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ENERGETICS OF ADSORPTION OF WATER MOLECULES TO AEROSIL

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Abstract: The article presents the characteristics of water molecules to aerosil based on thermodynamic laws. During the adsorption process, water molecules adsorbed to aerosil form ionic-molecular complexes with protons in active centers. Also, the adsorption isotherm, differential heat, entropy, and thermal equilibrium time were studied.

Keywords: Aerosil, adsorption, thermodynamics, isotherm, differential heat.

Introduction. Hydrated aerosil is a pure amorphous non-porous silica, consisting of spherical particles of colloidal size and characterized by high values of specific surface area. [1]. When SiO_2 is obtained during the drying stage, the particles initially retain their individuality, and then, under the influence of weak binding forces, they form aggregates of primary monodisperse spheres. [2]. The accumulation of particles is facilitated by the presence of water and hydrogen chloride complexes in the surface layer, which determine the magnitude of the surface charge and the hydrogen index of the aerosil hydrogel. During the drying stage, the acid is removed from the surface of the particles, restoring the hydration shell [3]. This reduces the friction between particles and prevents aggregation [2]. The formation of silanol groups during the aerosilation process also prevents the particles from sticking together during their growth.

The properties of dispersed silica as an active filler and thickener of the dispersion medium are determined by the chemical nature, quantity, and geometric arrangement of active centers in the surface layer. The listed factors determine the energy heterogeneity of the surface.

Existing studies have mainly allowed us to determine the chemical nature of the active sites present on the surface of hydrated aerosil. These include silicon atoms carrying OH groups, extended siloxane bridges, silane-diol groups, coordinatively unsaturated silicon atoms carrying isolated hydroxyl groups, as well as various forms of bound water [3-6]. There are two types of OH groups on the surface of aerosil: free and hydrogen-bonded, the latter increasing the activity of the former by their presence [4,7].

Free single silanol groups are more stable than bound ones and begin to be removed from the surface at temperatures above 873 K [8]. They are less reactive, but the electronic systems of aromatic compounds mainly interact with them [9]. They are also more sensitive in reactions with chlorosilanes [4,7].

The properties of surface hydroxyl groups mentioned above, as well as their small radius and the acidity of hydrogen atoms, determine the possibility of both specific interactions with electron-donor molecules and non-specific interactions with the dispersion medium [9].

The unique interaction of highly dispersed silica with the liquid phase leads to the formation of structures, which significantly improves the quality of many composite systems, in particular varnishes, paints and lubricants. There are known cases where the formation of structures leads to a decrease or loss of technological properties, for example, in unvulcanized rubbers.

To suppress the structure formation processes, aerosil is used, in which the hydroxyl groups are replaced by inert methyl groups that are not capable of specific interactions. Chemical methylation of the surface, improving its uniformity and simultaneously reducing the adsorption potential, changes the nature of the structure formation processes and the deformation of organogels and compositions based on them [10].

Initial attempts at surface modification involved replacing hydroxyl groups with methoxyl groups via esterification reactions [11], as well as hydrophobizing the surface by thermal dehydration. In this case, the structure of the silicon skeleton remains unchanged and the silicon surface becomes organophilic and hydrophobic.

The terms of change are considered in works [12-15]. According to [16], the reactivity and chemical stability of chemisorbed alcohols on the silica surface decrease from primary to tertiary, and in unsaturated alcohols, it depends on the state of the double bond. As a rule, free OH groups, not bound by hydrogen bonds and siloxane bridges, participate in the methoxylation reaction [17], but methyl groups are the only structures capable of reacting with all structural hydroxyls of the silicon surface, even at sufficiently small concentrations of OH groups [18].

