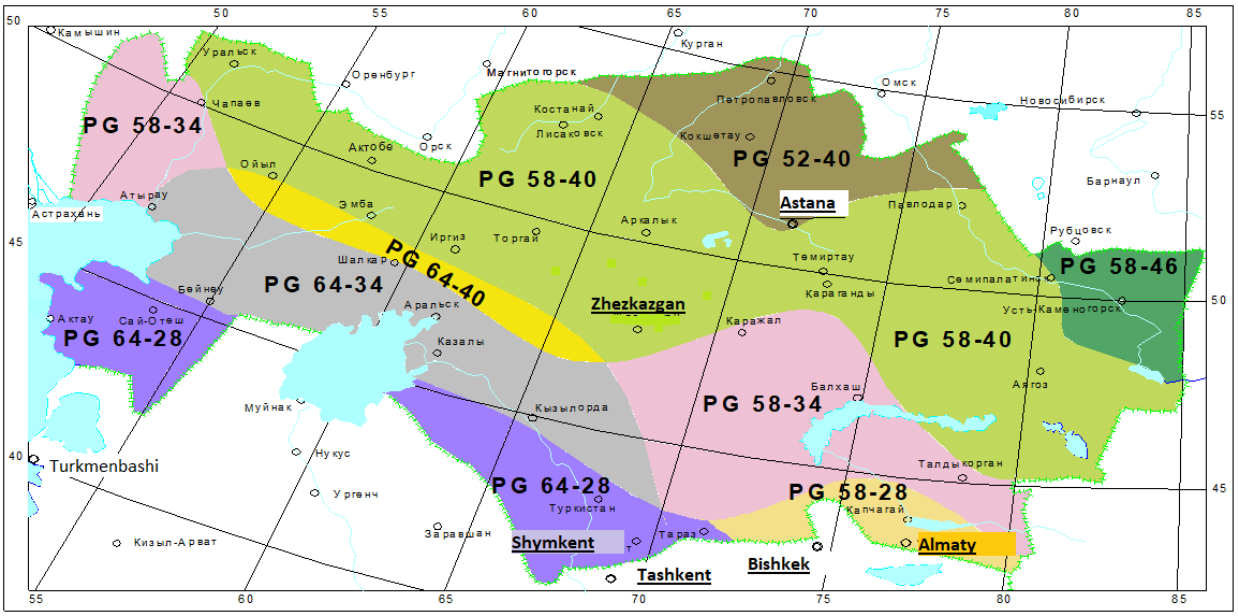


Figure 4 - Zoning map of Kazakhstan [10]

determine a complex shear modulus G* and phase angle δ of bituminous binders at design pavement temperatures. The tests are carried out according to the standard [[18], [19], [20]].

It can be seen from table 1 that the lowest temperature is minus 46℃ and the highest is 82℃. In fact, in the northern states, the cover temperature can drop to minus 46℃, but there is no state where the average 7-day summer cover temperature reaches 82℃. It usually does not exceed 70℃.

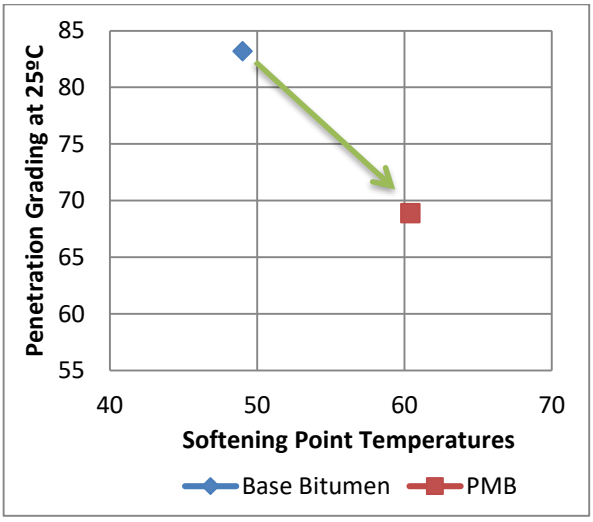
The point is that the grade of PG 64-22 indicates its suitability for 64℃ summer design temperature and for winter design temperature of minus 22℃ in fast-moving traffic (over 70km/h) of average intensity (up to 10 million axle passes with a design load of 80kN during the service life of the binder). But for sections with slow traffic (for example, for taxiways of airfields or for intersections), where the average speed is concluded within the limits of 20-70 km/h or for sections, where the number of passes during design load on one lane of roadway exceeds 10 million axles, it is recommended to choose the binder one grade (by 6℃) "hotter", i.e. in our example for Shymkent PG 70-22. If these conditions are combined, it is recommended that a two grades hotter binder is selected, i.e. PG 76-22.

For very slow traffic areas where the average speed does not exceed 20 km/h, two grades hotter, i.e. also PG 76-22, are selected. Therefore, an area with a realistic design summer pavement temperature of 70℃ may require a maximum grade shift of 12℃, which is accounted for in the table by the introduction of PG 82 [[21],[22],[23],[24],[25]].

Table 1 - Binder grades according to operating conditions (PG)

High temperature grades, ^{0}C	**Low temperature grades, 0C**						
PG 46	-34	-40	-46				
PG 52	-10	-16	-22	-28	-34	-40	-46
PG 58	-16	-22	-28	-34	-40		
PG 64	-10	-16	-22	-28	-34	-40	
PG 70	-10	-16	-22	-28	-34	-40	
PG 76	-10	-16	-22	-28	-34		
PG 82	-10	-16	-22	-28	-34		

Conventional Bitumen Tests Results. In the study that was done on the qualities of base bitumen and PMB, the findings showed that using a polymer composition led to a considerable improvement in most of the indicators. This was discovered via the examination of those data. In particular, the needle's ability to penetrate bitumen at a depth of 25 °C is lowered by 14.3 millimeters. The temperature at Softening point test begins to soften and rises from 49 degrees Celsius to 60.4 degrees Celsius, which lessens the propensity of bitumen to distort. The Flash Point temperature also increased for 12 degrees. All tests results given in following graphs.



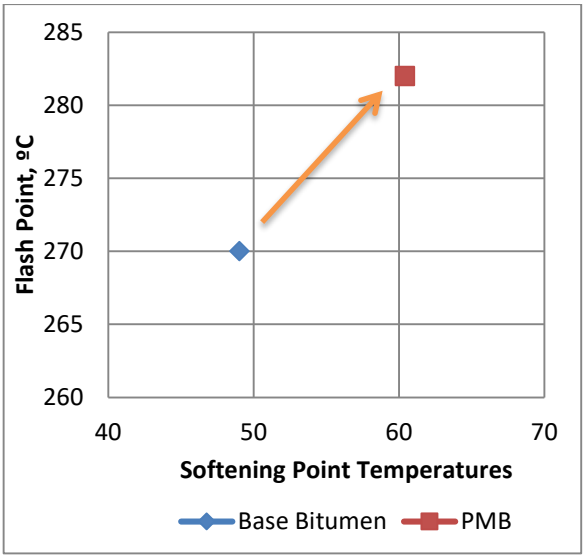


Figure 5 - Conventional Bitumen Tests Results

Figure 5 shows the results of penetration and softening point tests performed on bitumen. As can be seen in Figure 5, the penetration tended to decline to add any kind of polymer, here the Butonal NS was used as a polymer. With the addition of polymers, the bitumen's softening point increased, corroborating the penetration test results. These findings suggest that adding polymers to bitumen increases bitumen hardness significantly. However, because the bitumen gets harder following alteration, this is accompanied by a reduction in elastic recovery. Polymers, on the other hand, had an impact on bitumen's elastic characteristics, with the percentage of elastic recovery increases, the Butonal NS works well in increasing the recovery process between 85-95%. In terms of flash point testing, both the basic and polymer-modified bitumen samples were found to have values that were higher than the specified minimum of 220 ºC, on the other hand, there was still an increase with adding polymer.

Conclusions

Thus, in order to develop requirements for bitumen binder and asphalt concrete taking into account climatic features of the republic, as well as to make a decision on the use of polymer-bitumen binders and asphalt concrete on their basis taking into account design temperatures: its temperature in winter and summer, design traffic intensity and traffic flow rate of asphalt concrete surface calculations at 20 mm depth was studied. Furthermore, for the 64°C pavement temperature of Shymkent city, just with traditional ways of testing bitumen it was obvious that base bitumen characterizes wouldn't work properly, and could not resist to road loads without adding polymer.

In Kazakhstan, in 2013 the Kazakhstan Highway Research Institute (KazdorNII) developed a recommendation which is called "Rezoning of Kazakhstan Territory by Design Temperatures for Asphalt Concrete Pavements". However, the information in this report is based on climate change between 1987-2006, i.e. it is not considered suitable for the preparation of asphalt concrete at present. The data needs to be recalculated using climate change data for the last 20 years to determine the composition of high-strength asphalt concrete pavements.

Conflict of interest. On behalf of all the authors, the correspondent author states that there is no conflict of interest.

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ТҮЙІНДЕМЕ

Бұл мақалада Superpave жол төсемдері құрылысының жаңа технологиясы ұсынылады. Технологияның басынан бастап жол төсемінің температурасын есептеу әдістемесі келтіріледі және мысал ретінде Қазақстандағы Шымкент қаласының жолы алынады. Жоғары сапалы материалды есептеу нақты осы қаланың жаңа климаттық деректерімен жүргізілді. Жаңа әдіснамалық тәсіл нақты қала үшін 2000 жылдан 2020 жылға дейінгі кезеңдегі (20 жыл) метеорологиялық деректерге негізделіп арнайы әзірленген PG бағасының есебін пайдалана отырып, битум байланыстырғыш сорттарын неғұрлым дәл таңдап анықтайды. Бұл республиканың климаттық ерекшеліктерін ескере отырып, енуге, жұмсарту температурасына, тұтану температурасына сынақтар сияқты бастапқы және модификацияланған битумға дәстүрлі әдіспен битумды байланыстырғыштарды сынауға қойылатын талаптарды белгілеуге бағытталатын болады. Бүгінгі таңда ең көп таралған битум тұтқыры 70-100 пенетрация дәрежесіне ие екенін, яғни оны Шымкенттегі ең жоғары температурасы +41,3°С және ең төменгі температурасы -17,8°С болып тұрғанда пайдалануға болмайтынын ескеру қажет. Нәтижелер асфальтбетонды беттердің есептік температуралары мен жұмыс жағдайларын ескере отырып, байланыстырғыш заттардың полимерлі модификациясын қолдану туралы шешім қабылдауға көмектеседі

Түйін сөздер: битум маркасы, битум байланыстырғыш, Superpave, PG Grade есептеулері, ену дәрежесі, жұмсарту температурасы, тұтану температурасы.

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Усовершенствованный метод разработки высоконадежной асфальтобетонной смеси

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АННОТАЦИЯ

В данной статье представлена новая технология строительства дорожных покрытий Superpave. С самого начала применения технологии, метод расчета температуры дорожного покрытия был взят в качестве примера на дороге города Шымкент в Казахстане. Расчет материала на высокое качество был проведен с учетом новых климатических данных именно этого города. Новый методологический подход позволит определить наиболее точный выбор марок битумного вяжущего с помощью специально разработанного расчета PG Grade на основе метеорологических данных за период с 2000 по 2020 год (20 лет) для конкретного города. Это будет направлено на установление требований к испытаниям битумных вяжущих традиционным методом как для оригинального, так и для модифицированного битума, таких как испытания на пенетрацию, температуру размягчения, вспышку и температуру возгорания,

	с учетом климатических особенностей республики. На сегодняшний день приходится учитывать, что самое распространенное битумное вяжущее имеет степень пенетрации 70-100, а это означает, что совершенно некорректно использовать его при самой высокой температуре в Шымкенте +41,3°C и при самой низкой температуре -17,8°C. Полученные результаты помогут принять решение о применении полимерной модификации вяжущих с учетом расчетных температур и условий эксплуатации асфальтобетонных покрытий
	Ключевые слова: марка битума, битумное вяжущее, Superpave, расчеты марки PG, градация пенетрации, температура размягчения, температура вспышки.
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Earth sciences

On the degree of influence of waterflooding on the oil recovery factor from productive formations of high-viscosity reservoirs X, represented by terrigenous reservoirs

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ABSTRACT

On the basis of the generalization of experience in the development of multilayer high-viscosity fields X the influence of waterflooding in the late stage on the oil recovery factor of productive formations has been studied. By applying statistical methods of data processing the dependence of the oil recovery factor on the reservoir flushing factor, with a sufficiently high correlation coefficient, has been obtained. The dependence obtained confirms the theoretical basis of oil recovery from productive formations developed with waterflooding and can be used when designing the process on similar objects. In many oil-producing regions of the world, the tendency of deteriorating quality of the resource base and incomplete replenishment of oil production by the growth of their reserves due to the discovery of new fields is observed. At the same time, the costs of prospecting and exploration works are increasing, and geological, andphysical conditions and specific reserves per each discovered field are worsening.

Keywords: Field, deposit, development, flooding, flushing, extraction, compensation, selection.

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Introduction

As a result, an increasing number of fields are prepared for the development of a limited number of prospecting and exploratory wells, there is a problem associated with the lack of basic geological and field data for the reliable calculation of hydrocarbon reserves and addressing issues of a rational and profitable development of these objects. It is necessary to note, that now a number of these fields are developed by single exploration wells, and the overwhelming part of them is mothballed and commercially developed.

There is an urgent need to solve the issues of the effective development of these objects, including waterflooding since this method is the most widespread method of increasing the oil recovery factor due to relative ease of implementation and availability of water.

To solve these problems in practice, various methods of forecasting development indicators are used, which are conventionally divided into the following two groups:

1. Personal experience of the specialist and intuition.
2. The method of analogy.
3. empirical methods based on data correlation.
4. Waterflood and depletion curves.
5. Analytical mathematical models.
6. Numerical mathematical models.

Numerical mathematical models are the most modern, they allow taking into account known physical processes, heterogeneity of formation properties and complexity of structure, arbitrary well operation and their irregular arrangement, etc. At the same time, numerical mathematical models require a large quantity of quality input data, a high qualification of a specialist, and a lot of working time. Therefore, when analyzing and predicting the development, the need arises to use empirical dependencies and various waterflood curves, obtained on the basis of the generalization of geological and field data of long-developed fields.

For instance, this approach was used for substantiation of optimal compensation by the surrounding production wells and reduction of risk of water breakthrough by injecting wells through highly permeable reservoirs.

The waterflood curves are used to evaluate the influence of injected water volumes on the technological performance of wells. All the constructions and conclusions are based on the compensation of withdrawals by injection for each producing well.

In papers [[1], [2], [3], [4], [5]], the dependencies obtained as a result of the generalization of the experience of oil field development in the Timan-Pechora province of the Russian Federation are used to predict development indicators and the correctness of calculation results based on hydrodynamic models.

The results of the generalization show that one should not expect unambiguous answers from experts about flooding efficiency due to the diversity of mining and geological conditions of each oil-bearing region of the world, as well as geological and physical features and technological solutions implemented at specific sites. Despite this and considerable progress in the direction of providing a theoretical basis for the development of oil fields, the results of analysis, generalization, and comprehension of the accumulated experience do not lose their importance.

In this connection, let us consider a generalization of reservoir flushing factor (RFF) and oil recovery factor (ORF) dependence on the example of long-developed deposits of high-viscosity field X, represented by terrigenous reservoirs.

Research Materials

The geological structure of the X field and the features of their development are considered in many works. A more detailed description of the parameters of geological and physical conditions and the realized systems of development are given in works [[6], [7], [8]]. However, the authors of this study found it necessary to give a brief description of the geological and physical conditions and the implemented systems of development of field X, which is as follows.

Neogene, Paleogene, Mesozoic (Cretaceous, Jurassic), and Paleozoic sediments are involved in the structure of the X field area. The total thickness of the sedimentary cover in the central parts of the depression is more than 10.0 - 12.0 * 10^3 m. in the apparatus - 2.5 - 4.0 * 10^3 and more [[9], [10]].

A characteristic feature of the distribution of hydrocarbon deposits is a significant increase in gas content down the section. While Neogene and Paleogene sediments are mainly oil-bearing, and free gas accumulations are associated with gas caps and single gas deposits, in the Cretaceous and Jurassic sediments gas and gas condensate deposits are predominantly developed. In the Paleogene section, up to eight productive strata are distinguished. Strata I, III, and IV are represented by fine-grained sandstones and siltstones. Formations V, VI, VII, VIII, and IX are carbonate rocks (limestones and dolomites) [[11], [12]].

The reservoirs of the Mesozoic productive strata are, as a rule, sandstones with interlayers of siltstones. Only some horizons of the upper and lower Cretaceous are represented by limestones.

Known small deposits of oil are of non-industrial importance oil inflows from them are short-term and unstable.

Oils of Paleogene deposits are mainly light (826-884 kg/m^3), low-sulfur (0.05-0.75 %), paraffinic (1.4-10.1 %), highly resinous (silica gel tar 5.29-30.2). The viscosity of formation oils is low - 0.7-0.6 mPa*s, with initial gas saturation from 2-5 to 100-150 m^3/t.

Oil deposits are confined to narrow asymmetric folds, the length of which is 10 15*10^3 m, width does not exceed 2-3*10^3 m, bed dip angles are 20-30ºC and more. Known oil and gas reservoirs are mainly of formation-well type /5, 9, 10/. However, as a result of intensive tectonic activity, tectonically screened reservoirs are observed among them according to the degree of their complication by disturbances. Lithological screened deposits in the region are distributed limited.

The productive deposits of the objects under consideration are heterogeneous; they are characterized by layered, zonal heterogeneity and uneven fracturing.

Almost all fields are multilayer. Oil reservoirs are characterized by low height, a small difference between the initial reservoir pressure and the pressure of oil saturation with gas.

During the development of the studied oil reservoirs, regardless of the type of reservoirs, due to their small depth of comparable size (oil reserves), almost identical development systems were implemented.

The following features of the implemented systems are highlighted: [[5], [7], [8]]:

- drilling of deposits by a relatively dense network of wells, placed in a triangular grid;

- joint exploitation of deposits of KKS, Ia, Ib, III horizons of some fields;

- deposit exploitation in the initial period on a natural mode with subsequent use of various waterflooding systems (deposits with relatively small reserves are developed without reservoir pressure maintenance).

Due to close values of initial reservoir pressure of oil deposits and pressure of oil gas saturation, as well as late application of flooding, and low activity of contour waters, which more often than not had no significant influence on the development process, the vast majority of oil deposits were drained in the initial stage of development in the mode of dissolved gas.

Reservoir pressure maintenance by water injection began with waterflooding. Exploration wells and waterflooded oil wells were typically used to perform waterfloods underwater injection. Earlier studies on the efficiency evaluation of waterflooding point out that in spite of a number of factors favorable for its successful application (small size of deposits, low oil/water viscosity ratio), it turned out to be relatively inefficient due to [[13], [14], [15]]:

- poor hydrodynamic connection of the deposits with the zonation zone, due to a sharp deterioration of the reservoir properties of the productive formation in the area of the initial water-oil contact. The specified factor impeded the development of the designed fund for injection wells, as a result, the latter covered only separate, not long stretches of the perimeter of the oil-bearing area, and flooding had a focal character;

- significant heterogeneity of producing objects, caused by the presence of tectonic and lithologic screens and extensive zones of an outcropping.

Under the influence of these factors pumping affected small areas of the deposit, and most often only some of the production wells. Pressure redistribution occurred very slowly and unevenly, its growth was noted mainly in the zones of injection, while the central parts of reservoirs continued to be developed in the depletion mode:

- big difference in permeability of reservoirs, which did not allow even by increasing the injection pressure to cover the whole deposit by the flooding effect. When the injection pressure was increased, most of the injected water flowed into the borehole area or through communicating fracture systems penetrated deep into the reservoir, prematurely watering the production of the producing wells. After displacing small amounts of oil from the more permeable fractured interlayers, the injected water subsequently advanced along the same path, isolating areas of the reservoir with low permeability.

In 1960-1965, in order to intensify the waterflood process, many reservoirs widely used the transfer of the injection line from the initial to the current oil-bearing contour and the development of various types of intracircuit

waterflood. The transition from bypass to various types of bypasses waterfloods stabilized reservoir pressure and increased annual oil withdrawals.

Implementation of this flooding enabled many reservoirs to increase the efficiency of injected water use owing to exclusion of water leakage into the flooded zone, to stabilize the pressure in those zones of the deposit, which were not influenced by water flooding during flooding, to embrace tectonic and lithologic screened zones of deposits [[5], [7]].

At present all objects under consideration are at the fourth stage of development, which is characterized by low rates of oil recovery - less than 2.0% of the initial recoverable reserves, high watering of produced oil, and depletion of reserves of (more than 90%), a significant drop in reservoir pressure, despite the implementation of measures to maintain it and relatively low values of the oil recovery factor.

The achieved ORF values in connection with the presence of objects in the closing stage of development (in a part of them the development has already been suspended because of full watering of the produced oil) are close to their final values. Therefore, we consider the achieved ORF values as a result of the efficiency of the implemented development system, in particular the waterflooding method efficiency.

Research Methods

With the introduction of a new generation of multidimensional computer modeling software in recent years, there was a qualitative development of methods for the geological and hydrodynamic study of oil fields. As the experience of these software applications, the results are determined by the completeness and quality of the initial geological and field information.

Solving geological and development tasks on the basis of using literature and reference data or analogue objects when building geological and hydrodynamic models of oil fields can lead to distortion and reduction of the credibility of the results [[12], [16]].

In this connection, with the lack of necessary qualitative initial geological and reservoir information, research to solve various problems of development of models based on integral indices and obtained by a generalization of geological and reservoir materials of long-operated deposits is also relevant in the current level of knowledge of the theory and practice of oil field development [13].

On the basis of the above-mentioned arguments, we used the least squares method, which is the basic method of regression analysis in order to solve the assigned task. In connection with a choice of a method of studying the set problem, it is appropriate to remind the words of the scientist-geologist V.I. Vernadsky "Not hypotheses and theories, but scientific facts and empirical generalizations come into conflict with the theory but confirmed by new facts, then the scientific theory should change, not to contradict empirical generalizations" [17].

Theory. Currently, the main oil reservoir development technologies are based on the water displacement of oil. The efficiency of these technologies depends on the geological and physical properties of oil-saturated reservoirs, oil and water properties, and extraction conditions. As the experience of oil field development shows, the oil recovery factor (ORF) from reservoirs during waterflooding is most strongly influenced by: the ratio of oil and water viscosity; reservoir heterogeneity in permeability; average permeability and dissection, reservoir temperature; relative sizes of water-oil zones; microheterogeneity of the porous medium; oil saturation and capillary forces; density of the net and waterflood systems [[8], [9], [12], [13], [18]].

In [18] the results of studying the degree of influence of these factors on the oil recovery factor on the basis of multifactor analysis of 50 objects of the Ural-Volga region. The analyzed objects, confined to terrigenous reservoirs, were at a late stage of development. Out of 50 studied objects, 18 were developed by in-situ flooding, 15 - by out-of-situ flooding, and 17 - under the conditions of a natural water-storage regime. The values of geological and production factors at the analyzed objects changed in a rather large range: oil/water viscosity ratio from 1 to 25; average permeability from 0.15 to 2.5 μm^2; reservoir temperature from 25 to 75 ^oC; effective oil-saturated thickness from 3 to 20 m; sand ratio from 0.55 to 0.95 relative reserves of water-oil zone from 25 to 100%; oil saturation from 0.75 to 0.95; well grid density from 10 to 60 ha/sq.m.; fluid production rate from geological reserves from 2.5 to 7.5% [18].

In the indicated ranges of geological and production factors changes, their relative influence on the ORF was (%): oil/water viscosity ratio -21.1; average permeability +15.4; reservoir temperature +7; effective oil-saturated thickness +6; sand ratio +6; relative reserves of water-oil zone -5.6; oil

saturation +3.6; well grid density -3; flooding system +2.2; fluid production rate from geological reserves +0.6 ("+" and "-" are the positive and negative influence of factors, respectively) [18].

Thus, we can conclude that in the considered ranges of changes of geological and production factors, the strongest influence on EOR is provided by: the ratio of oil and water viscosity and average permeability of strata; almost the same influence - reservoir temperature, effective oil-saturated thickness, sand ratio and relative reserves of the water-oil zone; the least influence - oil saturation, the density of well grid, flooding system and the rate of fluid extraction.

At the same time, multifactor analysis established that at different stages of development, the degree of influence of geological and field factors on ORF changes. For example, at later stages the degree of influence of oil and water viscosity ratio decreases, whereas the role of well grid density increases. But at all stages of development, the dominating influence of natural factors on the oil recovery ratio is preserved.

At the same time according to many scientists, the reservoir operating mode is a determinant one. However, the wide use of this method of oil field development is impossible without its further improvement. In this connection, a lot of attention has been paid to the peculiarities of artificial waterflooding of reservoirs in various geological and physical conditions and to the ways of its improvement. Such studies, as is well known, on the one hand, allow using the accumulated experience of reservoir operation by artificial water flooding in the process of designing the development of new fields, and on the other hand, contribute to effective post-development of depleted objects, in which great material and technical resources have already been invested [[12], [13]]. This is evidenced by numerous works devoted to various issues of oil field development in Bashkortostan, Tatarstan, Kuibyshev, Orenburg and Perm regions, Ukraine and Azerbaijan using waterflooding [[18], [19]].

The dependence of oil recovery on the completeness of reservoir flushing in reservoir development with waterflooding is the basis of the well-known expression [20]:

$$ORF = K_d * K_{cv}, \quad (1)$$

where Kd - displacement coefficient, which is the ratio of displaced oil volume to its initial volume in the reservoir during prolonged and intensive flushing of the homogeneous element of the porous medium; Kcv - coefficient of reservoir coverage by volume of impact processes.

Coefficient Kd and Kcv change in time, as the front of incoming water into the reservoir, as it advances captures more new areas of the reservoir, layers, and with changes in the direction of filtration flows, stagnant and dead-end zones.

To evaluate the efficiency of the implemented development systems, many researchers recommend using the dependence of ORF on the degree of formation flushing [[12], [13], [18]].

In this case, as a criterion for evaluating the technological efficiency of development systems implemented in the field is taken as achieved, the oil recovery factor at the same degree of flushing the volume of pores occupied by oil,

$$ORF = ƒ(\tau), \quad (2)$$

where $\tau = \frac{\sum Qf}{IGOR}$ – flushing rate, $\sum Q_f$ – accumulated fluid withdrawal under reservoir conditions; IGOR - initial geological oil reserves.

In contrast to numerous forms and types of displacement characteristics long used in practice, this method is convenient because it allows to use of primary, and therefore less distorted source data, such as fluid withdrawal, accounted in the field conditions reliably enough, geological oil reserves at the late and final stages of development of category A + B, coefficients of conversion of physical parameters of the fluid into reservoir conditions and vice versa. The flushing ratio τ, being a relative value, is convenient for comparison, since it is equally applicable in the analysis of both small deposits and large fields [[12], [13], [20], [21]].

Calculation. Earlier in [13] for FNGO oil reservoirs represented by terrigenous reservoirs, straight-line dependences of ORF on RFF were proposed:

- for the whole sample

$$ORF = 0{,}1266 + 0{,}2329 * \tau, \quad (R=0{,}8890); \quad (3)$$

- for objects developed in the natural mode

$$ORF = 0{,}0169 + 0{,}5604 * \tau, \quad (R=0{,}8983); \quad (4)$$

- for objects developed with the use of water flooding

$$ORF = 0{,}1346 + 0{,}1946 * \tau, \quad (R=0{,}8700); \quad (5)$$

Analysis of the proposed dependences (3) - (5) show that at values τ > 1.5, the ORF becomes more than 1, which does not make physical sense.

In this regard, the initial data used in /13/ for obtaining dependencies (3) - (5) we re-processed and obtained exponential dependences (Fig. 1 - 2) of ORF on RFF:

- for all 25 development sites

$$ORF = 0{,}6996 * (1 - e^{-0{,}8915*\tau}), \quad (R=0{,}824); \qquad (6)$$

- for 16 objects developed with water flooding

$$ORF = 0{,}7553 * (1 - e^{-0{,}6204*\tau}), \quad (R=0{,}953); \qquad (7)$$

- for 9 objects developed without waterflooding

$$ORF = 0{,}2241 * (1 - e^{-0{,}8915*\tau}), \quad (R=0{,}954); \qquad (8)$$

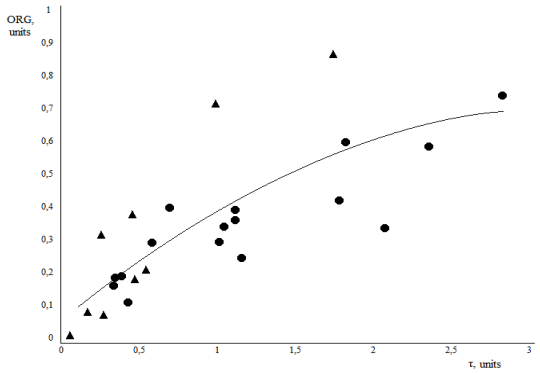

Fig. 1. Dependence of the oil recovery factor on the reservoir flushing ratio for all FNGO objects represented by terrigenous reservoirs:
●- objects developed by waterflooding;
- objects developed witho ▲ waterflooding.

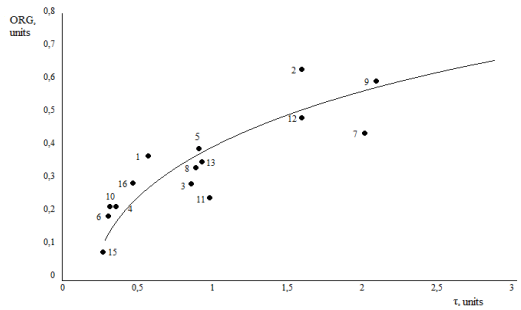

Fig. 2. Dependence of the oil recovery factor on the reservoir flushing ratio for FNGO objects, represented by carbonate reservoirs and developed with the use of waterflooding:

1 - Andijan field, CCS + I horizons; 2 - Andijan field, III horizon; 3 - South Alamyshik field, I+Ia horizons; 4 - South Alamyshik deposit, Ib horizon; 5 - South Alamyshik deposit, CCS horizon; 6 - South Alamyshik deposit, III horizon; 7 - South Alamyshik deposit, XVIII horizon; 8 - South Alamyshik deposit, XIX-XXII horizons;
9 - Khojaabad deposit, BPS+I horizons; 10 - Khojaabad deposit, III horizon; 11 - Western Palvantash deposit, BRS horizon; 12 - Western Palvantash deposit, III horizon; 13 - Hankyz deposit, II horizon; 14 - Chongora-Galcha deposit, IV horizons; 15 - Boston deposit, CCS+ I+Ia+Ib horizons; 16 - Boston deposit, III horizon.

Analysis of the obtained dependences (7) - (8) show that their separate consideration for the objects under development with and without waterflooding considerably increases their reliability in comparison with the earlier proposed (4) - (5). This allows us to conclude with their more correct description of the mechanism and efficiency of waterflooding of oil deposits.

Conclusions

The obtained results, the dependence of ORF on RFF, show that the mechanism of reservoir flushing by water inflow from the water-bearing area and water injection into the formation through the wells is the same. The achievable EOR value is determined by the degree of formation flushing. However, after each increase in the flushing ratio, the volume of displaced oil naturally decreases. The difference in the ORF values at the identical values of CPR for the objects being developed with and without waterflooding testifies that the effect of water injection is achieved due to the increase of coverage of the reservoir volume by the drainage.

It is recommended to use the obtained dependences of ORF on RFF to assess the flooding efficiency, and to compare and refine the results of hydrodynamic calculations under similar geological and physical conditions of the objects and the implemented development systems.

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Терригенді коллекторлардан тұратын, ұсынылған Х кен орнының тұтқырлығы жоғары коллекторларының өнімді қабаттарынан мұнай алу коэффициентіне су айдаудың әсер ету дәрежесі туралы

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АНДАТПА

Бұл мақалада көп қабатты тұтқырлығы жоғары кен орындарын игеру тәжірибесін жалпылау негізінде соңғы кезеңдегі суланудың салдарынан өнімді қабаттардың мұнай беру коэффициентіне әсері зерттелді. Деректерді өңдеудің статистикалық әдістерін қолдану арқылы мұнай алу коэффициентінің салыстырмалы түрде жоғары корреляция коэффициентімен қабаттарды жуу коэффициентіне тәуелділігі алынды. Алынған тәуелділік су айдау арқылы жасалған өнімді қабаттардан мұнай алудың теориялық негіздерін растайды және оны ұқсас объектілерде процесті жобалау кезінде қолдануға болады. Сулану жағдайы құбылыстары, соның ішінде қабат қысымын су айдау үрдісі арқылы тиімді дамыту мәселелерін шұғыл шешу қажет, өйткені бұл әдіс су айдау салдарынан мұнай беру коэффициентін арттырудың ең кең таралған әдісі болып табылады. Өнімнің сулану үрдісін тежеу және бақылау істері есепке алынған.

Түйінді сөздер: кен орны, игеру, су айдау, жуу, өндіру, өнім**.**

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О степени влияния заводнения на коэффициент извлечения нефти из продуктивных пластов высоковязких коллекторов месторождения Х, представленных терригенными коллекторами

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АННОТАЦИЯ

В статье рассматривается на основе обобщения опыта разработки многослойных высоковязких месторождений изучено влияние заводнения на поздней стадии на коэффициент нефтеотдачи продуктивных пластов. Путем применения статистических методов обработки данных получена зависимость коэффициента извлечения нефти от коэффициента промывки пласта с достаточно высоким коэффициентом корреляции.

	Полученная зависимость подтверждает теоретические основы извлечения нефти из продуктивных пластов, разрабатываемых заводнением, и может быть использована при проектировании процесса на аналогичных объектах. Возникает острая необходимость решения вопросов эффективной разработки этих объектов, в том числе заводнением, так как этот метод является наиболее распространенным методом повышения коэффициента извлечения нефти за счет заводнения.
	Ключевые слова: Месторождение, месторождение, разработка, заводнение, промывка, добыча, компенсация, отбор.
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Earth sciences

The use of chlorine-containing agents in the processing of spent blocks of uranium deposits

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ABSTRACT

The work is aimed at diversifying existing mines for the extraction and processing of natural uranium through additional processing of spent blocks of uranium deposits with chemical solutions using the method of in-situ well leaching (ISL) in order to extract associated useful components. A feature of this technology is the use of the existing production infrastructure for the extraction of associated useful components in existing uranium mines, without significant capital investments in production infrastructure and mining operations. The technology of underground borehole leaching has been reliably developed in uranium deposits for decades. The fundamental similarity of the technology for the extraction of uranium and a number of associated useful components (APC) - by the ISR method, allows the use of spent ore fields of uranium deposits for the extraction of PPC. The use of ready-made technological infrastructure (wells, pipeline network, pumping equipment, control units, etc.) allows, due to savings on infrastructure costs, to obtain profitability when mining ore-bearing blocks with a content of recoverable components from ≤ 1 g/t, up to 0.1 g/t. Taking into account the indirect savings of significant costs for the reclamation of spent blocks, it will be profitable to mine blocks with a content of recoverable components up to 0.01 g/t. In view of the foregoing, this technology has a good prospect for implementation in production.

Keywords: uranium mines, associated valuable metals, associated useful components, underground well leaching, productive solution, chlorine-containing solutions.

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Introduction

In industrial practice, in the hydrometallurgical method of gold mining, sodium cyanide solution is used as the main reagent.

Taking into account the global trend of the policy of strengthening the environmental friendliness of production, studies on the replacement of sodium cyanide with less toxic reagents for dissolving gold are relevant. One of the promising reagents for industrial use can be used solutions containing active chlorine, in the form of free halogen or in a compound in the form of hypochlorites. In this work, the studies were carried out on core samples obtained during exploratory drilling of a well at the Semizbay field.

Studies were carried out on the possibility of using the method of underground well leaching to extract associated useful components (gold, iron, aluminum, lanthanum, scandium, and others).

For gold leaching from underground horizons by the method of underground well leaching, the use of cyanides is practically excluded due to high environmental risks. As an alternative, among the leaching agents, chlorine-, iodine-, and bromine-containing reagents can be used, which are more environmentally friendly than cyanide reagents. In the case of using the method of underground well leaching, capital investments can be reduced by dozens of times, so the process of underground leaching can be economically efficient with a higher consumption of chlorine, with a duration of mining

of ore deposits of several months, as well as with the development of poor off-balance ores [1].

In [2], gold leaching was carried out without cyanides, using hydrochloride technology, which makes it possible to ensure the environmental safety of the work. As a solvent for the ores of the Ural deposits, potassium hypochlorite, sodium and chlorine are used.

When gold is leached [3] from crushed ore to a particle size of -12 + 0 mm using the addition of sodium acetate, it improves the kinetics of gold dissolution and increases gold recovery by ~ 4%.

In [4], sodium hypochlorite was used as a leaching agent in order to selectively extract gold and silver from copper concentrate. Hypochlorite leaching of copper concentrate was carried out with preliminary water oxidation under pressure. With direct hypochlorite leaching, the recovery of silver and gold was 45.0% and 42.7%. Using pressurized aqueous oxidation followed by hypochlorite leaching, a selective recovery of 92.5% silver and 90.0% gold from the copper concentrate was achieved.

The dissolution of gold using flat flakes and spheres in a chloride medium was studied in [5] by the method of obtaining chlorine. Based on thermodynamic and mass balance calculations performed in the study at 1.27×10-2 M Cl2 and 0.48 M Cl−, it was found that molecular chlorine (Cl2) is the most predominant species, followed by the Cl− ion at pH below 2.0. From pH 4.0 to 8.0 HClO and from pH 8.0 onwards, ClO− are reported to be more predominant.

In [6], tests on the leaching of a refractory mineral and tailings of a manganese-silver alloy with a low content of silver in manganese-silver minerals were carried out in two stages: extraction of manganese with sodium sulfite and sulfuric acid and leaching silver and gold with sodium hypochlorite and hydrochloric acid, yielding 97% silver recovery, over 80% gold recovery, and 98% manganese recovery. The concentration of hydrochloric acid is 200 g/l, and the concentration of sodium hypochlorite is 50 ml at a concentration of 3%. The tests were carried out at room temperature.

In [7], gold leaching was carried out with a solution of hydrochloric or sulfuric acid with the addition of sodium hypochlorite at a ratio of 0.1 N (HCl) and 0.1 N (NaClO). This solution is made immediately before injection into the ore.

In many sulfide refractory gold ores and concentrates, gold is often found as small inclusions in sulfide minerals, especially in pyrite [8].

In [9] proposed a hydroleaching method consisting of pre-treatment by pressure oxidation and chlorination for refractory gold concentrates with high sulfur content, and investigated the leaching by gold chlorination thermodynamically and experimentally.

It is advisable to maximize the concentration of chloride or reduce the concentration of $[AuCl4]^-$, taking into account the thermodynamics of the gold chlorination leaching reaction. The rational thermodynamic conditions for gold leaching are as follows: pH 3.5–7.8, redox potential over 0.9 V, chloride concentration over 1 mol/l, 10^{-5}–10^{-4} mol/l $AuCl4]^-$ concentration. The redox potential must be maintained above 1.0 V for 2 hours to obtain a high percentage leaching in the chlorination of the gold concentrate pre-treated by pressure oxidation.

If the redox drops below 1.0 V during leaching, the dissolved gold chloride precipitates again and the percentage of leached gold falls. The optimum conditions for chlorination are: pH 4, redox potential above 1.0 V, NaCl concentrations 75 g/l, reaction temperature 40 °C, liquid-solid ratio 3:1, and leaching time 2 hours. The percentage of gold leaching reaches 96.54%.

In studies on the processing of refractory ores and concentrates, the method of exposure to chlorine-containing solutions to extract gold is becoming more and more widely used [[10], [11], [12], [13], [14], [15], [16]].

In [17], the RSM-CCD statistical method was used to determine the optimal conditions for the experiment on leaching a gold ore sample with hypochlorite. SEM/EDX analysis showed that the gold particles are bonded to sulfide and silicate minerals. Moreover, the XRD spectrum showed that the main phases are silicate and muscovite. Elemental analysis by XRF confirmed the presence of Si, Al, Fe and K as major elements in the gold ore sample.

ANOVA analysis was applied to the results of leaching experiments, which showed that the amount of gold dissolved in the form of the gold chloride complex [AuCl4]- was highly dependent on the pH of the solution, followed by the concentration of calcium hypochlorite [Ca(OCl)2] and sodium chloride (NaCl) to facilitate the formation of [AuCl4]- ions. Optimization of leaching experiments at various pH values from 4 to 6,

Ca(OCl)2 molar amounts from 0.5 to 1.5 and NaCl mole amounts from 2.5 to 3.5 showed that the best theoretical conditions for hypochlorite leaching of gold-bearing ores the sample had a lower pH of 4.05 with NaCl and Ca(OCl)2 concentrations of 2.93 M at 1.08 M, respectively.

Verification experiments with optimized conditions showed that gold recovery rates were obtained from 78.99 to 82.46%. Characterization of the obtained residue from the verification experiment by XRD showed that quartz is the main phase. In addition, XRF analysis of the residue showed the presence of a high mass percentage of Ca and Fe compared with the original sample of gold ore.

In [18], the extraction of gold and silver was studied at a sodium chloride concentration in the range of 0.5–3 m, which increased the stability of gold chloride. However, within this range, more than 50% of the silver still remains in the solid AgCl form. Leaching of gold and silver using stirred reactors and static tests confirmed that the maximum recovery of gold and silver is controlled by the concentration of sodium chloride. However, recovery of gold and silver has reached 80% and 50% with reactor leaching.