Object and methods of research. The composition of the studied zeolite is pyrogenic SiO_2. For water purification, it was passed through a column containing zeolite. Differential molar adsorption-calorimetric studies of water adsorption on aerosil were carried out using the apparatus described in [9,10]. Dissolved gases are removed by freezing the adsorbate, followed by evacuation. The use of the Peltier effect in the heat flux compensation method allowed to significantly increase the accuracy of the adsorption temperature measurements. A calorimeter is capable of measuring the heat released over an unknown period of time. Adsorption measurements were performed using a universal high-vacuum volume setup, which allowed accurate adsorption measurements and adsorbate dosing.

Research results and discussion. The adsorption isotherms of water molecules on Aerosil were studied. The isotherm was measured at 303 K and is shown in Fig. 1. In the range of relative pressures $0.05 < P/Ps < 0.4$, the isotherm follows a linear pattern in the coordinates of the BET equation. For Aerosil containing sodium cations, the monolayer adsorption capacity (a_m) of water molecules is 1.767 μmol/g, and the binding energy constant is 14.32 μmol/g. The specific surface area of water on Aerosil is 162.1 m^2/g and

assuming that water molecules form a dense monolayer, the area occupied by each molecule (ω_m) is 15.8 Å² in sample 3.

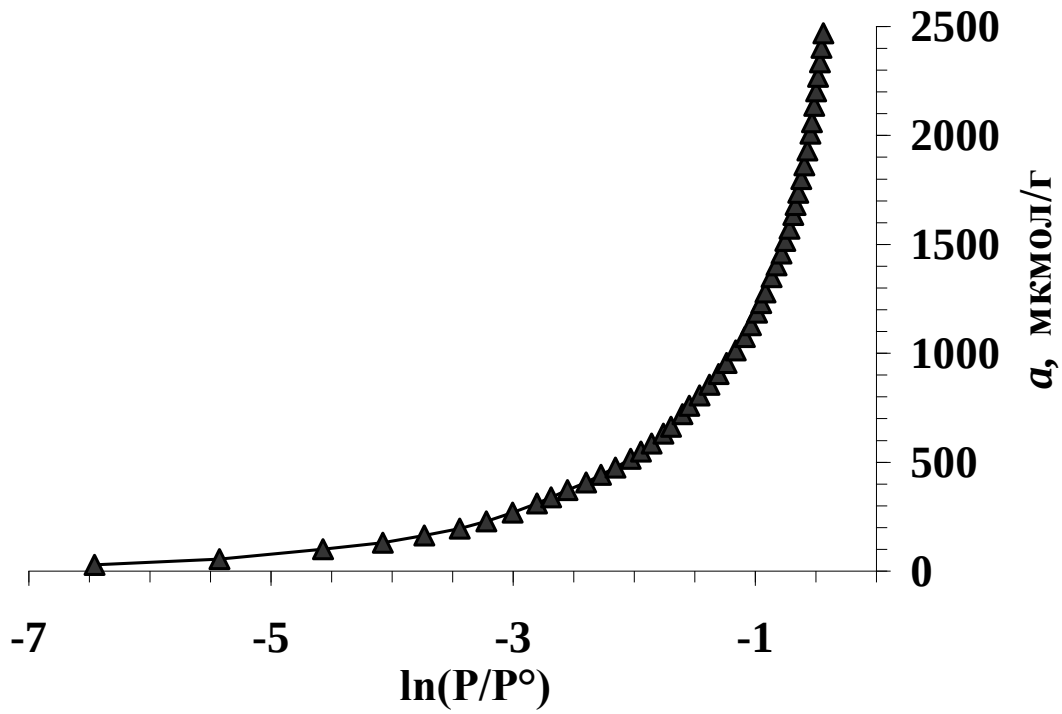

Figure 1. Adsorption isotherms of water molecules to aerosols

The differential heats of adsorption (Qd) for water molecules measured on Aerosil at 303 K are presented in Figure 2. Initially, the heat of adsorption for Aerosil is 69.09 kJ/mol at an adsorption level of approximately 1.20 μmol/g. This change in differential heat is due to the active sites on the Aerosil surface. The activation of these surface sites leads to a decrease in the heat of adsorption, as more water molecules are adsorbed. In all calorimetric tests performed, the differential heat of adsorption of water molecules is gradually observed. The activated and sodium cation-modified Aerosil samples show a higher differential heat of adsorption than the enriched sample. For Aerosil, the first stage shows a decrease in the heat of adsorption from 57.17 kJ/mol to 52.25 kJ/mol, with adsorption values ranging from 49 μmol/g to 62.52 μmol/g. In the second stage, the heat of adsorption decreases to 61.81 kJ/mol, and in the fourth stage, at approximately 348 μmol/g, it decreases again to 50.17 kJ/mol. A second additional stage is observed, leading to a total heat change of 1025 kJ/mol. The first stage corresponds to the adsorption of 20 μmol/g of water in structural defects. Furthermore, each step generates bilayers of 50.1

and ~45.6 kJ/mol, respectively, with a total curve length of 3155 μmol/g. Based on the adsorption data of the water molecule, it is determined that the initial active centers consist of 77 μmol/g of H+ cations. These additional centers are probably cations located at active surface centers similar to those found in clay minerals.

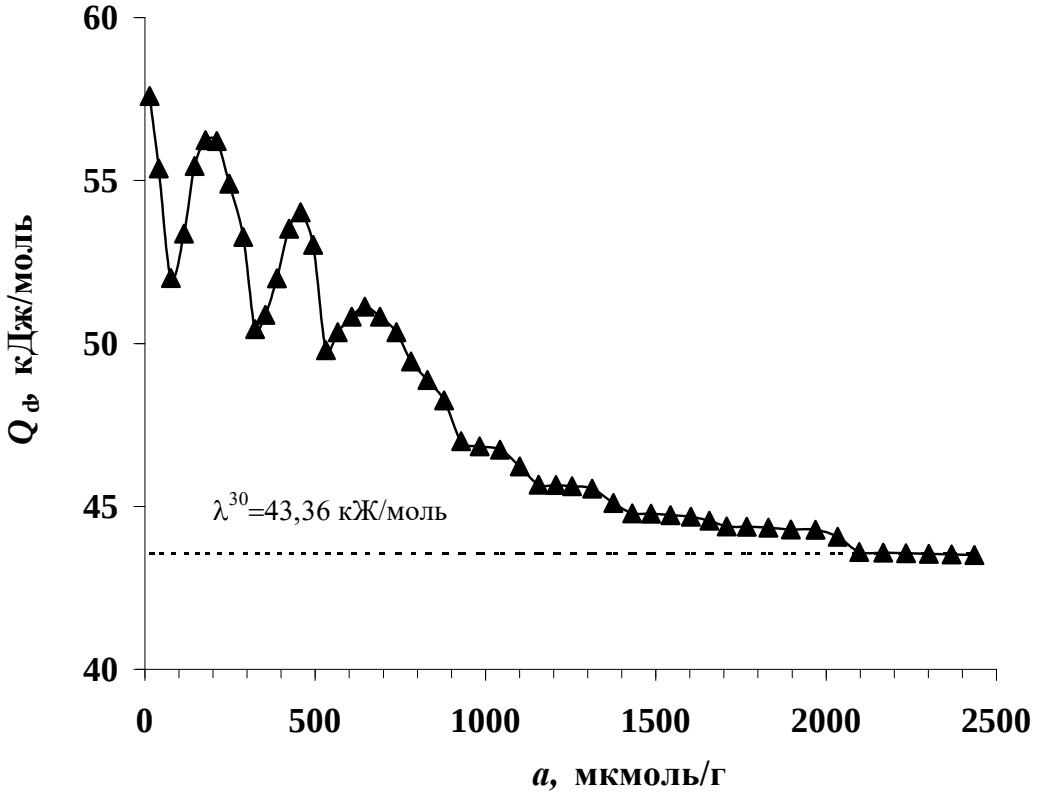

Figure 2. Differential heat of adsorption of water molecules to aerosols. Dashed lines – heat of condensation of water at 303 K

Then the number of H+ cations in the active centers increases to 118 μmol/g calculated from the difference 348-230=118 μmol/g. The number of Na+ cations in the active centers is approximately half of that in the basal surface, i.e. 230:2=115. The experimental and calculated values agree, which supports the hypothesis that 92 H+ cations are adsorbed on the active surface. The sodium cations on the surface exhibit relatively low charges, which, in contrast to muscovite, facilitate the close arrangement of adsorption complexes.