If chlorinated sea water was used for leaching (approximately 0.5 M NaCl and pH 5.5 and Eh less than 1.00 V compared to the Ag–AgCl reference), both gold and silver could not be fully recovered due to the formation of hydroxide gold and silver chloride. It is best to keep the pH below 4 and keep the solution potential Eh above 1.00 V to keep the gold in solution. Static tests simulating tailings leaching show lower recoveries of both gold and silver reaching 70% and 30% respectively.

In [19], the studied leaching parameters were the S/L ratio, the type of oxidant, i.e. [Cu2+]/[Fe3+] and [Cl−]. The results showed that gold could be dissolved under exceptionally mild conditions when an appropriate adsorption/reduction site (activated carbon) was provided immediately after leaching. It has been found that the impurity metals iron and copper originating from gold ore (Fe 1.6% and Cu 0.05%) are the preferred self-initiating oxidants and 87% gold can be dissolved in pure calcium chloride solution (2.8 M).

The authors of [20] studied the oxidation of sulfide minerals and the leaching of gold from a gold-bearing sulfide concentrate using a chloride–hypochlorite solution. The effect of calcium hypochlorite concentration, sodium chloride concentration and initial pH of the leaching agent on changes in pH and Eh of the slurry was investigated. Then, taking into account the stability range of the gold complex (Eh ~ 1000 mV) and the formation of gaseous chlorine (pH<3.5), the optimal leaching parameters were determined.

Optimum conditions were obtained at 200 g/dm3 calcium hypochlorite, 200 g/dm3 sodium chloride and an initial pH of 11 (with a concentrate of 200 g/dm3, agitation speed of 600 rpm and a temperature of 25°C), at which about 82% gold.

In [21], the effect of various leaching conditions on the extraction of gold from sulfur-containing gold ore using an acidic sodium chlorate solution was studied. The content of gold and sulfur in the ore was 55.7 g/t and 11.67 wt. % respectively. The presence of sulfur has been found to significantly hinder gold recovery.

The optimum desulfurization temperature, desulfurization time, leaching temperature, leaching time, NaClO3 addition rate, HCl concentration, NaCl mass ratio to sample, stirring speed, and liquid leachate volume to solid sample weight ratio were 650°C, 2 h, 40°C, 45 min, 0.25 ml/min, 3M HCl, 0.2, 250 rpm and 10, respectively. The percent gold recovery and percent weight loss of the sample were 97% and 8.8%, respectively, under these optimum conditions. The leaching of gold by this method was quite fast and efficient, and the temperature was low. These characteristics make it possible to apply this method in continuous operation in industry.

Experimental part

Analyzing various literary sources based on the results of experimental studies of the leaching process of gold and other associated useful components, a leaching solution with the following contents of reagents was selected as the most optimal:

NaClO + HCl(c) (hydrochloric acid with sodium hypochlorite), pH=2.8-3.2.

For experimental work on leaching, samples of the core material of the Semizbai deposit were taken from three intervals - ore, supraore, and subore.

The mother liquors obtained as a result of agitation leaching were analyzed by the atomic absorption method to determine the concentrations of various elements (Table 1).

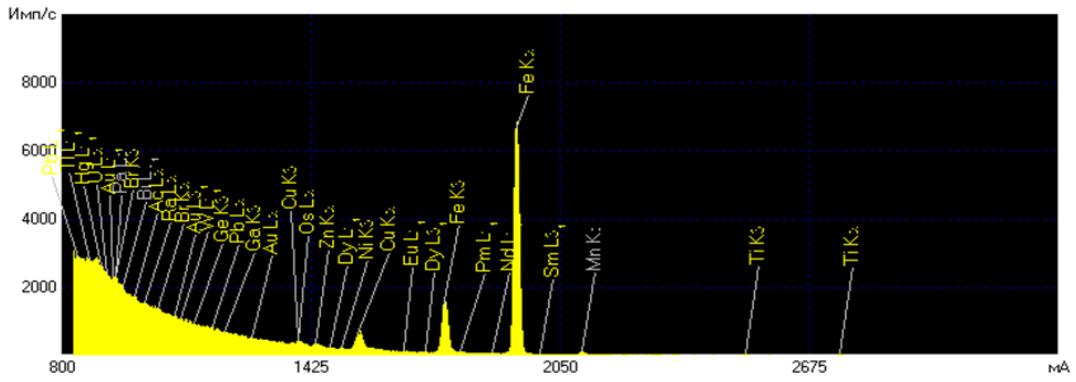
Leaching of gold (Au) from their technological samples. Works to determine the concentration of gold in technological samples were carried out by the laboratory "Technologies for the hydrocarbon and mining and metallurgical sectors and related service industries" of JSC "IMiO" in Almaty. Analyzes in solutions were carried out on an atomic absorption spectrometer. To determine the gold content in the combined samples of the core material, the assay method of analysis was used. The results of the analyzes are presented in Tables 2 and 3.

Table 1 - The concentration of related and useful components in technological solutions

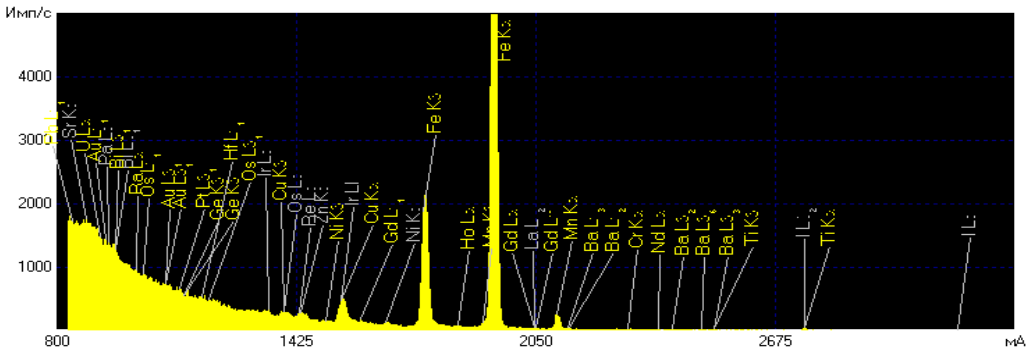
№	№ samples	Fe general, mg/dm³	Al, mg/dm³	La, mg/dm³	Sc, mg/dm³	Leaching solution concentration, %
1	P-4-1(H)	0.63	4.15	0.0053	0.00085	5
2	P-4-2(H)	22.96	15.09	0.0010	0.00027	
3	P-4-3(H)	0.56	10.28	0.014	0.0041	
4	P-5-1(H)	4.08	15.91	0.013	0.0048	8
5	P-5-2(H)	99.96	89.02	0.0043	0.056	
6	P-5-3(H)	2.10	28.13	0.087	0.0023	
7	P-6-1(H)	0.48	0.37	0.0088	0.00062	16
8	P-6-2(H)	136.03	14.43	0.26	0.0053	
9	P-6-3(H)	34.24	12.90	0.023	0.0087	

Table 2- Results of chemical analysis of pooled core samples using the assay method for gold

№	№ samples	Au g/l	Interval
1	Well 1	0.20	Suprarutal interval
2	Well 2	0.34	Ore interval
3	Well 3	0.34	Under-ore interval



Picture 1– Results of X-ray phase analysis of the core sample

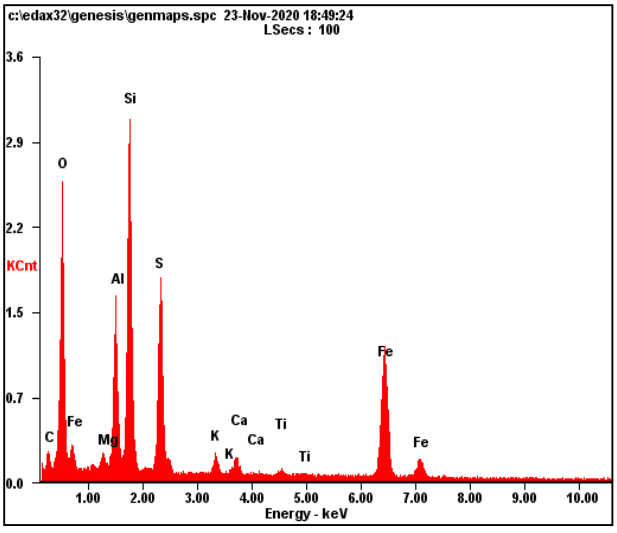


Picture 2 – Results of X-ray phase analysis of the core sample

The method of X-ray phase analysis was used to determine the chemical composition of the samples. The data of X-ray phase analysis are presented in Figures 1 and 2.

To study the local elemental composition of core samples, chemical analysis was carried out using a scanning electron microscope. Elemental analysis of core samples was carried out by energy-dispersive analysis on a Fei Quanta 3d 200i scanning electron microscope.

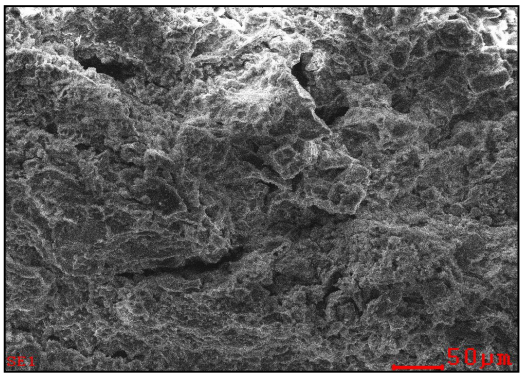
The resolution of the microscope is up to 1 nm at a voltage of 30 kV, the accelerating voltage is from 0.5 to 30 kV, the magnification is from x 10 to x 1,000,000, the beam current is up to 200 nA, the elemental analysis was carried out in vacuum for a number of elements C, O , Na, Mg, Al, Si, S, Cl, U, Ca, K and Fe. The characteristic X-ray spectrum for a number of elements and the general view of the micrograph of the core sample under study with the detected area are shown in Figure 3.



a) sample spectrum (first point)

Element	Wt%	At%
CK	17.44	27.23
OK	42.91	50.32
MgK	0.78	0.60
AlK	6.43	4.47
SiK	13.59	9.08
SK	6.95	4.07
KK	0.78	0.37
CaK	0.76	0.36
TiK	0.27	0.11
FeK	10.07	3.38
Matrix	Correction	ZAF

b) Elemental analysis of the obtained spectra in weight percent Wt %



c) micrograph 50 µm

Figure 3 - Characteristic X-ray spectrum

Mineralogical analysis of core samples for gold. To study the material composition of the core sample of the ore interval, a polished artificial briquette (size 0.25 mm) was made.

The sample material was studied under a microscope of the LEICA DM 2500 P brand in reflected light in order to diagnose and describe ore

minerals, in immersion preparations for the diagnosis of rock-forming minerals.

The result of the mineralogical analysis is shown in Figure 4.

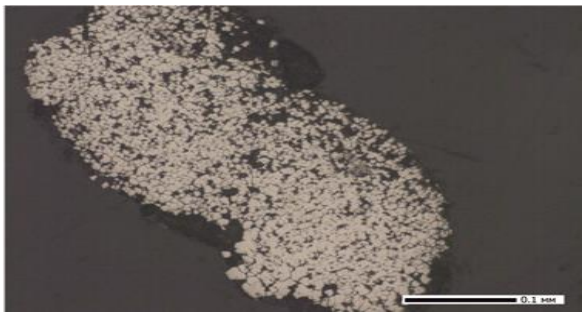


Figure 4 - Accumulation of globular pyrite.
Sample 1 (ore interval)
Polished briquette, led away. x 200

Table 3 - Comparison of the degree of recovery of recoverability of gold from ore samples at various concentrations

№	№ samples	Au mg/dm³	Leaching solution concentration, %
1	Well 1	0.033	5 (Surface interval)
2	Well 2	0.0171	8 (ore interval)
3	Well 3	0.057	16 (Under-ore interval)

More clearly, the degree of extraction of Au is shown in Figure 5.

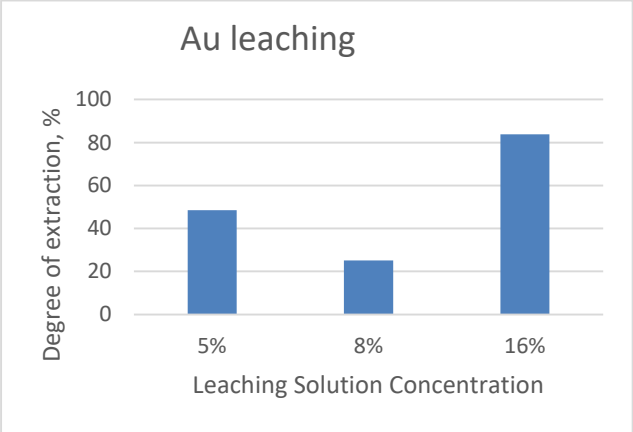


Figure 5 - The degree of extraction of Au during agitation leaching with NaClO solution with the introduction of oxidizing agents at various concentrations

The discussion of the results

Analyzing the output of associated and useful components in technological solutions (Table 1), it can be noted that the highest output of components in the solution was achieved when core material was leached from the uranium-bearing ore interval. Thus, the concentration of iron was 136.03 mg/dm^3, the concentration of aluminum - 89.02 mg/dm^3, scandium - 0.056 mg/dm^3, lanthanum - 0.26 mg/dm^3. X-ray phase analysis of the core sample (Figure 1 and Figure 2) in the range from 800 to 2050 nm in the decomposition spectrum shows fluctuations in the peaks of uranium (U), iron (Fe), copper (Cu), manganese (Mn), zinc (Zn). In addition to the above, the decomposition spectrum contains fluctuations of rare earth elements, such as osmium (Os), rhenium (Re), neodymium (Nd), actinium (Ac), scandium (Sc), cesium (Ce), as well as bismuth (Bi), etc. Note that the decomposition spectra show spectral lines of mercury (Hg) and unexpressed fluctuations of gold-Au.

The presence of mercury is interpreted by the content of mercury in sulfide minerals that are genetically related in chemical nature, which in turn are formed during the formation of uranium ores. During the amalgamation reaction, mercury forms compounds with gold. For this reason, the detection of lithophaneous reflections of gold and clear spectra of mercury indicate the possible presence of gold.

The characteristic X-ray spectrum (Figure 3) shows that the elements Si, O, S, Fe and Al are the main components of the sample, in which these elements have the following percentages: silicon (Si) - 13.59%, oxygen (O) - 42.91%, sulfur (S) - 6.95%, Iron (Fe) - 10.07% and aluminum (Al) - 6.43%.

Mineralogical analysis of a briquette of a core sample of the ore interval (Figure 4) shows the presence of pyrite, sphalerite and technogenic material in a small amount, resembling gold in color. Pyrite is found in the form of fine dissemination in non-metallic material and several free grains up to 0.3 mm in size. After testing the effect of HNO_3 acid on a grain resembling gold, it dissolved, thus its technogenic nature was established.

The result obtained from the data in Figure 5 characterizes a NaClO + HCl + H_2O solution with a concentration of 16% as more effective for Au than a solution with a concentration of 8% and 5%. The degree of extraction of Au for solutions with 16% leaching reagents reached 83.82%.

Conclusions

Based on the results of laboratory experiments on agitation leaching on samples of core material of ores from the Semizbay deposit of the supra-ore, ore and under-ore interval, it can be concluded that for the extraction of associated useful components by the ISR method from depleted uranium wells, a chlorine-containing solution of the composition $NaClO + HCl_{(c)} + H_2O$ (hydrochloric acid with sodium hypochlorite) at pH=2.8-3.2.

The highest extraction of gold (83.82%) into solution was observed during leaching of material from the sub-ore interval, while for associated useful components the highest degree of extraction was observed during leaching of material from the ore interval: iron - 136.03 mg/dm3, aluminum - 89.02 mg/dm3, scandium - 0.056 mg/dm3, lanthanum - 0.26 mg/dm3.

At the moment, the sodium hypochlorite leaching method together with hydrochloric acid is not used for gold mining on an industrial scale. In this work, the experiments were carried out directly on the ore material, which makes it possible to test this method in industry and the result was achieved, which makes it possible to apply this method on an industrial scale.

Thus, the use of the ISL method on spent ore uranium blocks for the extraction of valuable components is a breakthrough direction in research in the mining industry.

Conflicts of interest. On behalf of all authors, the author declares that there is no conflict of interest.

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Уран кен орындарының пайдаланылған блоктарын өңдеу кезінде құрамында хлор бар агенттерді пайдалану

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Қабылданды: *28 қараша 2022*

ТҮЙІНДЕМЕ

Жұмыс ілеспе пайдалы компоненттерді алу мақсатында жерасты ұңғымалық сілтісіздендіру әдісімен уран кен орындарының пайдаланылған блоктарын химиялық ерітінділермен қосымша өңдеу есебінен табиғи ураны өндіру және қайта өңдеу бойынша жұмыс істеп тұрған кеніштерді әртараптандыруға бағытталған. Бұл технологияның ерекшелігі (жүргізіліп жатқан жұмыс) қолданыстағы уран кеніштерінде ілеспе пайдалы компоненттерді өндіру үшін қолданыстағы өндірістік инфрақұрылымды өндірістік инфрақұрылым мен тау-кен жұмыстарына елеулі күрделі салымдарсыз пайдалану болып табылады. Жерасты ұңғымаларын шаймалау технологиясы уран кен орындарында ондаған жылдар бойы сенімді түрде пысықталды. Уран өндіру технологиясының және бірқатар ілеспе пайдалы компоненттердің түбегейлі ұқсастығы - жерасты ұңғымалық сілтісіздендіру әдісімен пайдалы компоненттерді өндіру үшін уран кен орындарының пайдаланылған кен өрістерін пайдалануға мүмкіндік береді. Дайын технологиялық инфрақұрылымды (ұңғымалар, құбырлар желісі, сорғы жабдықтары, басқару блоктары және т. б.) пайдалану инфрақұрылымдық шығындарды үнемдеу есебінен өндірілетін компоненттері ≤ 1 г/т-дан 0,1 г/т-ға дейінгі кенді блоктарды өңдеу кезінде рентабельділік алуға мүмкіндік береді. Пайдаланылған блоктарды қалпына келтіруге кететін айтарлықтай шығындарды жанама үнемдеуді ескере отырып, алынатын компоненттердің құрамы 0,01 г/Т дейін болатын блоктар тиімді жұмыс істейді, жоғарыда айтылғандарды ескере отырып, бұл технология өндіріске енгізу үшін жақсы перспективаға ие.

Түйін сөздер: уран кеніштері, ілеспе бағалы металдар, ілеспе пайдалы компоненттер, жер асты ұңғылап шаймалау, өнімді ерітінді, хлорлы ерітінділер.

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Использование хлорсодержащих агентов при обработке отработанных блоков урановых месторождений

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АННОТАЦИЯ

Работа нацелена на диверсификацию действующих рудников по добыче и переработки природного урана за счет дополнительной обработки отработанных блоков урановых месторождений химическими растворами методом подземного скважинного выщелачивания (ПСВ) с целью извлечения попутных полезных компонентов. Особенность (проводимой работы) этой технологии состоит в использовании существующей производственной инфраструктуры для добычи попутных полезных компонентов на действующих урановых рудниках, без значительных капитальных вложений в производственную инфраструктуру и горные работы. Технология подземного скважинного выщелачивания за десятки лет надежно отработана на урановых месторождениях. Принципиальная схожесть технологии добычи урана и ряда попутных полезных компонентов (ППК) - методом ПСВ, позволяет использовать отработанные рудные поля урановых месторождений для добычи ППК. Использование уже готовой технологической инфраструктуры (скважины, сеть трубопроводов, насосное оборудование, управляющие блоки и т.д.) позволяет за счет экономии на инфраструктурных затратах, получить рентабельность при отработке рудоносных блоков с содержанием извлекаемых компонентов от от ≤ 1 г/т, вплоть до 0,1 г/т. При учете косвенной экономии значительных затрат на рекультивацию отработанных блоков, то рентабельно будет отрабатывать блоки с содержанием извлекаемых компонентов вплоть до 0,01 г/т. С учетом вышесказанного данная технология имеет хорошую песпективу для внедрения в производство.

Ключевые слова: урановые рудники, попутные ценные металлы, попутные полезные компоненты, подземное скважинное выщелачивание, хлорсодержащие растворы.

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Metallurgy

Study of silicon production process in ore-smelting furnace and optimization of technological process

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ABSTRACT

This article presents the results of production experiments to optimize the modes of silicon smelting in an industrial arc furnace. The main factors of the melting process are the size of the fractions of the charge components and the temperature regime of heating. The rate of charge heating in the reaction zone in the temperature range from 950 to 14100C has a special effect on productivity. In this temperature range, the formation of refractory silicon carbide on pieces of quartzite was established, which causes a drop in the magnitude of the electric current in the reaction zone and its freezing. The gornisage, which is formed, displaces the electrodes into the zone of greater charge electrical conductivity - up, which leads to an increase in silicon monoxide emissions through the reduced charge layer. Correction of such a process requires an increase in the temperatures in the reaction zone and the duration of the melt. A method has been developed for calculating the size of quartzite fractions, depending on the power of the furnace and the size of the reaction zone. An example of a simplified calculation is proposed.

Keywords: Silicon, quartzite, coke, carbide, monoxide, arc furnace

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Introduction

The practical value of such optimization is lost due to the inadequacy of the recommended modes to practical results [1]. This publication analyzes this conclusion, undeserved by thermodynamics, and provides an example of industrial studies explaining such a delusion. Industrial experimental studies were carried out on an electric arc silicon furnace with a power of 1.8 kVA (2 kW). The reasons for the furnace freezing were determined and a solution to the problem is proposed by optimizing the fractional composition of the charge components and adjusting the modes of conducting the melting process.

Experimental part

The study of high-temperature reduction processes is a technically and economically difficult task. For this reason, researchers in the field of

carbothermal reduction of silicon from quartzites take the results of thermodynamic modeling as a basis. Existing computer programs for thermodynamic analysis do not allow taking into account the reality of changing conditions in the system. And, experiments, apparently, are also carried out “virtually”. This opinion was formed since in practice, especially when starting new silicon productions and when changing raw materials, such recommendations turn out to be useless for production technologists. The authors were convinced of this when launched the production of silicon from quartzites of South Kazakhstan in the metallurgical shop of StekloK LLP.

In the production of metallurgical silicon, problems arose, leading to the “freezing” of the reaction zone in the furnace hearth, the displacement of electrodes by the furnace encrustation consisting of a mixture of silicon carbides, inclusions of polycrystalline silicon, oxides, and inclusions of coke (Figure 1). Such an accident in production leads to an increase in energy costs for process corrections and, in the worst case, to a forced shutdown of the melting process. According to the terminology of metallurgists, this is the formation of a “bear” in the furnace [bear – metal frozen in the volume of the furnace or ladle as a result of a violation of the normal technological process course or an emergency release of metal from the furnace or pouring ladle. [http://metaltrade.ru/ abc/a.htm].

The photo in Figure 1 shows a piece taken from a “frozen” silicon furnace – the lower part of the piece, lighter, is a solidified mixture of silicon crystallites, silicon carbides and oxides. And the upper part is a partially melted charge – a mixture of coke, quartzite and reduction products: carbides, silicon monoxide and silicon crystallites.

Specialists when developing technological modes of the carbothermal process of silicon reduction from quartzites use the results of thermodynamic analysis, including thermodynamic modeling [[1], [2], [3], [4], [13], [19]]. It should be understood that thermodynamic studies allow to assess the possibility of implementation, however in practice it is very problematic to obtain the expected result according to the thermodynamic analysis without adjustment, taking into account specific processes in the furnace.

The reasons for this problem lie in the fact that in a real process there are changes in the component composition in the system, temperature changes in the contact zones of the system components, which create kinetic obstacles to mass transfer processes in the reaction zone of the furnace. For example, in a system of charge consisting of pieces of quartzite, coke, coal, wood chips [5], intermediate chemical compounds are formed at the boundary between the reagents in the reaction zone of the furnace, changing the component composition of the system, therefore, the thermodynamic conditions for chemical reactions change. Taking into account the changing conditions of the system’s thermodynamic state is a very difficult and not yet solved problem in modeling heterophase multicomponent systems.

Figure 1 - Photo of the frozen part of the charge and the furnace encrustation in an industrial arc furnace: crystallized mixture of silicon carbide with inclusions of silicon and quartzite (silicon oxides) reduced by 20 times M1:20

In this article, the authors consider the process that occurs in an industrial silicon arc furnace, studied by industrial experiments.

The efficiency of silicon reduction melting in an electric arc ore-smelting furnacedepends on many interdependent factors: the fractional size of the charge components, the homogeneity of the charge components’ distribution, the temperature in the reaction zone, the duration of reaching the temperature condition for the main process – theprocess of silicon formation. Let us omit the elemental and phase composition of the charge components from discussions, since many scientific works are devoted to this [[6], [7], [8], [12], [13], [14], [17]]. This publication discusses the results of studying technological factors that affect the

effectiveness of the metallurgical process for obtaining metallurgical silicon from quartzites. It is known [[9], [10], [13], [18], [19], [20]] that the production of silicon by the carbothermal method is carried out according to the main four reduction process stages. In the process of moving the charge from the top zone to the electric arc furnace hearth, the charge components are heated by gases sublimated from the reaction zone, in which the temperature reaches 2000-3000°C. The industrial experiments were carried out on an industrial three-electrode furnace with electrodes buried in the charge by 1.2-1.5 m:

Stage 1. Heating the mixture with hot gas – sublimates from the furnace reaction zone, consisting of carbon monoxide (CO), carbon dioxide (CO_2) and silicon monoxide (SiO).

It is known [11] that a chemical reaction occurs at the boundary of the solid phase with the gaseous phase:

$$CO + CO = CO_2 + C \quad (1)$$

The resulting atomic carbon has an increased activity, which was confirmed in practice and used for a long time in the technology of carburizing parts from structural steels in mechanical engineering. This happens at a temperature of 940°C [9].

The formation of an active reducing agent, atomic carbon, provides the conditions for the reactions:

$$SiO_2 + C = SiO + CO \quad (2)$$

$$SiO + C = Si + CO \quad (3)$$

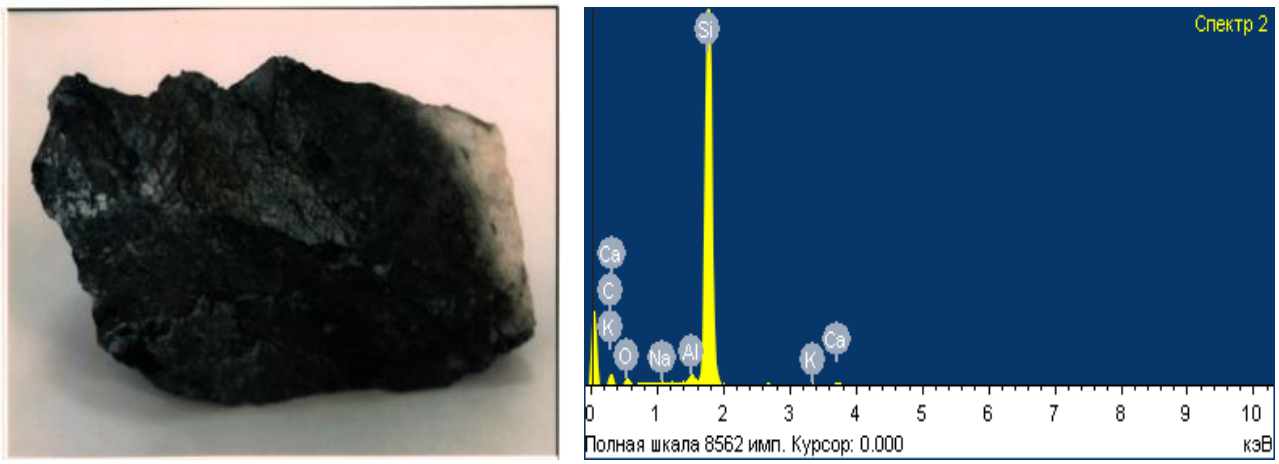
The products of reactions (2) and (3): SiO and CO are gaseous, and Si is condensed under these temperature conditions, SiO and Si are adsorbed on the charge components' solid phasesurface: quartzite, coal and coke pieces (Figure 2) and the gaseous product, carbon monoxide gas (CO), reacts (1), and unreacted carbon monoxide and silicon monoxide (SiO) above the top surface interact with atmospheric oxygen to form carbon dioxide CO_2 and SiO_2 – carbonwhite.

Stage 2. Silicon formed as a result of reaction (3) is adsorbed by the charge components' surface and forms a film in the temperature range 950°C – 1410°C (up to the melting temperature of silicon – 1410°C).

In this temperature range, atomic carbon and a silicon film are converted into a silicon carbide shell according to reaction (4) Figure2:

$$Si + C = SiC \quad (4)$$

The process of silicon reductionfrom quartzite at this stage is determined by the carbon atoms' diffusion rate through the shell of silicon and silicon carbide, let's call it "shell". The shell is a sufficiently strong and refractory screen between the reagents (the melting point of silicon carbide is above 2730°C [10]). With the melting of the quartzite pieces inside the shell (the melting temperature of which is above 1750°C), a change in the density of quartzite occurs, as a result of which the shell is destroyed – itcracks if it is not strong enough, which depends mainly on the shell thickness.



a) b)

Figure 2 - Photo of the quartzite piece extracted from the upper zone of the furnace shaft at a depth of 0.5-0.6 m from the top:
a) a sample of quartzite coated with a thin layer of silicon and soot (carbon),
b) spectrum from light areas with a blue tint (the most intense peak of the silicon spectrum)

With the melting of quartzite, the aggregate composition of the components in the system changes. The system, which consisted of solid and gaseous reagents, is transformed into a system: liquid – gas. Such changes in the system conditions provide an increase in the rate of chemical processes and implementation of chemical reactions (2), (3) and reaction (5):

$$2SiC + SiO_2 = 3Si + 2CO \quad (5)$$

The resulting liquid silicon flows down to the furnace bottom, where it accumulates and after a certain time the silicon melt is poured from the furnace into the ladle.

In addition to the shell formation on the quartzite pieces, reactions (3) and (4) also occur on the reducing agent surface: pieces of coke and coal, since SiO at temperatures exceeding the condensation temperature of gaseous silicon monoxide is adsorbed by coke and coal.

The result of these processes is the formation on the charge pieces' surface of a film of silicon and then silicon carbide, which have a lower electrical conductivity, this is accompanied by a decrease in the electrical conductivity of the charge. The consequence of such a change in the charge composition and the intermediate products' properties is a decrease in the electric current value in the reaction zone of the main heat source, the result of this is a decrease in temperature in the reaction zone. In support of what – Figure (a)with the quartzite piece coated with silicon carbide. A very interesting fact was the discovery of the formation of carborundum in the form of needle-shaped crystals similar to twigs – dendritesin the frozen reaction zone of the furnace (Figure 2b). This fact indicates the crystallization of silicon carbides from the gas phase – crystallizationof carborundum dendrites occurs on the surface of the formed SiC layer as a result of reactions (1), (2), (3) and (4).

Under these temperature conditions, CO, SiO are gas phases, and C, Si, SiC are condensed phases.

The silicon reduction process abnormalityin the furnace can be excluded if the technological parameters are strictly observed: the charge fractions' size, the current density in the reaction zone and the temperature, which should be higher than the melting point of quartzite – above1750°C (Since meltedquartzite has a high dynamic viscosity, the heating temperature should be significantly higher than the melting point of quartzite). Therefore, the electric power in the furnace hearth should provide the possibility of increasing the electric voltage to maintain the current density in the reaction zone at the required level, which is determined by the quartzite fractions' sizein the charge.

Figure 3 - Quartzite coated with silicon carbide during silicon carbothermy in an electric arc furnace

As a result of these chemical processes in the reaction zone of the furnace, the "shell" becomes sufficiently strong, the active electrical resistance of the charge increases, the electric current between the electrodes decreases, the furnace encrustation is formed on the furnace hearth, the furnace electrodes are forced upwards into the more electrically conductive part of the charge, and on the hearth there is a freezing of "slag-carborundum" – amixture of quartzite, silicon and carborundum (Figure 4).

Figure 4 - Photo of the furnace encrustation pieces – Slagcarborundum from the "frozen" furnace

The algorithm for solving this complex multi-factorial problem is as follows:

1. Taking into account the peculiarity of the carbothermal process in a silicon ore-smelting furnace, where the reaction zone is under a layer of charge with electrodes buried in the charge, its sizes and shape are determined by the interelectrode space with the temperature range of more than 1750° C – thescheme is shown in Figure 5.

The production process can be with a continuous discharge of the silicon melt, however in practice it took root periodically, that is, at the beginning, the silicon melt is accumulated in the furnace, then the melt is released (discharged) into a ladle for refining by blowing with oxygen and pouring into molds. Therefore, the process is carried out in cycles. The temperature mode in the furnace hearth is shown in Figure 6.

After discharging the silicon melt from the furnace, the charge is deposited in the hearth. The temperature of the charge entering the reaction zone of the furnace should be 950...1000°C. The authors recommend pushing the charge prepared for loading into the furnace for the next cycle from the top zone sides to the top center, while maintaining the components' distribution homogeneity in the charge. The charge enters the hearth in each subsequent cycle with a temperature not lower than 950...1000°C.

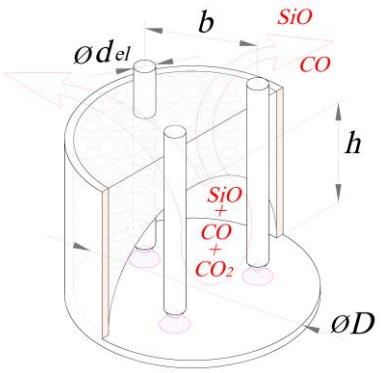


Figure 5 - Scheme of the reaction zone in the carbothermal furnace

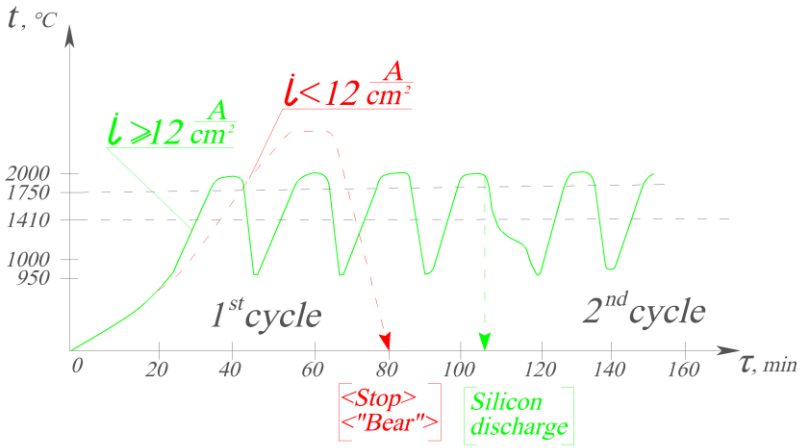


Figure 6 - Temperature mode in the reaction zone of the silicon carbothermal furnace:
Red line – the heatingmode at the current density
less than 12A/cm^2,
leading to the furnace freezing;
Blue line – the heating mode at the current density greater than 12A/cm^2
(for experiments in the studied production conditions –
the furnace with the power of 2 kW or 1.8 kVA)

In the furnace hearth, the most important and responsible process for melters begins: chemical reactions (2, 3 and 4), it is necessary to prevent the formation of a critical layer on the quartzite pieces – "shells" of silicon carbide.

Important technological parameters in this process are the quartzite pieces' sizesin the charge and the duration of their melting in the reaction zone. The duration of heating until the melting of the quartzite pieces provides the conditions for the formation of the "shell"from SiC. The intensity of heating the quartzite pieces in the reaction zone depends on the current density in the reaction zone.Coarse fractions take longer to heat up, therefore, the duration of heating to the melting temperature of quartzite increases, creating conditions for the "shell" formation.

The charge's fine fraction has a low gas permeability, which contributes to the formation of wormholes (a powerful torch from the reaction zone, as a rule, along the electrodesurfaces). This leads to thermal energy losses in the reaction zone and the release of CO and SiO. Therefore, from this point of view, the determining indicator of the charge fractions' sizes is the provision of gas permeability. The larger the quartzite fraction, the higher the charge's gas permeability, and on the other hand, with large quartzite fractions, it is more likely to get "shells" from silicon carbides on the quartzite pieces,which increases the charge's electrical resistance, and reduces the electric current value in the hearth, therefore, reduces the temperature in the reaction zone – fromwhich the furnace "freezing" begins, that is, the "bear" formation.

Figure 7 - Photoof the carbothermal furnace, used in the experiments

In the industrial experiments in the furnace with the power of 2 MW (Fig. 7), the authors experimentally established: for quartzite – piecesin cross section should be from 50 mm to 110 mm, for coke – from20 to 50 mm. The task is to calculate the heating time of the quartzite pieces in the reaction zone – abovethe melting temperature, taking into account the quartzite melt viscosity, the temperature should be above 1750°C.

Results and Discussion

Simplified calculation of the heating time of the quartzite pieces in the reaction zone before the melting:

Step 1. Determining the reaction zone size (see Figure 3). The volume under the dome is calculated using formula 1.1 [6]:

$$V_{rz} = \pi h^2(D/2 - h/3) \qquad (1.1)$$

where: V_{rz} – the volume under the reaction zone dome; π – geometric constant ~ 3.14 – the ratio of the circumference to the diameter; D – the diameter of the working furnace hearth space, m; h – the reaction zone dome height, m.

For approximate calculations, it is possible to take the reaction zone dome height equal to D/2. In the experiments, according to the results of measurements of "frozen" working areas, h = 0.5-0.7D. For approximate calculations of the charge mass in the reaction zone, it is necessary to take into account that part of this volume is occupied by the graphite electrodes of the furnace.

Example: In the ore-smelting furnace with the power of 2 MW, D = 1.75 m, h = 1.05 m, according to the results of measurements of the melted and "frozen" zones in the furnace, the reaction zone volume was determined to be 1.8 – 2.02 m^3.

Step 2. Based on the bulk density of the charge and its composition, the authors determine the charge mass in the reaction zone – $G_{charge\ rz}$, which occupies the reaction zone volume in the furnace hearth. With an average bulk density g_{bulk} of the charge in the industrial experiments equal to 0.85–0.90 t/m^3:

$$G_{chargerz} = g_{bulk}V_{rz}$$

The reaction zone volume occupied by the electrodes is approximately 30%. Therefore, the charge volume in the reaction zone will be $0.7V_{rz}$.

In the example under consideration: $G_{charge\ rz} = g_{bulk}0.7V_{rz} = 2.02 \cdot 0.7 \cdot 0.85 = 1.19$ t.

Step 3. Knowing the charge mass and composition in the reaction zone, the authors perform an approximate calculation of the energy costs for heating quartzite in the charge and the entire charge from 950°C to 1750°C.

The energy required to heat the charge is very difficult to determine according to the laws of heat transfer [[7], [8], [9]], in this case, there is no need for an accurate calculation, therefore, the calculation is limited to determining the difference between the heat content of the charge components at 1750°C and 950°C, taking into account the furnace efficiency.

Taking into account the fact that intensive mass transfer of the process of silicon reduction from quartzite occurs after the melting of quartzite, the authors assume that the quartzite mass changes insignificantly. Then the amount of thermal energy required to achieve the melting temperature of quartzite in the charge:

$$\Delta Q_{charge\ rz} = G_{quartzite\ rz} \cdot (C_{quartzite\ 1750} - C_{quartzite\ 950}) + G_{coke\ rz} \cdot (C_{coke\ 1750} - C_{coke\ 950}) + G_{coal\ rz} \cdot (C_{coal\ 1750} - C_{coal\ 950})$$

Where: $\Delta Q_{charge\ rz}$ – change in the heat content of the charge in the reaction zone during heating from 950 to 1750°C; $Q_{charge\ rz\ 1750}$, $Q_{coke\ 1750}$, $Q_{coal\ 1750}$ – the heat content of quartzite, coke and coal in the reaction zone at 1750°C; $Q_{charge\ rz\ 950}$, $Q_{quartzite\ 950}$, $Q_{coke\ 950}$, $Q_{coal\ 950}$ – the heat content of quartzite, coke and coal in the reaction zone at 950°C; $C_{quartzite\ 1750}$, $C_{quartzite950}$, $C_{coke\ 1750}$, $C_{coke\ 950}$, $C_{coal\ 1750}$, $C_{coal\ 950}$ – the heat capacity coefficients of quartzite, coke, coal, respectively, at 1750 and 950°C.

Step 4. The authors determine the time required to heat the charge in the reaction zone to 1750°C. Knowing the nominal power of the furnace and the efficiency, it is possible to determine the useful power of the furnace, that is, the power that is spent on heating the charge in the reaction zone. The efficiency according to the furnace passport is η=0.60, therefore

$$P_{useful} = P_{nominal} \cdot \eta = P_{nominal} 0.60$$

The heating time (approximate) of the charge in the reaction zone (τ) is calculated as follows:

$$\tau = \Delta Q_{chargerz} / P_{useful,c}$$

Step 5. The authors determine the duration by the time required for the formation of the "shell", which reduces the electrical conductivity of the charge, which is easy to control by the readings of the ammeters on the control panel. An increase in the active electrical resistance of the charge reduces the current density in the reaction zone, and the result of such a change in the process is "freezing" in the reaction zone.

Figure 6 shows the scheme of the temperature mode in the reaction zone of the carbothermal furnace. The mode was selected experimentally.

For the practical use of the industrial experiment results, the authors present a simplified calculation of the optimal size of the quartzite pieces in the charge according to the maximum allowable duration of their heating until the melting in the furnace hearth zone from 950°C. During this duration – the time of heating the quartzite piece, its heat content should increase from $Q_{950°C}$ to $Q_{1750°C}$:

$$Q_{950°C} = C_{1123K} \times M_{quartzite\ piece} \times T_{start}$$
$$Q_{1750°C} = C_{2023K} \times M_{quartzite\ piece} \times T_{end}$$

Where: C_{1123K}, C_{2023K} – the heat capacity of quartzite at 950°C and 1750°C; $M_{quartzite\ piece}$ – the mass of the quartzite piece in the charge, to simplify calculations, let's take the form of the quartzite pieces as spherical, $M_{quartzite\ piece} = V_{quartzite} \times \rho_{SiO2}$, where: $V_{quartzite}$ – the volume of the quartzite piece in the charge, for a sphere – $2\pi R^3/3$, ρ_{SiO2} – the specific gravity of quartzite.