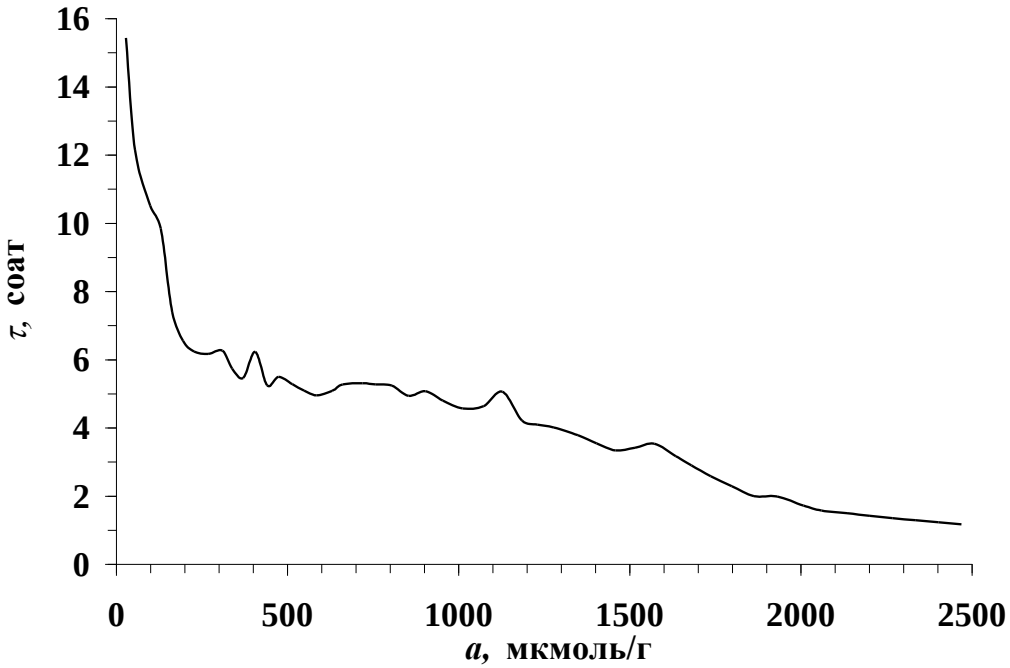

Figure 3. Differential molar entropy of adsorption of water molecules on aerosil. Dashed lines are the average integral entropy. The liquid state entropy of water molecules is assumed to be zero

The relationship between the time required to establish adsorption equilibrium and the surface coverage (Figure 4) shows that the equilibrium time slows down significantly up to about 110 μmol/g. At lower coverages, equilibrium is reached in about 5 hours, while at higher coverages the time decreases to 1–1.5 hours.