The difference between the heat content of the quartzite piece from $Q_{1750°C}$ to $Q_{950°C}$ is the amount of energy △Q that must be transferred to the quartzite piece in the furnace hearth for a limited time – this is the time during which the "shell" of critical thickness does not have time to form.

Observations of the melting process in the ore-smelting furnace allow to establish the duration of the charge melting from the start of filling the reaction zone with the charge to its melting with different current densities at the furnace electrodes. The current density (i) in the reaction zone of the furnace is controlled by the operator, by changing the voltage on the furnace electrodes and by changing the electric current value between the electrodes by their immersing into the reaction zone. Taking into account that the nominal power of the furnace is as follows:

$$P_{nominal} = I \cdot U, W$$

The electrical conductivity in the reaction zone also depends on the component composition of the charge, that is, the proportion of electrically conductive components: coke, coal, and the degree of gas ionization ($SiO + CO + CO_2$) in the reaction zone. The current density on the electrodes has a limiting value and depends on the electrode material. It is known that graphite electrodes provide the highest current density, for example, for electrodes with a diameter of 250 mm, the maximum allowable current density is 21 A/cm^2.

$$I_{nominal} = P_{nominal} / U \cdot S_{el} = I \cdot U / U \cdot S_{el} = I / S_{el}$$

Where: i – the electric current density in the reaction zone, A/cm^2; U – the electrical voltage on the electrodes, V; S_{el}– the electrode surface area located in the reaction zone of the furnace, cm^2, I – the electric current value in the reaction zone, A.

The heating time of the quartzite piece in the reaction zone before the melting, that is, up to 1750°C, can be approximately calculated by dividing the amount of energy spent on heating the quartzite piece to 1750°C by the thermal energy perceived by the quartzite piece surface:

$$T_{heating\ the\ quartzite\ piece} = \triangle Q_{quartzite\ piece} / i \cdot U \cdot S_{quartzite\ piece}$$

Where: $\triangle Q_{quartzite\ piece}$ - the amount of energy that must be transferred to the quartzite piece for its melting in the reaction zone – spent on heating the quartzite piece in the reaction zone from 950°C to 1750°C; $S_{quartzite\ piece}$ – the quartzite piecesurface area. Let's take a sphere for the shape of the quartzite pieces, then $S_{quartzite\ piece} = \pi d^2$, cm^2.

The quartzite piece heating rate in the furnace hearth is carried out by radiant energy and thermal energy transfer by thermal conductivity from the quartzite piece surface to its center. Therefore, to ensure heating of the quartzite piece in the reaction zone of the furnace, the quartzite's thermal conductivity plays a decisive role in the heating rate. This is clearly seen from the calculation results (Table 1)

$$\tau_{heating\ the\ quartzite\ piece} = \triangle Q_{quartzite\ piece} / i \cdot U \cdot \pi d^2_{quartzite\ piece}$$

Let's transform this formula to calculate the cross-section size of the quartzite pieces in the charge:

$$d_{quartzite\ piece} = (\triangle Q_{quartzite\ piece} / i \cdot U \pi \tau_{heating\ the\ quartzite\ piece})^{-2};$$

$$i_{quartzite\ piece} = \triangle Q_{quartzite\ piece} / \tau_{heating\ the\ quartzite\ piece} \cdot U \cdot \pi d^2_{quartzite\ piece},$$

$$i_{nominal} = P_{nominal} / U \cdot S_{el}$$

Let's set the cross-section sizes of the quartzite pieces in the charge and calculate the corresponding values of the electric current density in the reaction zone of the carbothermal furnace.

Analysis of the results of these calculations shows that it is energetically more profitable to process a fine fraction than a coarse fraction of quartzite, this is without taking into account heat losses to the environment and losses on the duration of the process due to the low thermal conductivity of quartzite, due to which the duration of melting of large quartzite fractions increases to 1750°C – temperature of liquid-phase reduction of silicon.

Table 1- Calculation results of energy costs for melting the quartzite piece in the charge of the arc ore-smelting furnace

$d_{quartzitepiece}$, cm	5	10	20	30	40
$R_{quartzitepiece}$, cm	2.5	5	10	15	20
$\tau_{heating\ the\ quartzite\ piece}$, C	0.36	72	1.433	433204.9/3.5 min	274.3/4.6 min
$i_{quartzitepiece}$,A/cm^2	1.22	2.44	4.88	7.32	9.76
$V_{quartzite\ piece}$	32.656	261.7	2093	7053.75	16746
$M_{quartzite\ piece} = V_{quartzite} \times \rho_{SiO2} 10^{-3}$, kg	84.9	680.42	5441.8	18339.75	43539.6
$\triangle Q_{quartzitepiece}$, J	59.43	476300	3780926	12833.8	30539.6

Example of approximate calculation of the technological parameters of a carbothermal process in a 2 MW furnace

Calculation of sizes of the reaction zone

Knowing the geometrical parameters of the furnace: the electrodes' diameters d_{el}, the electrodes' decay diameter D_{decay}, the furnace bottom diameter (the furnace top diameter) $D_{furnace}$, it is possible to calculate the volume of the reaction zone. But for this it is necessary to determine the area above the top (above the furnace hearth) with a temperature – abovethe quartzite's melting temperature, we chose a zone with frozen pieces of charge with melted traces. As a result, a dome with a height of 650-700 mm was determined (see Figure 4).

Approximately the volume of such a dome can be calculated by the formula:

$$V_{rz} = \pi h^2 (D_{furnace\ hearth}/2 - h/3) \quad [6]$$

Where: V_{rz} – the dome volume – the reaction zone of the carbothermal furnace;

π- ≈ 3.1415926535 ≈ 3.14; h – the dome height, m; $D_{furnace\ hearth}$ – the furnace hearth diameter, m.

For the furnace in our experiments:

$$V_{rz} = 3.14 \cdot 0.7^2 \cdot (1.0/2 - 0.7/3) = 0.41 m^3$$

The charge volume in the reaction zone is less by the volume occupied by the furnace electrodes. Therefore, the volume occupied by the charge in the reaction zone will be: $V_{chrz} = V_{rz} - V_{erz}$; $V_{erz} = 3\pi d_{el}^2 \cdot h_{erz}/4$

Multiplying the value of the charge bulk densityg_{bd}(g_{bd}= 0.9 t/m^3) by the reaction zone volume, we determine the charge mass in the reaction zone M_{chrz}.

$$M_{chrz} = V_{rz} \cdot g_{bd}$$

Knowing the charge composition in the reaction zone, taking into account the fact that wood chips in the charge completely burn out inthe reaction zone, the charge consists of quartzite, coke, coal and charcoal (in our experiments, coal from Shubarkul deposit was used instead of charcoal – itsproperties are close to charcoal).

$$M_{chrz} = V_{rz} \cdot g_{bd} = 0.41 \cdot 0.9 = 0.369 t = 369 kg$$

Next, we calculate the percentage composition of the charge in the reaction zone, the zone where the temperature reaches the melting temperature of quartzite. Then we determine the mass content of the charge components in the reaction zone. In our experiments, the calculated composition was verified by chemical analysis of samples from the frozen reaction zone. The content, mass %, were determined: quartzite 51-53, oil coke and coal 17-19, Shubarkul coal 30-32. The calculated mass of the charge (M_{chrz} = 369 kg) was distributed according to the percentage values of their content and obtained that in the reaction zone in our furnace, in kg: quartzite 191-192, coal and coke combined 66-69, Shubarkul coal 111-113. It was difficult to analytically separate coal and coke in the frozen part of the reaction zone. We combined all reducing agents into one numerical value $V_{reduction}$ = 177kg with the quartzite mass 192 kg.

We calculate the change in the heat content of the charge in the reaction zone by the average value of the specific heat of the charge components (for accurate calculation, it is necessary to calculate the change in the heat content in the reaction zone for each component of the charge and then sum it up). Taking into account that the coefficients of specific heat capacity of the components differ little and our calculation is approximate, we took $C_{thermal}$= 1.3 kJ / (kg · °C). [10].

To heat the charge in the reaction zone from 950°C to 1750°C, it is necessary:

In our experiments:$Q_{chrz} = M_{chrz} \cdot C_{thermal} \cdot (1750-950) = 369 \cdot 1.3 \cdot 800 = 383760$ kJ

The furnace power taking into account its efficiency, η =0.60– usefulpower, $P_{useful} = 2 \cdot 10^6 \cdot 0.60 = 1.2 \cdot 10^6$ W = $1.2 \cdot 10^3$ kW

The charge heating time in the reaction zone from 950°C to 1750°C is calculated by the formula:

$\tau = Q_{chrz} / P_{useful} = 383760 / 1.2 \cdot 10^3 = 319.8$ s =320 s =5.3 min

With such a power of electric current, the voltage between the electrodes was 90-100 V, the current density on the electrode was 12.2 A/cm^2.

Conclusion

A set of studies performed on the industrial ore-smelting furnace with the capacity of 2 MW for production of silicon allows us to conclude the following:

1. The most important factors determining the success of the efficient production of silicon by the carbothermal method in the electric arc furnace:

- the size and shape of the charge components, which depend on the furnace power and the maximum allowable electric current density on the electrodes;

- the temperature in the reaction zone of the furnace must be higher than the melting temperature of quartzite;

- the important technological parameter is the duration of heating the charge in the range: 950-1750°C – the temperature range of the shell formation containing silicon carbides.

2. In the reaction zone for the implementation of a chemical reaction: $SiC + SiO_2 = 2Si + CO_2$, temperature should exceed 2250°C [7], and this is an increase in the specific energy consumption for obtaining the product, therefore, to ensure this condition, it is necessary to carry out the silicon reduction process with a minimum amount of silicon carbide formed, which is formed in the temperature range of 950-1750°C. Therefore, the heating of the charge in this temperature range should be carried out in a minimum time, the possibility of this is ensured by the maximum allowable current density for the electrodes, in our experiments it is 12.5 A/cm^2, and by upsetting the charge into the reaction zone with the help of crush (a mechanism for upsetting and crushing sintered pieces from the charge) every 20-30 minutes of a “quiet” process with the obligatory “peaking” (piercing the charge layer with birch peaks) of the charge on the top for uniform gas permeation.

3. After the release of the silicon melt from the furnace and the collapse of the roof of the reaction zone to move the charge with the temperature of 950-1000°C into the reaction zone, it is necessary to switch the furnace to the maximum possible power for the period of time of melting the quartzite pieces in the reaction zone (in our experiments for 5.3 min – heating until melting the quartzite pieces and for the implementation of the main reactions of silicon reduction for another 10-20 minutes.

4. In the temperature range of 950-1000°C, the furnace should provide the maximum charge heating rate in the cyclic mode of unloading the product from the furnace (discharging the silicon melt). With the continuous release of the silicon melt from the furnace, it is necessary to maintain a mode with a current density on the electrodes above 12 A/cm^2. When the size of quartzite pieces in the charge is not more than 200 mm.

5. Do not allow the charge to enter the reaction zone with a temperature below 950°C.

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Кен балқыту пешінде кремний өндіру процесін зерттеу және технологиялық процесті оңтайландыру

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ТҮЙІНДЕМЕ

Бұл мақалада өнеркәсіптік доғалы пеште кремнийді балқыту режимдерін оңтайландыру бойынша өндірістік тәжірибелердің нәтижелері берілген. Балқыту процесінің негізгі факторлары шихта компоненттерінің фракцияларының мөлшері және қыздырудың температуралық режимі болып табылады. 950-ден 1410^{0}С-қа дейінгі температура диапазонында реакция аймағында шихтаны қыздыру жылдамдығы өнімділікке ерекше әсер етеді. Бұл температура диапазонында кварцит бөліктерінде отқа төзімді кремний карбиді түзіледі, бұл реакция аймағындағы электр тогының шамасының төмендеуіне және оның қатуына әкеледі. Түзілген горнизаж электродтарды заряды жоғары электр өткізгіштік аймағына ығыстырады, бұл төмендетілген заряд қабаты арқылы кремний тотығы шығарындыларының ұлғаюына әкеледі. Мұндай процесті коррекциялау реакция аймағындағы температураларды және балқыма ұзақтығын арттыруды талап етеді. Пештің қуатына және реакция аймағының көлеміне байланысты кварцит фракцияларының мөлшерін есептеу әдісі әзірленді. Жеңілдетілген есептеудің мысалы ұсынылды.

Түйін сөздер: кремний, кварцит, кокс, карбид, оксид, доғалы пеш.

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Исследование процесса получения кремния в рудно-плавильной печи и оптимизация технологического процесса

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АННОТАЦИЯ

В данной статье представлены результаты производственных экспериментов по оптимизации режимов выплавки кремния в промышленной дуговой печи. Основными факторами процесса плавки являются размер фракций компонентов шихты и температурный режим нагрева. Особое влияние на производительность оказывает скорость нагрева шихты в зоне реакции в диапазоне температур от 950 до 14100°С. В этом интервале температур установлено образование тугоплавкого карбида кремния на кусках кварцита, что вызывает падение величины электрического тока в зоне реакции и ее замерзание. Образующийся горнисаж смещает электроды в зону большей электропроводности заряда – вверх, что приводит к увеличению выделения оксида кремния через слой пониженного заряда. Коррекция такого процесса требует повышения температур в зоне реакции и продолжительности плавки. Разработан метод расчета размеров фракций кварцита в зависимости от мощности печи и размера зоны реакции. Предлагается пример упрощенного расчета.

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Removal of Ferrous using Citric Acid in Patchouli Oil Purification by Complexometry

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ABSTRACT

This study is aimed to examine citric acids as a potential chelating agent to decrease colloidal impurities in patchouli oil to improve its quality. It covers colour, specific density, refractive index, acid value, iron content, oleoresin oil content, and patchouli alcohol. Complete Randomized Design with factorial design is used with two factors and repeated 3 times. Factors are (1) citric acid concentration consists of 0.25%, 0.5%, 1.0% and 1.50% (w/v), (2) stirring time of 30, 60 and 90 minutes. Further, purified oil by citric acid was compared to purified oil by Ethylene Diamine Tetra Acetate (EDTA). Findings show that the concentration of chelating agents and the time of stirring have an effect on the quality of patchouli oil. The higher the chelating concentration and the more the stirring time, the better the quality of purified patchouli oil in terms of colour, specific density, refractive index, acid value, and iron content. Findings also show that citric acid has almost the same performance as EDTA. The main components in patchouli oil (patchouli alcohol and oleoresin oil) are not affected by treatment. Purified patchouli oil by using citric acid meets Indonesian National Standard (SNI) requirements so citric acid is one of the potential chelating agents.

Keywords: Purification process, essential oil, chelating agent, patchouli oil, iron content.

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Introduction

Patchouli oil is extracted from the Pogostemon patchouli plant or Pogostemon cablin Benth. It is one of the priceless essential oil and until now there has been no other type of essential or synthetic oil that can replace patchouli oil as a fragrance binder (perfume) [1]. Patchouli oil is usually not fractionated into its derivatives such as citronella oil, vetiver, clove and others, because it has very harmonious components and no one component is very prominent and is the most difficult volatile essential oil compared to other essential oils.

Others, so that it can be used to make compositions of a fragrance or perfumery compound as a cosmetic
raw material [2]. Patchouli oil has fixative properties, namely the ability of the oil to bind several other odours/perfumes so that the composition forms a unified odour. The smell of patchouli oil is pungent, strong, long-lasting, and musty.

However, in recent years, the price of patchouli oil from local communities has fallen significantly due to the poor quality of patchouli oil (high colloidal impurities). The impurities cover dark brown colour, high acid number, high iron content (ppm) and high oleoresin oil content (%). The nature of patchouli oil

is determined by its chemical compounds. These components can be terpenes, alcohols, aldehydes, acids, esters, ketones and others. In addition, these components may contain saturated or unsaturated bonds so that patchouli oil is easily oxidized, hydrolyzed and polymerized [3].

Most local community farmers in developing countries use metal iron distillers [[4], [5]]. Patchouli oil turns dark in colour due to the influence of Fe_2O_3 which is a sensitizer to double bonds and the compounds contained in the oil [6]. Due to the influence of base and temperature, the resinification reaction in essential oils is accelerated [7]. The reaction between metal ions and acids in the oil will form salts which will make the oil darker and more concentrated. The decline in the quality of patchouli oil can be prevented by using stainless steel distiller. However, it is considered expensive by local community farmers [8].

Patchouli oil purification can be done by vacuum distillation and re-distillation [3], adsorption [9], and using chelating agents [10]. The redistillation purification method has the disadvantage of being relatively expensive, long processing time and charred-smelling oil [[11], [12]]. Purification of dark-coloured patchouli oil by flocculation in principle is to bind the metals contained in the oil by adding a chelating agent to form a complex salt that coagulates and settles. Furthermore, the precipitate formed is separated from patchouli oil through filtration. Chelating agents have been used such as tartrate acid and Ethylene Diamine Tetraacetic acid (EDTA). However, those chelating agents are quite difficult and more expensive for local community farmers. Meanwhile, citric acid has a chelating agent effect, is organic, and has abundant availability [13]. Thus, this study aims to examine citric acids as a potential chelating agent to decrease colloidal impurities in patchouli oil to improve its quality.

Experimental part

Patchouli oil used in this study is patchouli oil obtained from a local community farmer in West Sumatera, Indonesia. The citric acid solution used was a 65% concentration with the addition of 1% ethanol 96%. Then, the amount of patchouli oil was added with a solution of a chelating agent (65%) as much as 0.25%, 0.5%, 1%, and 1.5% by weight of patchouli oil and stirred with a rotation speed of 300 rpm for 30 minutes, 1 hour, and 1.5 hours. CaO 1% of oil is poured into the solution and stirred again for 2 hours with a rotation speed of 500 rpm. The mixture is allowed to stand overnight until a layer of patchouli oil is formed at the top and a precipitate at the bottom which will then be separated by filtration. The treatment which is a combination of the level of concentration of each chelating agent and the time of stirring is planned in a completely random factorial with 3 repetitions. The purified oil is examined based on Indonesian National Standard (SNI 06-2385-2006). Oil clarity is in Transmittance percentage (%T).

Results and Discussion

Preliminary analysis is carried out to determine the physicochemical properties of patchouli oil before purifying. It is compared with physicochemical properties after purifying and standard SNI 06-2385-2006. Table 1 describes the physicochemical characteristics of patchouli oil before purification.

Table 1 - Physicochemical characteristics of patchouli oil before purification

Characteristics	Standard based on SNI 06-2385-2006	value
Color - Visual - Transmisi,%	Yellowish to dark brown	Dark brown 2.8±0.13
Specific density (25^0C)	0.943-0.983	0.988±0.15
refractive index (ND^{20})	1.504-1.514	1.512±0.12
Acid value	Max. 5.0	8.04±1.86
Iron content, Fe (ppm)	-	328.04± .98
Patchouli alcohol (%)	Min.30	28.54%

Table 1 shows the physicochemical characteristics of patchouli oil based on standard, before and after purifying. Based on the results of the preliminary analysis of both the physical and chemical characteristics of patchouli oil from local community farmers, some characteristics such as specific density, acid value, oleoresin oil content, and patchouli alcohol do not meet standard SNI 06-2385-2006. Patchouli oil is one of the important characteristics that are below standard.

The dark colour of the oil causes the level the clarity of the oil to be very low, and this is due to its high iron content [[13], [14]] argues that contamination by iron occurs during the distillation process which uses a metal iron distiller. Iron ions

can stimulate oxidation reactions in conjugated double bonds found in patchouli alcohol compounds in patchouli oil to produce colour-forming chromophore compounds from groups >C=C< or >C=C [15]. Patchouli alcohol compound is the main component in patchouli oil. Dark colours cause low clarity. Colour is a parameter that is easily visible, therefore greatly affecting consumer acceptance and can reduce quality [[16], [17]].

In the purification process, the effect of concentration (EDTA, and citric acid) on the patchouli oil clarity is presented in Figure 1.

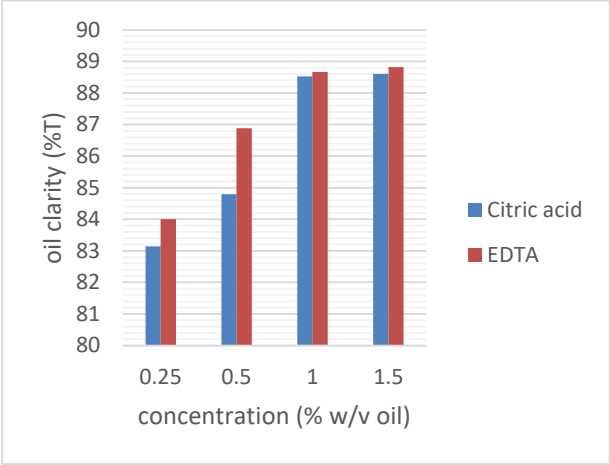

Figure 1 – Effect of concentration on the oil clarity

Findings showed that chelating agent concentration has a significant effect on oil clarity. The oil clarity could be 88.52%T by using 1% citric acid and 88.61%T by using 1.5% citric acid as a chelating agent. The significant oil clarity is increased from a concentration of 0.5% to 1% citric acid (w/v). Meanwhile if using EDTA as a chelating agent, the oil clarity could be 88.67%T by using 1% and 88.82%T by using 1.5% of concentrations. The significant oil clarity is also increased from a concentration of 0.5% to 1% EDTA (w/v). Citric acid has nearly the performance as EDTA in concentrations of 1% and 1.5%.

Fe content in patchouli oil is the cause of the dark colour and low quality of the oil. Thus, the effect of the chelating agent, concentration, and time of stirring on Fe content is presented in Table 2.

Findings showed that the interaction of concentration and stirring time has a significant effect on Fe content. The higher concentrations and the longer the stirring time, the less Fe content. The least Fe content is by using a 1.5% chelating agent during 90 minutes time of stirring. However, the result of the 1% chelating agent during 60 minutes is not different significantly when compared to 1.5% chelating agent during 90 minutes.

Citric acid has 3 pairs of free electrons meanwhile EDTA has 6 pairs of free electrons form C=O and N atoms. Thus, EDTA has better performance. However, citric acid has almost similar performance to EDTA. The further result before and after purification is presented in Table 3.

Table 2 – Interaction between the chelating agent, concentration, time of stirring and Fe content

Chelating agent	Concentration (%)	Stirring time (minutes)	Fe content (ppm)
EDTA	0.25	30	19.65
		60	17.28
		90	15.34
	0.50	30	18.35
		60	14.13
		90	13.04
	1	30	14.05
		60	13.04
		90	10.34
	1.5	30	11.57
		60	9.05
		90	8.87
Citric acid	0.25	30	19.85
		60	18.08
		90	16.24
	0.50	30	18.05
		60	13.83
		90	13.04
	1	30	15.05
		60	12.54
		90	11.04
	1.5	30	11.92
		60	9.25
		90	9.30

Table 3 showed that after purifying, specific density (25°C), refractive index (ND^{20}), acid value, iron content (ppm), oleoresin oil content, and patchouli alcohol meet the standard. Citric acid as chelating agent has three carboxyl functional group

(-COOH) by setting its concentration and strirring time, H atoms occured deprotonation [18]. Ion H^+ replaces Fe^{2+}. Thus, it could decrease Fe content and increase clarity of patchouli oil [19].

Patchouli oil from local community farmers is dark brown due to it contains colloidal Fe. The addition of citric acid as a chelating agent reacts with Fe metal to form chelate complex ions [20].

Table 3 - Physicochemical characteristics of patchouli oil before and after purification by using citric acid as a chelating agent

Characteristics	Standard based on SNI 06-2385-2006	Before purification	After purification
Color Visual Transmision, %	Yellowish to dark brown	Dark brown 2.8±0.13	Light yellow 57.3±0.16
Specific density (25^0C)	0.943-0.983	0.988±0.15	0.963±0.26
refractive index (ND^{20})	1.504-1.514	1.512±0.12	1.509±0.31
Acid value	Max. 5.0	8.04±1.86	0.12±1.63
Iron content, Fe (ppm)	-	328.04± .98	9.3±4.12
Patchouli alcohol (%)	Min.30	28.54%	34.84%

A chelating agent forms a complex salt that binds to the iron occurred in the oil. The chelating agent in forming the complex salt lumps is supported by calcium oxide which also neutralizes the acidity in the oil [21]. The decrease in Fe content significantly from 328.04±3.98 ppm to 9.3±4.12 cause a significant change in the colour of the oil. Patchouli oil changes colour to clear yellow and is the preferred colour in the market.

Conclusion

Purification of patchouli oil by using citric acid as a chelating agent can significantly improve the quality of the oil value. Its performance is almost similar to EDTA in certain conditions. This refining can improve the characteristics of the oil from the aspect of colour, patchouli alcohol, acid number, iron content (ppm) and oleoresin oil content (%). Citric acid can reduce iron content by much as 97.16%. Therefore, citric acid has promising potential to be used in patchouli oil refining for local community farmers.

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Комплексометрия бойынша пачули майын тазартуда лимон қышқылын қолдану арқылы темірді жою

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ТҮЙІН

Бұл зерттеу пачули майындағы коллоидты қоспаларды азайту арқылы оның сапасын жақсарту үшін лимон қышқылдарын потенциалды хелатизатор ретінде пайдалануды зерттеуге бағытталған. Зерттеулер түсті, меншікті тығыздықты, сыну көрсеткішін, қышқылдық мәнін, темір құрамын, олеорезин майының мазмұнын және пачули спиртін қамтиды. Факторлық дизайнмен толық рандомизацияланған дизайн екі фактормен пайдаланылады және 3 рет қайталанады. Факторлар (1) лимон қышқылының концентрациясы 0,25%, 0,5%, 1,0% және 1,50% (салм/көлем) тұрады, (2) араластыру уақыты 30, 60 және 90 минут. Одан әрі лимон қышқылымен тазартылған май этилендиамин тетра ацетаты (EDTA) арқылы тазартылған маймен салыстырылды. Нәтижелер пачули майының сапасына хелаттандырушы заттардың концентрациясы мен араластыру уақыты әсер ететінін көрсетеді. Хелатизатордың концентрациясы неғұрлым жоғары болса және араластыру уақыты неғұрлым көп болса, тазартылған пачули майының түсі, меншікті тығыздығы, сыну көрсеткіші, қышқылдық мәні және темір мөлшері бойынша сапасы соғұрлым жақсы болады. Нәтижелер сонымен қатар лимон қышқылы EDTA-мен бірдей сипаттамаларға ие екенін көрсетті. Пачули майындағы негізгі компоненттер (пачули спирті және олеорезин майы) өзгермейді. Лимон қышқылын қолдану арқылы тазартылған пачули майы Индонезия ұлттық стандартының (SNI) талаптарына сәйкес келеді, сондықтан лимон қышқылы потенциалды хелат агенттерінің бірі болып табылады.

Түйін сөздер: Тазарту процесі, эфир майы, хелат агенті, пачули майы, темір құрамы

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Удаление железа с помощью лимонной кислоты при очистке масла пачули по комплексометрии

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АННОТАЦИЯ

Это исследование направлено на изучение использования лимонной кислоты в качестве потенциального хелатирующего агента для улучшения качества масла пачули за счет уменьшения количества коллоидных примесей. Исследования включают цвет, удельный вес, показатель преломления, кислотное число, содержание железа, содержание олеорезинового масла и спирт пачули. Полностью рандомизированный план с факторным планом используется с двумя факторами и повторяется 3 раза. Факторы включают (1) концентрацию лимонной кислоты 0,25%, 0,5%, 1,0% и 1,50% (вес/объем), (2) время перемешивания 30, 60 и 90 минут. Кроме того, масло, рафинированное лимонной кислотой, сравнивали с маслом, рафинированным этилендиаминтетраацетатом (ЭДТА). Результаты показывают, что на качество масла пачули влияет концентрация хелатирующих агентов и время смешивания. Чем выше концентрация хелатора и дольше время смешивания, тем лучше качество рафинированного масла пачули с точки зрения цвета, удельного веса, показателя преломления, кислотного числа и содержания железа. Результаты также показали, что лимонная кислота имеет те же характеристики, что и ЭДТА. Основные компоненты масла пачули (спирт пачули и масло олеорезина) не изменились. Масло пачули, рафинированное с использованием лимонной кислоты, соответствует требованиям Национального стандарта Индонезии (SNI), поэтому лимонная кислота является одним из потенциальных хелатирующих агентов.

Ключевые слова: процесс очистки, эфирное масло, хелатирующий агент, масло пачули, содержание железа.

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Metallurgy

Distribution of antimonium chalcogenides under conditions of vacuum thermal processing of mattes

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ABSTRACT

It was established based on the analysis of the results of published works and the results obtained by the authors that there is no information on the behavior and distribution of antimony chalcogenides - Sb_2S_3, Sb_2Se_3, Sb_2Te_3, as well as double systems - Sb_2S_3-Sb_2Se_3, Sb_2S_3-Sb_2Te_3 and Sb_2Se_3-Sb_2Te_3 under the vacuum processing conditions for polymetallic mattes performed at 1100-1250 °C and a vacuum of 15 - 0.7 kPa. It was found based on the saturated vapor pressure values for monochalcogenides that the vapor pressure of free antimony sulfide will be 58.95 kPa at 1100 °C, i.e. the lower limit of the technological interval, which indicates its complete transfer to the vapor phase when the mattes are evacuated; the vapor pressure of free antimony selenide at 1100 °C exceeds the atmospheric pressure value (101.3 kPa), and Sb_2Se_3 would be completely extracted into the vapor phase in vacuum; the boiling point of liquid antimony telluride at atmospheric pressure corresponds to 971 °C, and it would be extracted into the vapor phase under the conditions of matte evacuation. The thermodynamic evaporation characteristics of antimony chalcogenides were found. It was concluded based on the location of the boundaries of the liquid and vapor phase coexistence fields that it is impossible to separate binary systems of antimony chalcogenides into separate compounds in the process of one evaporation cycle – condensation, in binary systems. Different effects of pressure reduction over melts were found. Lowering the pressure from atmospheric one to 0.7 kPa in Sb_2S_3-Sb_2Se_3 system did not change the position of the boundaries of the liquid and vapor fields (L + V) under the temperature; field width (L+V) decreases with decreasing pressure in Sb_2S_3-Sb_2Te_3 system; the field width first decreases with temperature, then increases in system Sb_2Se_3- Sb_2Te_3. At the same time, the position of the boiling curves of antimony chalcogenide solutions indicates the complete transfer of compounds into the vapor phase under the conditions of matte distillation processing (at 1100-1250 °C) at atmospheric pressure which is important for assessment of the distribution of antimony and rare metals - selenium and tellurium by processed products.

Keywords: antimony, sulfur, selenium, tellurium, chalcogenide, vapor pressure, vacuum, matte, thermodynamics, distribution.

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Introduction

Copper sulfides (Cu_2S) and iron (FeS) are the basis of polymetallic mattes of copper and lead plants forming an unstable complex - $FeCu_2S_2$ in melt. There are compounds of non-ferrous metals (PbS, ZnS), rare elements cadmium, antimony, indium, etc. in the form of sulfides, as well as selenium and tellurium, isomorphically replacing sulfur in sulfides in addition to the main components in the matte. Moreover, the largest amount in the matte after PbS and ZnS is represented by antimony chalcogenides.

Binary antimony sesquisulfide systems with Cu_2S and FeS as applied to the conditions of vacuum-thermal processing of sulfide melts are considered in detail in the monograph [1], the behavior of copper chalcogenides under the same conditions - in [2]. As a result, it was found that almost complete release of antimony sulfide into the condensate during distillation in a vacuum should be expected, and it was confirmed in the process of factory technological tests for the processing of mattes. The dissociation pressure of pure copper sulfide will fluctuate within 0.5-7 Pa, of copper selenide - 28-230 Pa, of copper telluride - 1.5-9 Pa in the process of distillation of volatiles at low pressure [2], performed, as a rule, at 1100-1250 °C. Therefore, copper sulfide and telluride completely, and copper selenide in the main degree will concentrate in the stillage residue.

The liquid-vapor equilibrium in the chalcocite-antimonite system at low pressures was studied in the study [3], and it was found that almost complete release of antimonite into the condensate should be expected during distillation in a vacuum, however, the pressure during the technological implementation of the matte distillation separation process should be at least 700 Pa in order to avoid Cu_2S build-up formation.

The removal of non-ferrous metal impurities from copper matte was studied by the authors in [4] where it was found that the antimony content was below 0.1 % after vacuum treatment at 1200 °C and a pressure of 60 Pa.

The authors [[5], [6], [7], [8], [9], [10]] conducted studies to determine the oxide solubility of copper, lead, arsenic and antimony from copper-lead mattes into slag. The results obtained can be used to predict the loss of valuable components with slag, and to develop optimal solutions to reduce the total loss of metals during processing.

The teams of authors [[11], [12]] studied the decomposition of jamsonite ($Pb_4FeSb_6S_{14}$), and the optimal temperatures equal to 650 and 900 °C were established for the separation of antimony and lead sulfides. Up to 98 % Sb_2S_3 with a purity of 99.17 % and up to 99.5 % PbS with a content of 98.7 % of the main compound was obtained.

The extraction of non-ferrous metals from secondary raw materials by vacuum sulfiding at 1050 °C was studied in [13], and a new method intended to remove impurities from secondary copper raw materials was proposed.

The authors of [14] found that when copper mattes are evacuated at 1250 °C and a pressure of 130 Pa, up to 92 % of antimony in the form of sulfide can be extracted into the vapor phase and, accordingly, into the condensate.

To date, a modified volumetric model of the molecular interaction of the components in a PbS - Sb_2S_3 binary system was developed and used consistent with experimental data. It can be used for the technological separation and purification of sulfides [15].

Despite a significant number of studies for the Sb_2S_3 isolation from sulfide melts, there are no studies on the behavior of other antimony chalcogenides and their binary systems which form a continuous series of solid solutions [16] under the conditions of vacuum thermal processing of mattes.

In this regard, the behavior of antimony monochalcogenides and binary systems of antimony chalcogenides is of great interest at high temperatures and low pressure, with the construction of vapor-liquid equilibrium fields for binary systems to consider which enable to judge the distribution of elements, as well as the possibility of their concentration in a separate middling product.

Distribution of antimony monochalcogenides

Judgment on the behavior and distribution of antimony chalcogenides is based on the saturation vapor pressure of each of them.

Vapor pressure over antimony sulfide. A significant number of studies, including [[17], [18], [19], [20], [21], [22], [23], [24], [25], [26]], and summarized in monographs [[27], [28], [29]], are devoted to determination of the magnitude of the pressure and composition of vapor over antimony sesquisulfide. Such attention to the thermodynamic studies of Sb_2S_3 is due to the semiconductor properties of the compound and the very complex

composition of the vapor phase. When the composition of vapor over liquid antimony trisulfide is studied, it was established [[17], [21], [22]] that SbS, S_2, Sb_2S_2, Sb_2S_3, Sb_2S_4, Sb_3S_2, Sb_3S_3, Sb_3S_4, Sb_4S_3, Sb_4S_4, Sb_4S_5 molecules and other fragment ions were present in it.

Due to the complex composition of the vapor phase, when the boiling points of alloys were calculated, the saturation vapor pressure of antimony sulfide found by the boiling point method was used [[23], [24], [30]]. This method does not require knowledge of the molecular weight of the vapor, and the corresponding dependences: $\ln p_{Sb_2S_3(l)}[Pa] = 23{,}889 - 17718 \cdot T^{-1}$, while the thermodynamic functions of evaporation: $\Delta H^{vap}_{Sb_2S_3(l)} = 147{,}31$ kJ/mol, $\Delta S^{vap}_{Sb_2S_3(l)} = 102{,}79$ J/(mol K).

The vapor pressure of free antimony sulfide at 1100 °C, i.e. at the lower boundary of the technological interval, will be 58.95 kPa indicating its complete transfer to the vapor phase when the matte is evacuated at a rarefaction of 15 - 0.7 kPa.

Vapor pressure over antimony selenide. A number of researchers were involved in physicochemical studies of liquid alloys. The authors of [31] measured the kinematic viscosity and density of melts in the composition range of 40 mol. % Sb_2Se_3 + 60 atm. % Se – 20 mol. % Sb_2Se_3+80 atm. % Sb from melting temperature to 1100-1200 °C.

The crystallization kinetics of glassy alloys was studied in [32] using differential scanning calorimetry at different heating rates. $Se_{100-x}Sb_x$ ($2 \le x \le 10$), the activation energy of the crystallization process, the order parameter, the rate constant, the frequency factor was determined, and it was found that chalcogenide glasses with a higher crystallization rate, have lower thermal stability.

The authors of [33] studied the behavior of antimony sesquiselenide during sublimation in vacuum and established the congruent character of evaporation.

Mass spectrometric determination of vapor composition over Sb_2Se_3 established [[34], [21]] the predominant presence of SbSe molecules, half less - Sb_2Se_2, triple less – Sb_2Se_3 and so on in the descending order: Sb_3Se, Sb_4Se_4, Sb_4Se_3, Sb_3Se_3, Sb_3Se_2, Se_2, Sb_2Se_4 and very few Sb_2Se_4.

The vapor pressure of antimony sesquiselenide using radioisotopes in the temperature range from 491 to 687 K (218–414 °C) was determined in studies [[35], [36]]. The temperature dependence of the vapor pressure corresponded to the expression:

lg *p*[mm Hg]= 8,7906-6432,3·T^{-1}.

Vapor pressure over liquid antimony selenide in the temperature range of 550–868 °C (823–1201 K) was determined by a static method using a quartz membrane manometer in the study [20]. The results of the determinations are described by the equation: lg *p*[mm Hg]= (8,4130±0,0328) – (7220,4±250)·T^{-1}.

Later, the authors of [[37], [38]] determined the vapor pressures and activities of selenium and antimony at 994 K (721 °C) by the isopiestic method over the entire concentration range of the system.

The equation of the dependence of vapor pressure on temperature recommended in [39] was used by us to calculate the boundaries of the liquid-vapor phase transition, and it was transformed by us to the form: $\ln p_{Sb_2Se_3(l)}[Pa] = 23{,}908 - 16498 \cdot T^{-1}$.

Hence the enthalpy and entropy of liquid Sb_2Se_3 vaporization: $\Delta H^{vap}_{Sb_2Se_3(l)} = 137{,}17$ kJ/mol, $\Delta S^{vap}_{Sb_2Se_3(l)} = 102{,}95$ J/(mol K).

The vapor pressure of free antimony selenide at 1100 °C, the lower boundary of the technological range, exceeds atmospheric pressure (101.3 kPa), and Sb_2Se_3 will be completely extracted into the vapor phase in a vacuum.

Vapor pressure over antimony telluride. The number of works devoted to thermodynamic studies of antimony sesquitelluride is not so large in comparison with sulfide and selenide. The physical and chemical properties of the antimony - tellurium and Sb_2Te_3 system were studied by the authors of [[19], [20], [25], [29], [39], [40], [41], [42], [43]]. When the composition of the vapor was studied by the mass spectrometric method, the existence of the following molecules was established [[11], [29]]: Sb_4, Sb_2, SbTe, Sb_2Te_2, Te_2. However, the authors of [41] noted that the “degree of incongruence” is very low during the sublimation of antimony sesquitelluride in vacuum at 770–830 K.

Sb_2Te_3 steam pressure values found by different methods differ from each other. In this regard, the dependence of the vapor pressure of antimony telluride obtained based on measurements by the torsion-effusion method in the latest work [26] were used by us and converted to the form: $\ln p_{Sb_2Te_3(\partial)}[Pa] = 31{,}776 - 25181 \cdot T^{-1}$

The change in the enthalpy and entropy of evaporation corresponds to the following values:

$\Delta H^{vap}_{Sb_2Te_3(l)} = 209{,}39$ kJ/mol,

$\Delta S^{vap}_{Sb_2Te_3(l)} = 168{,}36$ J/(mole·K).

The boiling point of liquid antimony telluride at atmospheric pressure corresponds to 971 °C, and it will be extracted into the vapor phase under the vacuum conditions of matte.

Distribution of chalcogenides in Sb_2S_3-Sb_2Se_3, Sb_2S_3-Sb_2Te_3 and Sb_2Se_3-Sb_2Te_3 systems

Measurements of microhardness and thermal conductivity of Sb_2S_3-Sb_2Se_3 alloys and X-ray diffractometry established the formation of solid solutions between these compounds which crystallize in a rhombic lattice, in the same way as the initial compounds [44]. When antimony selenide is added to sulfide, sulfur atoms statistically replace selenium atoms, and the lattice parameters gradually increase as sulfur atoms are replaced by selenium atoms with a larger atomic radius. In connection with the formation of a continuous series of solid solutions and the unlimited solubility of antimony chalcogenides in the liquid phase, the boundaries of vapor-liquid equilibrium are calculated in Sb_2S_3-Sb_2Se_3, Sb_2S_3-Sb_2Te_3 and Sb_2Se_3-Sb_2Te_3 systems.

There are no studies on vapor-liquid equilibrium in binary systems of antimony chalcogenides.

Due to the fact that the chalcogenides of one metal, as a rule, have a very slight deviation or lack thereof under the Raoult law [16], Sb_2S_3-Sb_2Se_3, Sb_2S_3-Sb_2Te_3 and Sb_2Se_3-Sb_2Te_3 binary systems were regarded as ideal ones.

The boundaries of the vapor-liquid equilibrium fields were calculated based on the partial pressure values of the saturated vapor of the alloy components. At the same time, the temperature at which the sum of the partial pressures of the vapor of the components is equal to atmospheric pressure or another one corresponding to the conditions of vacuum distillation of mattes (15 - 0.7 kPa) was considered as the boiling point of the melt.