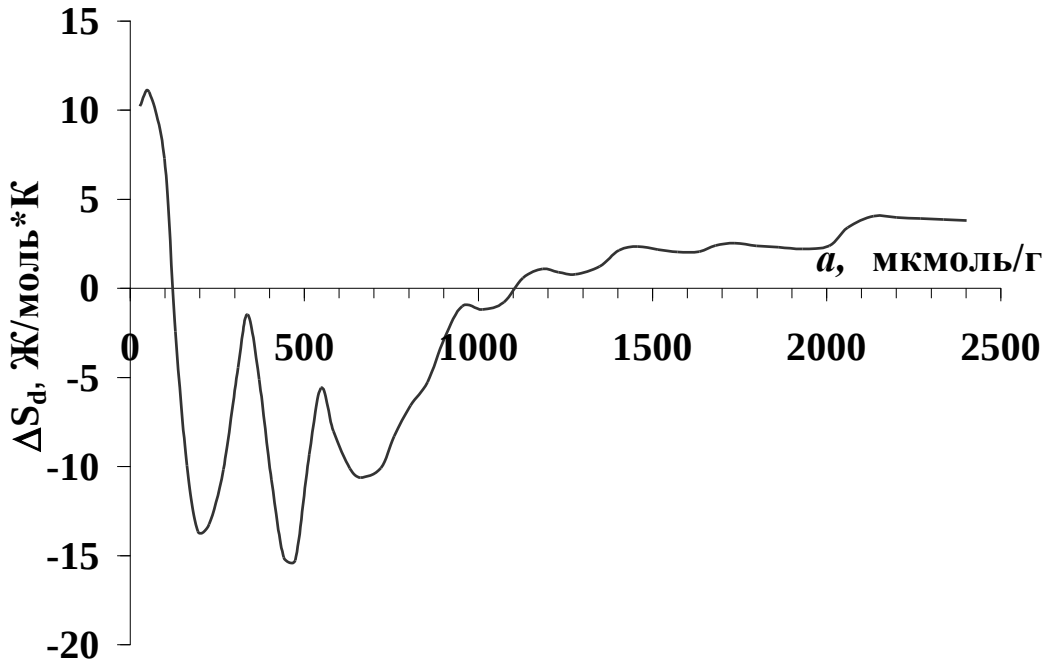

Figure 4. Equilibrium time of adsorption of water molecules on aerosil

This significant difference in the kinetics of equilibrium establishment is due not only to the redistribution of water molecules on the surface, but also to the migration of cations into vacancies at basal and active surface centers.

Conclusion. Silica adsorbents differ from other types of sorbents in their hydrophilic properties. Aerosil is mainly composed of silicon oxide and hydrogen compounds of silicon. The differential heat of water adsorption on aerosil initially occurs with a high temperature. The subsequent heats of adsorption of water molecules occur in the form of wavy steps in the form of minimum and maximum lines. The surface areas of aerosil were determined using the BET equation using the adsorption isotherm values.

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CONTENTS

PRIMARY PROCESSING OF COTTON, TEXTILE AND LIGHT INDUSTRY

Korabayev Sh. From street traffic to space: innovations in autonomous vehicles	**3**
Egamov N. Investigation of vertical forced vibration in the longitudinal - vertical plane of a binder that softens the crush between cotton rows	**10**
Khamraeva S., Kadirova D., Davlatov B. Determination of alternative technological factors for the production of functional fabric with a complex structure	**15**
Khamraeva S., Kadirova D., Daminov A. Designing fabrics for a given stretchability	**21**
Kuliyev T., Rozmetov R., Tuychiev T., Sharipov Kh. The effect of the angle of heat agent supply to the drying - cleaning equipment on cotton quality and cleaning efficiency of the equipment	**28**
Abdujabbarov M., Alieva D., Karimov R. Determination of the influence of the length of the tested yarn samples on their mechanical characteristics	**35**
Jurayeva M., Nabidjonova N. Research on physical and mechanical properties of fabric selected for special clothing of preschool children	**41**
Yangiboev R., Allakulov B., Gulmirzayeva S. Studying the alternative technological factors of the loom in the production of textiles based on basalt yarn	**45**
Ganikhanov Kh., Mavlyanov A., Abdusamatov A., Mirzaumidov A. Analysis of the maintechnologicalparameters of the condenser	**55**
Mavlyanov A., Mirzaumidov A. The scientific basis of the lightened shaft	**60**
Elmanov A., Mirzaumidov A. Modeling of laser processingof thin-walled steel gears	**69**
Nurillaeva Kh., Mirzaumidov A. Cotton cleaner with multifaceted grates	**77**
Ganikhanov Kh., Mavlyanov A., Abdusamatov A., Mirzaumidov A. The equation of motion of cotton fiber in the condenser	**83**
Khuramova Kh., Xoshimxojaev M. Progressive method of cotton regeneration	**89**