The composition of the vapor phase at the boiling point was determined on the basis of the Clapeyron–Mendeleev equation as the ratio of the partial pressure of one component to the total pressure at this temperature. The boundaries of vapor-liquid equilibrium in binary systems of antimony chalcogenides are shown in Figure 1.

State diagrams of condensed systems are constructed only for binary Sb_2S_3-Sb_2Se_3 and Sb_2Se_3-Sb_2Te_3 [44]. Sb_2S_3-Sb_2Te_3 system liquidus line is drawn between the melting points of chalcogenides conditionally, by a dotted line.

It can be concluded based on the position of the boundaries of the liquid and vapor phase coexistence fields that it is impossible to separate binary systems of chalcogenides into separate compounds in the process of one evaporation cycle - condensation. The different influence of the decrease in pressure over the melts should be noted. A decrease in pressure from atmospheric to 0.7 kPa does not change the position of the boundaries of the liquid and vapor fields *(L + V)* in temperature in Sb_2S_3-Sb_2Se_3 system; field width (*L+V*) decreases with decreasing pressure in Sb_2S_3-Sb_2Te_3 system; the field width first decreases with temperature, then increases in Sb_2Se_3- Sb_2Te_3 system. Apparently, inaccuracies in determination of the vapor pressure values of the initial antimony chalcogenides are the reason for such a change in the position of field boundaries (*L+V*) in the last two systems.

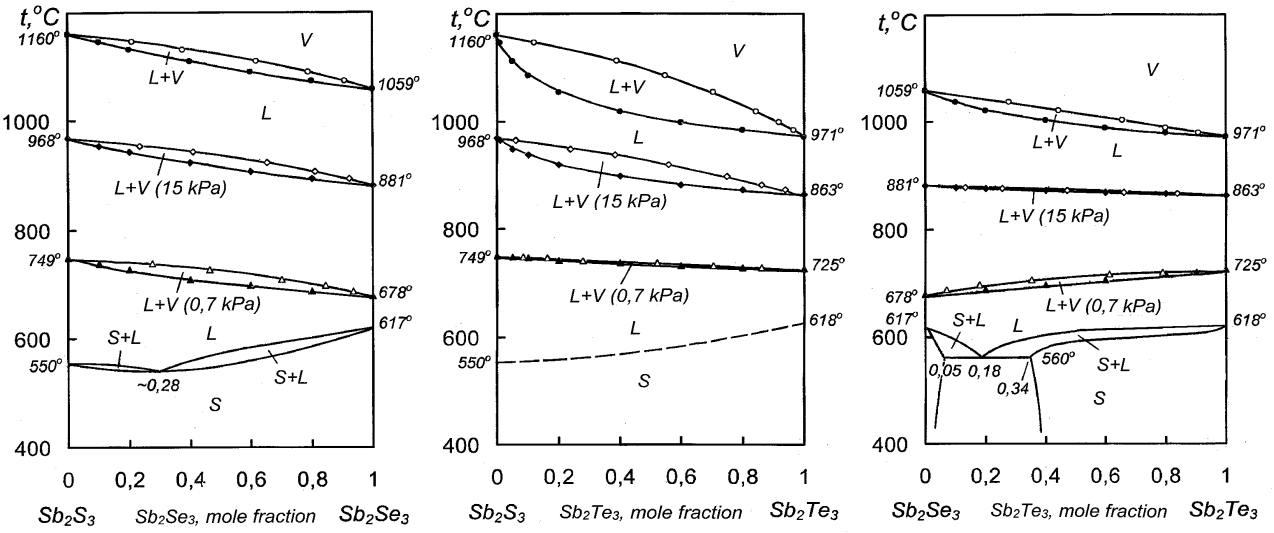

Figure 1 - Liquid-vapor phase equilibria in binary systems of antimony chalcogenides.

At the same time, the position of the boiling curves for the solutions of antimony chalcogenides indicates the complete transfer of compounds into the vapor phase under the conditions of distillation processing of mattes (at 1100-1250 °C) even at atmospheric pressure.

Conclusions

It was established based on the saturated vapor pressure values for antimony monochalcogenides and the construction of the boundaries of vapor-liquid equilibrium in Sb_2S_3-Sb_2Se_3, Sb_2S_3-Sb_2Te_3 and Sb_2Se_3-Sb_2Te_3 systems that antimony monochalcogenides, as well as solutions of antimony chalcogenides will be completely transferred to the condensate under the conditions of distillation processing of polymetallic mattes performed, as a rule, at 1100-1250 °C and with a rarefaction of 15 - 0.7 kPa. Separation of antimony chalcogenides into individual compounds during distillation is not possible.

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ШТЕЙНДЕРДІ ВАКУУМДЫҚ-ТЕРМИЯЛЫҚ ҚАЙТА ӨҢДЕУ ЖАҒДАЙЫНДА СҮРМЕ ХАЛЬКОГЕНИДТЕРІНІҢ ТАРАЛУЫ

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ТҮЙІНДЕМЕ

Жарияланған жұмыстардағы және авторлар алған нәтижелерді талдау негізінде 1100-1250 ^{0}C температурада және қысымды 15-0,7 кПа дейін сирету кезінде жүзеге асырылатын полиметалл штейндерді вакуумдық қайта өңдеу жағдайында Sb_2S_3, Sb_2Se_3, Sb_2Te_3 сүрме халькогенидтерінің, сондай-ақ Sb_2S_3-Sb_2Se_3, Sb_2S_3-Sb_2Te_3 және Sb_2Se_3-Sb_2Te_3 қос жүйелерінің әрекеті мен таралуы туралы мәліметтердің жоқ екендігі анықталды. Монохалькогенидтердің қаныққан бу қысымы мөлшерлерінің негізінде 1100^{0}C кезінде – технологиялық аралықтың төменгі шекарасында бос сүрме сульфидінің бу қысымы 58,95 кПа-ны құрайтыны анықталды, бұл штейндерді вакуумдау кезінде оның бу фазасына толық ауысатынын көрсетеді; 1100^{0}C-та бос сүрме селенидінің бу қысымы атмосфералық қысымның шамасынан асады (101,3 кПа) және вакуумдағы Sb_2Se_3 толығымен бу фазасына өтеді; сұйық сүрме теллуридінің атмосфералық қысымдағы қайнау температурасы 971^{0}C-ға сәйкес келеді және штейндерді вакуумдау жағдайында ол бу фазасына шығарылады. Сүрме халькогенидтерінің булануының термодинамикалық сипаттамалары табылды. Қос жүйелерде сұйық және бу фазаларының қатар болатын өрістерінің шекарасын орналастыру негізінде сүрме халькогенидтерінің қос жүйелерінің бір булану – конденсация циклі барысында жеке қосылыстарға бөлінбейтіні туралы қорытынды жасалды. Sb_2S_3-Sb_2Se_3 жүйесінде қысымның атмосфералық қысымнан 0,7 кПа-ға дейін төмендеуі температура бойынша сұйықтық пен бу (L+V) өрістері шекараларының жағдайын өзгертпейді; Sb_2Se_3-Sb_2Te_3 жүйесінде – өрістің ені (L+V) қысым төмендеген сайын азаяды; Sb_2Se_3-Sb_2Te_3 жүйесінде өрістің ені температура бойынша алдымен азаяды, содан кейін ұлғаяды. Сонымен қатар, сүрме халькогенидтері ерітінділерінің қайнауының қисық сызығының орналасуы атмосфералық қысымда штейндерді дистилляциялық қайта өңдеу жағдайында (1100-1250^{0}C температурада) қосылыстардың бу фазасына толық ауысатынын көрсетеді, бұл сүрме және сирек кездесетін металдардың – селен мен теллурдың қайта өңдеу өнімдері бойынша таралуын бағалау үшін маңызды.

Түйін сөздер: сүрме, күкірт, селен, теллур, халькогенид, бу қысымы, вакуум, штейн, термодинамика, таралу.

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РАСПРЕДЕЛЕНИЕ ХАЛЬКОГЕНИДОВ СУРЬМЫ В УСЛОВИЯХ ВАКУУМ-ТЕРМИЧЕСКОЙ ПЕРЕРАБОТКИ ШТЕЙНОВ

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АННОТАЦИЯ

На основании анализа полученных авторами результатов и опубликованных работ установлено отсутствие сведений о поведении и распределении халькогенидов сурьмы Sb_2S_3, Sb_2Se_3, Sb_2Te_3, а также двойных систем Sb_2S_3-Sb_2Se_3, Sb_2S_3-Sb_2Te_3 и Sb_2Se_3-Sb_2Te_3 в условиях вакуумной переработки полиметаллических штейнов, осуществляемой при температуре 1100-1250°С и разрежении 15 – 0,7 кПа. На основании величин давления насыщенного пара монохалькогенидов было установлено, что давление пара свободного сульфида сурьмы при 1100°С – нижней границе технологического интервала будет составлять величину 58,95 кПа, что свидетельствует о его полном переводе в паровую фазу при вакуумировании штейнов; давление пара свободного селенида сурьмы при 1100°С превышает величину атмосферного давления (101,3 кПа) и в вакууме Sb_2Se_3 полностью будет извлечен в паровую фазу; температура кипения жидкого теллурида сурьмы при атмосферном давлении соответствует 971°С и в условиях вакуумирования штейнов он будет извлечен в паровую фазу. Найдены термодинамические характеристики испарения халькогенидов сурьмы. В двойных системах на основании размещения границ полей сосуществования жидкой и паровой фаз сделан вывод о невозможности разделения двойных систем халькогенидов сурьмы на отдельные соединения в процессе одного цикла испарение – конденсация. Отмечено разное влияние понижения давления над расплавами. В системе Sb_2S_3-Sb_2Se_3 понижение давления от атмосферного до 0,7 кПа не изменяет положение границ поля жидкости и пара (L+V) по температуре; в системе Sb_2S_3-Sb_2Te_3 – ширина поля (L+V) уменьшается с понижением давления; в системе Sb_2Se_3- Sb_2Te_3 – ширина поля по температуре вначале уменьшается, затем увеличивается. Вместе с тем, положение кривых кипения растворов халькогенидов сурьмы свидетельствует о полном переводе соединений в паровую фазу в условиях дистилляционной переработки штейнов (при температурах 1100-1250°С) при атмосферном давлении, что важно для оценки распределения сурьмы и редких металлов – селена и теллура по продуктам переработки.

Ключевые слова: сурьма, сера, селен, теллур, халькогенид, давление пара, вакуум, штейн, термодинамика, распределение.

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Metallurgy

Determination of the quality of special coke as a result of heat treatment of coal from the Shubarkol field

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ABSTRACT

To date, there is a tendency to increase the pace of production in the field of ferrous metallurgy. The constant demand for steel products is accompanied by an increase in prices for raw materials, including carbon reducing agents. In the conditions of the domestic market of Kazakhstan, of great interest is the study and the possibility of using low-baking and non-baking coal as a raw material for the production of special coke used as a reducing agent in metallurgy, the relevance and expediency of which is also due to the resource conservation and energy efficiency program in the use of raw materials put forward by the Government of the Republic of Kazakhstan. In this article, as a result of the search for high-quality, alternative types of reducing agents used in the production of ferroalloys, experimental data of thermal oxidation treatment (coking) of long-flame, non-baking coals of the Shubarkol deposit (Kazakhstan) are presented. In laboratory conditions, during the experiments, the tested grade D coals with a fraction of 70-80 mm were subjected to temperature exposure at temperatures of 800, 850, 900, 950 °C with various preset heating speeds to determine the quality characteristics that meet the requirements for reducing agents for the metallurgical industry, in particular for the production of ferroalloys, in electro thermal, steelmaking, for agglomeration of iron and non-ferrous ores, etc. A technical analysis of long–flame coal was carried out, the volatile and moisture content of which are Vdaf – 44.5%, W – 14.8%, respectively. Also, the obtained special coke was evaluated by the content of volatile components as a result of heat treatment of coals from the Shubarkol deposit: the volatile content averaged 1.73-3.15%, the moisture was 0.73-1.65%.Based on the results of the studies, the possibility of obtaining a special coke from these types of coals with appropriate characteristics was shown.

Keywords: coals, reducing agent, special coke, coke, ferroalloy production, long-flame.

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Introduction

The use of new (alternative) carbon reducing agents from non–baking and low-baking coals for the production of ferroalloys is relevant today - this measure allows partially or completely eliminating the use of expensive traditional coke [[1], [2], [3], [4]].To use alternative reducing agents in the production of metals, it is necessary to meet the requirements of consumers in terms of technical and chemical compositions, electrical resistivity, reactivity, porosity, durability, etc. [[5], [6]].

The reduction of metals by carbon at high temperatures has been called carbon thermal reduction. Coke nut, anthracite, and special coke are used as carbon-containing reducing agents in the production of ferroalloys.

A feature of the carbon-thermal reduction of metals is the formation of carbides, which leads to the production of carbon-saturated alloys. Therefore, such reduction is used in cases where there are no restrictions on the content of carbon in the ferroalloy or its decarburization is carried out. Reduction of metals from oxides by carbon and

CO can be represented by the following reactions [1]:

$$Me_x O_y + yC = xMe + yCO \quad (1)$$

$$2Me_x O_y + yC = 2xMe + yCO_2 \quad (2)$$

$$Me_x O_y + yCO = xMe + yCO_2 \quad (3)$$

Thus, the reducing agent plays a crucial role in the production of ferroalloys.

The use of coal from the Shubarkol deposit for the production of coke is one of the examples of the use of these reducing agents in the production of ferroalloys [[7], [8], [9], [10], [11], [12]]. Coals are characterized by low ash content, as well as low sulfur and phosphorus content. The quality characteristics of this coal are presented in Table 1 [[13], [14], [15], [16]].

The special coke obtained from Shubarkol coal has a low ash content (8-10%), an average sulfur content (0.26%) and phosphorus (0.015%), also has a high reactivity for CO_2 (4.61 cm^3 / g·s), has a high resistivity (33.0 Ohm·cm), porosity is P = 62%, [[2], [3], [4], [5]].The technical characteristics of the special box are shown in Table 2 [[17], [18], [19]].

Table 1 – Characteristics of the quality of Shubarkol coal

Indicators	Unitsof measurement	Content
Workingmoisture– W^p	%	13.2
Analyticalmoisture– W^a	%	4.6
Yield of volatile substances of the analytical sample – V^a	%	39.8
The release of volatiles to a dry ash-free mass V^{daf}	%	44.1
Carboncontent– C^a	%	65.0
Ash content of the analytical sample – A^a	%	5.2
Sulfur– S	%	0.46
Lower heat of combustion Q^r_i	kJ/kg	23700

Table 2 – Technical characteristics of special coke

Material	Technicalcomposition,%		
	Ash	*Volatile*	*Moisture*
Special coke	8 – 10	15 – 25	12 – 15

The resulting special coke meets the basic requirements for carbon reducing agents for the ferroalloy industry and is successfully used at such enterprises as branches of TNC Kazchrome JSC Aktobe and Aksu ferroalloy plants.

Since this type of coal belongs to non-baking long-flame coals of the D brand, the production of special coke in conventional coke batteries is impossible, therefore, a thermal oxidation method of coking on grate grates is recommended [[18], [19], [20]].

Despite the large number of scientific works carried out in the field of production of special coke from non-baking coals of Kazakhstan, there is incomplete information about the features of the pyrolysis process of this type of coal and the dependence on the physical and physicochemical parameters of the special coke and on the parameters of coking [[17], [18], [19], [20], [21], [22], [23]].

For example, the increased humidity of the special coke has a negative effect, leading to a violation of the technological and electrical modes of the furnace by reducing the accuracy of the dosage of reducing agents. The high volatile content also has a negative impact on the technological process, in particular, leading to an increase in temperature and electrical conductivity, sintering of the charge and low fit of the electrodes. According to VUKhIN'sresearch, in order to produce a special coke for ferroalloy production, the volatile yield should be 8-9% to ensure the absence of resinous substances, which lead not only to sintering of charge materials, but to clogging of the reducing agent pores during pyrolysis, contributing to a decrease in the reaction surface and reactivity [4].

In turn, the stable content of humidity and volatile parameters contribute to an increase in the electrical resistance and reactivity of the special coke [[1], [5], [18], [19], [20]].

The experimental part

To assess the quality of the obtained special coke, laboratory experiments were carried out on thermo-oxidative coking of long-flame coal at various temperatures. Fractionated coals of the Shubarkol deposit were considered as the test material.

The purpose of this experiment was to study the quality of the special coke in terms of volatile content and humidity as a result of heat treatment.

To achieve this goal, the following tasks were defined:

- to carry out technical analysis of volatile substances and humidity of coal and special coke;

- to determine the change in the volatile content in long-flame coal during heat treatment at temperatures of 800, 850, 900 and 950 °C.

The main equipment used was a muffle electric furnace "SNOL", designed for analytical work with various materials and various types of heat treatment at temperatures up to 1300 °C. Temperature regulation and control is carried out by an electronic microprocessor-based thermostat working in conjunction with a thermocouple. The experimental program provided for heat treatment in accordance with a given heating of 800, 850, 900 and 950 °C with different heating speeds.

For the experiments, 12 groups of lump coal with a fraction of 70-80 mm were selected (Figure 1), 4 pieces of the starting material (three samples for each temperature level).

The choice of the 70-80 mm fraction was made on the basis of studies conducted on medium-temperature coking of long-flame coals [24].

The technical analysis carried out in accordance with GOST 10742-71 showed that the yield of volatile substances of the initial coal (V^{daf}) was 44.5%, humidity (W) – 14.8%.

Figure 1 – Coals of the Shubarkol deposit

In accordance with the specified heating, each group of fractional coal was placed in a furnace for heat treatment. Visual observation of changes in the shape of the sample was carried out at 300-500 °C and a given maximum temperature, as well as changes during cooling.

Cooling for each of the two groups was carried out to 600 °C and 400 °C, followed by immersion in water at 40 °C and natural cooling to room temperature for each third group. In the obtained samples, after cooling, the content of volatile substances and humidity were measured.

Figures 2-5 show the temperature dependences of the heat treatment of samples with specified heating modes up to temperatures of 800, 850, 900, 950 °C, to assess the behavior of samples under specified modes.

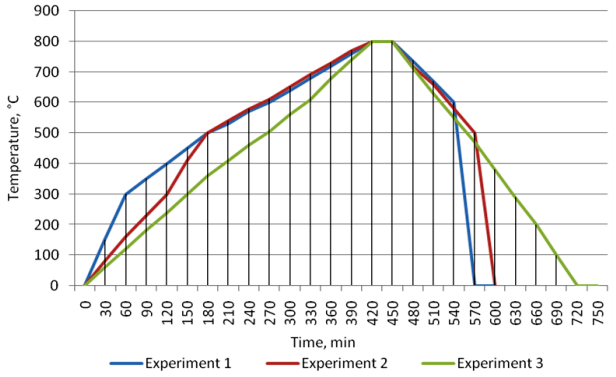


Figure 2 – Temperature dependence of heat treatment of samples up to 800 °C with 30 min exposure and cooling, experiments 1-3

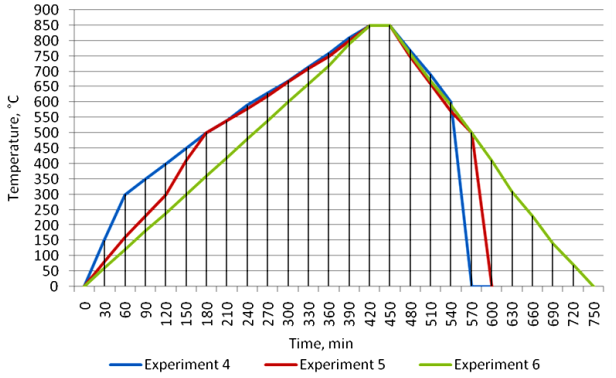


Figure 3 – Temperature dependence of thermal treatment of samples up to 850 °C with 30 min exposure and cooling, experiments 4-6

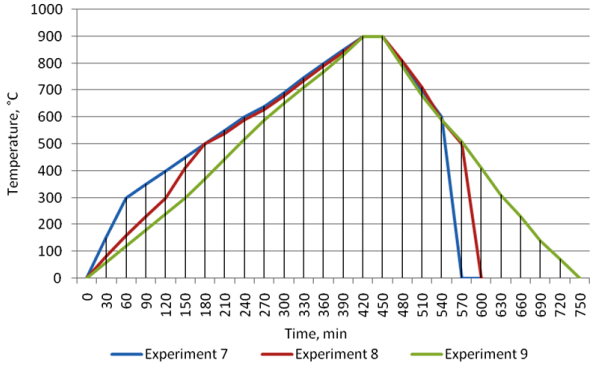


Figure 4 – Temperature dependence of heat treatment of samples up to 900 °C with 30 min exposure and cooling, experiments 7-9

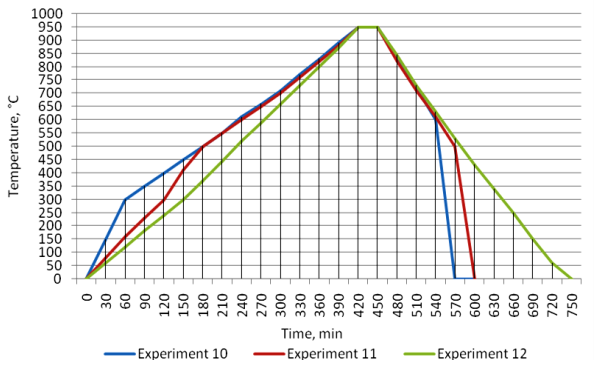


Figure 5 – Temperature dependence of heat treatment of samples up to 950 °C with 30 min exposure and cooling, experiments 10-12

According to the results of the data obtained during visual observation, heating of the samples to a temperature of 800 and 850 °C does not lead to a significant change in the shape of the samples and their destruction: there is a decrease in size, the formation of pores and microcracks.

At 900 and 950 °C, a slight decrease in indicators is observed: fractures and bevels are formed, a decrease in the size of samples with increased ash formation on the entire surface of the samples is noted.

Table 3 – Heating modes of the conducted experiments

Sample heating mode up to 800 °C		
Experiment1	***Experiment2***	***Experiment3***
heating up to 300 °C, speed 5 °C/min	heating up to 300 °C, speed 2.5 °C/min	heating up to 800 °C, speed 1.9 °C/min
heating from 300-500 °C, speed 4.2 °C/min	heating from 300-500 °C, speed 8.3 °C/min	
heating from 500 to 800 °C, speed 3.3 °C/min	heating from 500 to 800 °C, speed 3.3 °C/min	
cooling up to 600 °C, cooling in water at 40 °C	cooling up to 400 °C, cooling in water at 40 °C	naturalcooling
Sample heating mode up to 850 °C		
Experiment4	***Experiment5***	***Experiment6***
heating up to 300 °C, speed 5 °C/min	heating up to 300 °C, speed 2.5 °C/min	sample heating up to 850 °C at a heating rate of 2.02 °C/min
heating from 300-500 °C, speed 4.2 °C/min	heating from 300-500 °C, speed 8.3 °C/min	
heating from 500 to 800 °C, speed 3.3 °C/min	heating from 500 to 800 °C, speed 3.5 °C/min	
cooling up to 600 °C, cooling in water at 40 °C	cooling up to 400 °C, cooling in water at 40 °C	naturalcooling
Sample heating mode up to 900 °C		
Experiment7	***Experiment8***	***Experiment9***
heating up to 300 °C, speed 5 °C/min	heating up to 300 °C, speed 2.5 °C/min	sample heating up to 900 °C at a heating rate of 2.14 °C/min
heating up to 500 °C, speed 4.2 °C/min	heating up to 500 °C, speed of 8.3 °C/min	
heating from 500 to 900 °C, speed 3.75 °C/min	heating from 500 to 850 °C, speed of 3.75 °C/min	
cooling to 600 °C, further cooling in water at 40 °C	cooling to 400 °C, cooling in water at 40 °C	naturalcooling
Sample heating mode up to 950 °C		
Experiment10	***Experiment11***	***Experiment12***
heating up to 300 °C, speed 5 °C/min	heating up to 300 °C, speed 2.5 °C/min	sample heating up to 950 °C at a heating rate of 2.26 °C/min
heating up to 500 °C, speed 4.2 °C/min	heating up to 500 °C, speed 8.3 °C/min	
heating from 500 to 950 °C, speed 3.96 °C/min	heating from 500 to 950 °C, speed 3.95 °C/min	
cooling up to 600 °C, cooling in water at 40 °C	cooling to 400 °C, cooling in water at 40 °C	naturalcooling

Table 3 shows the values of the heating modes of the samples with the specified heating rates of all 12 experiments conducted.

Discussion of the results

1) Coking of coals to a temperature of 800 °C.

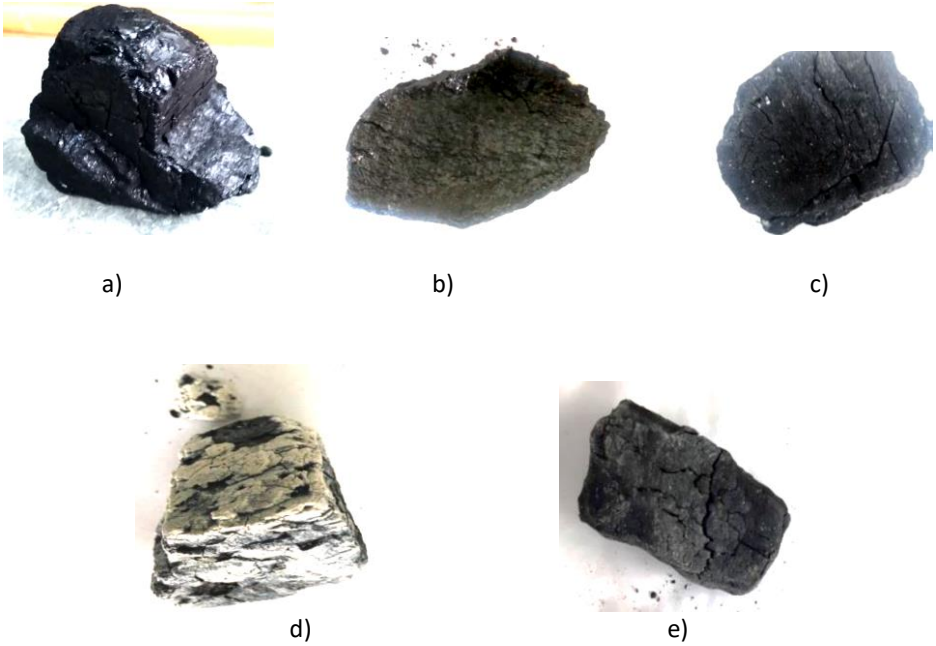
As a result of heat treatment, the obtained samples retained their original shape: up to a temperature of 300 °C, an endothermic reaction occurs with the absorption of heat and the formation of microcracks and micropores, then there is an abundant release of heat and gases, which affects the reduction in the size of coal, pores and cracks expand and increase. After slow cooling, there is a small ash content on the surface of the coke, which is washed off after cooling with water with the appearance of characteristic pigment spots in the places of salinity.

Figure 6 shows a photo image of the samples at different heating rates.

a) b) c) d)

a) heating up to 300 °C; b) heating up to 500°C; c) heating up to 800°C; d) cooling

Figure 6 – Coal samples as a result of the conducted experiment when heated to 800°C



a) b) c)

d) e)

a) heating up to 300 °C; b) heating up to 500 °C; c) heating up to 850 °C;
d) natural cooling to 400 °C; e) water cooling at 40 °C

Figure 7 – Coal samples as a result of experiments 4-6

2) Coking of coals to a temperature of 850 °C.

Heating up to 300 °C is accompanied by heat absorption with the formation of microcracks, the shape and characteristic luster of coal is preserved.

Further heating up to 600 °C is accompanied by gas release, an increase in the size and number of cracks and pores.

Further heating to 850 °C – the samples retained their shape, there is a slight decrease in size, and thereare no gas emissions. As a result of natural cooling, ash formation, microcracks, pores are observed on the surface; after cooling, characteristic spots and small bevels are present in the water at the sites of salinization.

The samples obtained are shown in Figure 7.

3) Coking of coals to a temperature of 900 °C.

Heating up to 300 °C is accompanied by the formation of small cracks, the samples have retained their shape, and there are no destructions, chips. Further heating up to 500 °C is accompanied by strong gas emission, an increase in the number of microcracks and pores.

When the temperature reaches 900 °C, the samples retained their shape, the surface is covered with deep microcracks, chips, and there are no gas emissions.

As a result of natural cooling, ash, microcracks, pores are present on the surface, there is an increase in bevels, samples have less strength; after cooling in water, and characteristic spots are present at the sites of salinization. The samples obtained are shown in Figure 8.

4) Coking of coals to a temperature of 950 °C.

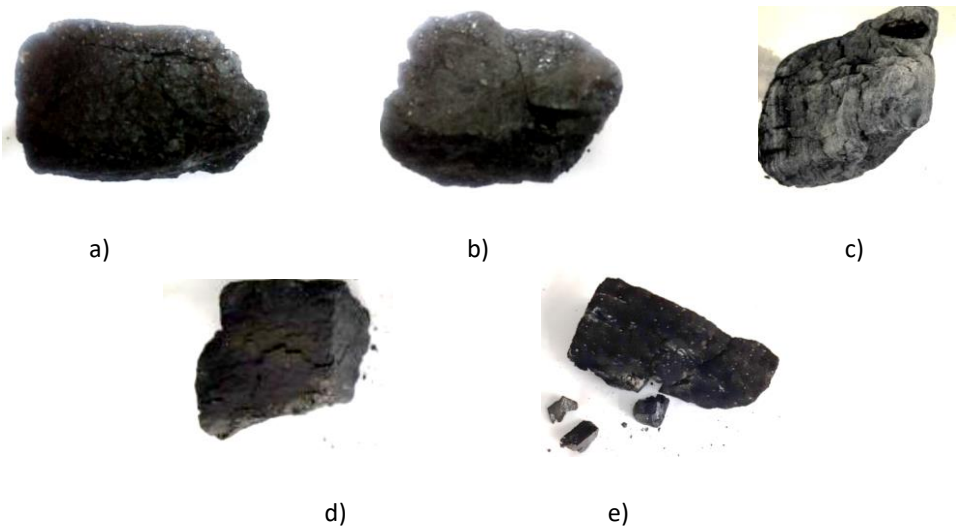
The samples obtained are shown in Figure 9.

Heating up to 300 °C is accompanied by the formation of small cracks, the samples have retained their shape, and there is no destruction, no chips. Further heating up to 500 °C is accompanied by strong gas emission, an increase in the number of microcracks and pores.

When the temperature reaches 950 °C, the destruction of samples is observed, the surface is covered with deep microcracks, chips, and there are no gas emissions.

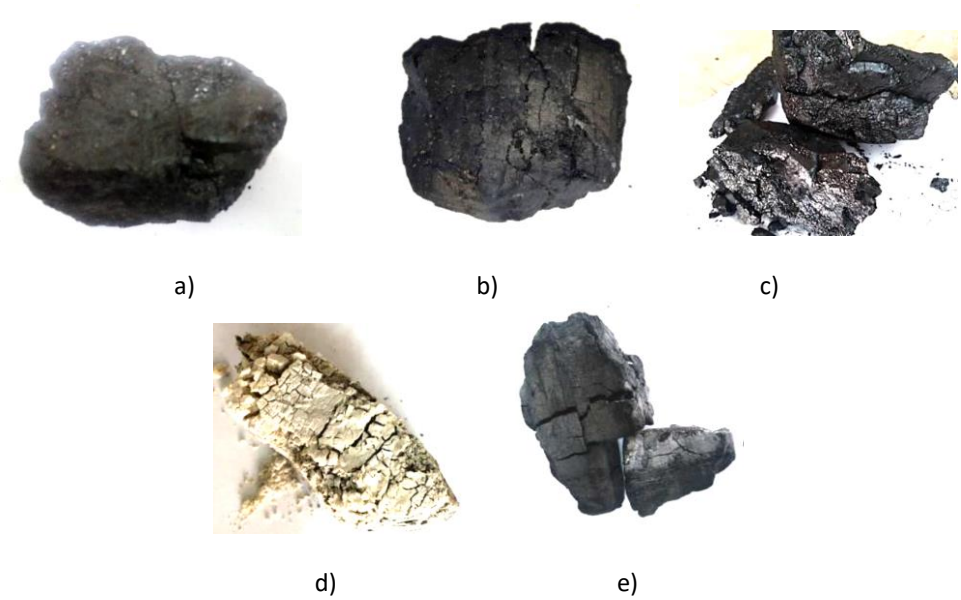
As a result of natural cooling, ash, microcracks, pores are present on the surface; after cooling, characteristic spots are present in the water at the sites of salinization, the formation and increase in the size of bevels is observed, the destruction of samples is observed.

Data on the volatile content and humidity of the samples obtained from all the experiments are presented in Table 4.



a) heating up to 300 °C; b) heating up to 500 °C; c) at 900°C; d) natural cooling; e) water cooling

Figure 8 – Coal samples as a result of the conducted experiment when heated to 900°C



a) b) c)

d) e)

a) heating up to 300 °C; b) at 400 °C; c) at 750 °C; d) natural cooling; e) water cooling

Figure 9 – Coal samples as a result of the conducted experiment

Table 4 – Data on volatile content and humidity

Core size class, mm	Cooling after removal from the furnace at 600 °C.		Cooling after removal from the furnace at 400°C		Cooling to room temperature	
	Experiment1		***Experiment2***		***Experiment3***	
	V	W	V	W	V	W
Inner core 0-5 mm	3.11	1.54	2.72	1.23	2.23	1.10
Inner core 5-10mm	3.19	1.39	2.54	1.88	2.30	1.18
Outer part 10-25 mm	3.04	1.26	2.45	1.91	3.03	1.20
Outer part 25-40mm	3.27	1.28	2.61	1.59	3.06	1.15
Average per piece	3.15	1.37	2.58	1.65	2.65	1.16
	Experiment4		***Experiment5***		***Experiment6***	
Inner core 0-5 mm	2.91	1.53	2.63	1.36	2.19	0.97
Inner core 5-10mm	2.97	1.40	2.51	1.48	2.27	1.01
Outer part 10-25 mm	2.99	1.28	2.42	1.61	2.61	1.12
Outer part 25-40mm	3.01	1.27	2.56	1.57	2.72	1.05
Average per piece	2.97	1.37	2.53	1.50	2.45	1.04
	Experiment7		***Experiment8***		***Experiment9***	
Inner core 0-5 mm	2.83	1.44	2.53	1.37	2.01	0.83
Inner core 5-10mm	2.85	1.49	2.43	1.45	1.97	0.87
Outer part 10-25 mm	2.73	1.41	2.37	1.53	1.85	0.91
Outer part 25-40mm	2.91	1.47	2.44	1.49	1.73	0.99
Average per piece	2.83	1.45	2.44	1.46	1.89	0.90
	Experiment10		***Experiment11***		***Experiment12***	
Inner core 0-5 mm	2.21	1.42	1.97	1.40	1.85	0.75
Inner core 5-10mm	2.15	1.44	2.01	1.45	1.79	0.78
Outer part 10-25 mm	2.30	1.38	1.91	1.52	1.65	0.73
Outer part 25-40mm	2.31	1.41	1.85	1.51	1.61	0.81
Average per piece	2.24	1.41	1.93	1.47	1.73	0.77

According to Table 4, as a result of thermal exposure to the coals of the Shubarkol deposit, the samples have the required humidity and volatile values required for reducing agents.

In experiments 6, 9, 12, a decrease in humidity indicators is observed due to the high rate of coking.

At the same time, when temperatures reach 900 and 950 °C, there is a decrease in the size of the pieces and their integrity, the formation of bevels and, according to visual signs, strength.

Conclusions

In this work, experiments were carried out to assess the quality of non-baking coals of the Shubarkol deposit, and the influence of temperature conditions on the quality of samples of special coke obtained in laboratory conditions, used as reducing agents for metallurgical production, was investigated.

In total, 12 experiments of thermal exposure to long-flame coals of the Shubarkol deposit were carried out in the temperature range up to 800...950 °C with different preset heating speeds.

s a result, the following results were obtained:

- technical analysis of long–flame coal with a grain size of 70-80 mm, volatile V^{daf} – 44.5%, humidity W – 14.8 %;

- samples were obtained as a result of heat treatment up to 800, 850, 900 and 950 °C of long-flame coals with a size of 70-80 mm;

- the assessment of the special coke on the content of volatile components as a result of heat treatment of coals of the Shubarkol deposit was carried out: the content of volatile on average is 1.73-3.15%, the humidity was 0.73-1.65%.

Based on the obtained research data, the following coking mode is recommended:

- coal size 70-80 mm;
- coking temperature800-850 °C;

The evaluation of the obtained results proves the effectiveness of using long-flame coal from the Shubarkol deposit as reducing agents for the metallurgical industry, in particular ferroalloy production, electrothermal, steelmaking productions, for agglomeration of iron and non-ferrous ores and other.

Conflict of interest. On behalf of all the authors, the correspondent author declares that there is no conflict of interest.

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Шұбаркөл кен орнының көмірін термиялық өңдеу нәтижесінде арнайы кокстың сапасын анықтау

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ТҮЙІНДЕМЕ

Бүгінгі таңда қара металлургия саласындағы өндіріс қарқынын арттыру үрдісі байқалады. Болат өнімдеріне тұрақты қажеттілік шикізат бағасының, оның ішінде көміртекті тотықсыздандырғыштарға өсуімен қатар жүруде. Қазақстанның ішкі нарығы жағдайында металлургияда тотықсыздандырғыш ретінде қолданылатын арнайы кокс алу үшін шикізат ретінде төмен жентектелген және жентектелмейтін көмірлерді пайдалану мүмкіндігін зерттеу үлкен қызығушылық тудырады, оның өзектілігі мен мақсаттылығы ҚР Үкіметі ұсынған шикізатты пайдаланудағы ресурстарды үнемдеу және энергия тиімділігін арттыру бағдарламасына да байланысты. Бұл мақалада ферроқорытпалар өндірісінде қолданылатын тотықсыздандырғыштардың сапалы, балама түрлерін іздеу нәтижесінде Шұбаркөл кен орнының (Қазақстан) ұзын жалынды, күйежентектелмейтін көмірді термо тотықтырғыш өңдеудің (кокстеудің) тәжірибелік деректері келтірілген. Зертханалық жағдайда, тәжірибелер

жүргізу барысында 70-80 мм фракциясы бар Д маркалы сыналатын көмірлер сапалық сипаттамаларын анықтау үшін әртүрлі берілген қыздыру жылдамдықтарымен 800, 850, 900, 950 °С температурада температуралық әсерге ұшырады металлургия өнеркәсібі үшін, атап айтқанда ферроқорытпалар өндірісі үшін, электротермиялық, болат балқыту өндірісінде, темір және түсті кендерді агломерациялау үшін және т. б. тотықсыздандырғыштарға қойылатын талаптарға сәйкес келеді. Ұзын жалынды тас көмірге техникалық талдау жүргізілді, олардың ұшпа және ылғал мөлшерлері, сәйкесінше Vdaf – 44,5%, W – 14,8% құрайды. Сондай-ақ, Шұбаркөл кен орнының көмірін термиялық өңдеу нәтижесінде ұшпа компоненттердің құрамы бойынша алынған арнайы коксты бағалау жүргізілді: ұшқыштардың құрамы орта есеппен 1,73-3,15%, ылғалдылығы 0,73-1,65% құрады. Жүргізілген зерттеулердің нәтижелері негізінде тиісті сипаттамалары бар көмірдің аталған түрлерінен арнайы кокс алу мүмкіндігі көрсетілді.

Түйін сөздер:көмір, көмір, тотықсыздандырғыш, арнайы кокс, кокс, ферроқорытпа өндірісі, ұзын жалынды.

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Опытное определение качества спецкокса в результате термической обработки углей Шубаркольского месторождения

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АННОТАЦИЯ

На сегодняшний день наблюдается тенденция наращивания темпа производства в области черной металлургии. Постоянная потребность на стальную продукцию сопровождается с ростом цен на сырье, в том числе и на углеродистые восстановители. В условиях внутреннего рынка Казахстана большой интерес представляет изучение и возможность использования слабоспекающихся и неспекающихся углей в качестве сырьевого материала для производства спецкокса, использующегося в качестве восстановителя в металлургии, актуальность и целесообразность которого также обусловлена в рамках программы ресурсосбережения и энергоэффективности в использовании сырьевых ресурсов, выдвигаемых Правительством РК. В данной статье, в результате поиска качественных, альтернативных видов восстановителей, применяющихся в производстве ферросплавов, представлены экспериментальные данныетермоокислительной обработки (коксования) длиннопламенных, неспекающихся углей Шубаркольского месторождения (Казахстан). В лабораторных условиях, в процессе проведения опытов, испытуемые угли марки Д фракцией 70-80 мм подвергались температурному воздействию при температурах 800, 850, 900, 950 °С с различными заданными скоростями нагрева для определения качественных характеристик, соответствующих требованиям к восстановителям для металлургической промышленности, в частности для производства ферросплавов, в электротермическом, сталеплавильном производстве, для агломерации железных и цветных руд и т.д. Был проведен технический анализ каменного длиннопламенного угля, содержание летучих и влаги которых составляют, соответственно, V_{daf} – 44,5 %, W – 14,8 %. Также, проведена оценка полученного спецкокса по содержанию летучих компонентов в результате термической обработки углей Шубаркольского месторождения: содержание летучих в среднем составляет 1,73-3,15 %, влажность составила 0,73-1,65%. На основании результатов проведенных исследований, была показана возможность получения спецкокса из данных типов углей с соответствующими характеристиками.

Ключевые слова: угли, восстановитель, спецкокс, кокс, производство ферросплавов, длиннопламенный.

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