Abdukarimova M., Lutfullaev R., Usmanova N., Mahsudov Sh.
Evaluation of aestheticity of women's dress models based on deep learning models **94**

GROWING, STORAGE, PROCESSING AND AGRICULTURAL PRODUCTS AND FOOD TECHNOLOGIES

Zufarov O., Isroilova Sh., Yulchiev A., Serkayev K.
Theoretical aspects of obtaining oxidation-stable vegetable oils **101**

Toshboyeva S., Dadamirzaev M.
Filling sauces for canned fish and their layer kinetics **110**

Atamirzaeva S., Saribaeva D., Kayumova A.
Prospects for the use of rose hips in food technology **115**

Turgunpolatova Sh.
Study of the quality of fruit pastela products **121**

Sultanov S.
Analysis of experiments on the process of deodorization of vegetable oil using floating nozzles **126**

Adashev B.
Physical-chemical analysis of oil taken from seeds of safflower **132**

Ismailov M.
Influence of surface layer thickness on hydraulic resistance of the device **137**

Khurmamatov A., Boyturayev S., Shomansurov F.
Detailed analysis of the physicochemical characteristics of distillate fractions **142**

Madaminova Z., Khamdamov A., Xudayberdiyev A.
Preparing peach seed for oil extraction and improving oil extraction through pressing **154**

Aripova K.
Methods of concentration of fruit juices and their analysis **162**

Djuraev Kh., Urinov Sh.
Theoretical and experimental study of the crack formation device in the shell of apricot kernels **168**

CHEMICAL TECHNOLOGIES

Urinboeva M., Abdikamalova A., Ergashev O., Eshmetov I., Ismadiyarov A.
Study of the composition and main characteristics of petroleum oils and their emulsions **175**

Tursunqulov J., Kutlimurotova N.
Application of 1-(2-hydroxy-1-naphtoazo)-2-naphthol-4-sulfo acid in amperometric determination of scandium ion **182**

Kucharov A. **191**

Development of coal enrichment and gas extraction technology for the use of construction materials industrial enterprises	
Abdulkhaev T., Mukhammadjonov M., Mirzarakhimova F.	
Isotherm of benzene adsorption and differential heat of adsorption on AgZSM-5 zeolite	**198**
Vladimir L., Eshbaeva U., M.Ergashev	
Innovative environmental packaging for separating storage of two components, allowing to extend the lifetime without preservatives	**204**
Kodirov O., Ergashev O.	
Energetics of adsorption of water molecules to aerosol	**212**
Yusupov K., Erkabaev F., Ergashev D., Rakhimov U., Numonov M.	
Synthesis of melamine-formaldehyde resins modified with n-butanol	**219**
Ergashev O., Abdikamalova A., Bakhronov Kh., Askarova D., Xudoyberdiyev N., Mekhmonkhonov M., Xolikov K.	
Thermodynamics of Congo red dye adsorption processes on mineral and carbon adsorbents	**228**
Ergashev O., Maxmudov I.	
Water vapor adsorption isotherm in zeolites regenerated by microwave thermoxidation method	**235**
Jumaeva D., Zaripbaev K., Maxmudov F.	
The elements and oxide content of the chemical composition of the feldspar	**242**
MECHANICS AND ENGINEERING	
Khudoyberdiev U., Izzatillaev J.	
Analysis of research on small wind energy devices	**249**
Atajonova S.	
Mathematical model of system analysis of technological processes in the form of key principles for effective decision-making	**258**
Kuchkarbayev R.	
Mathematical modeling of heat transfer through single-layer and multi-layer cylindrical walls in buildings and structures	**264**
Atambaev D.	
Difference in the length of individual yarn composition of twisted mixed yarn and comparative analysis of single-thread elongation deformations	**269**
Abdullayev S.	
Modeling the functionalities of an automated system for managing movement in the air	**276**
Turakulov A.	
Describing computational domains in applications for solving three-dimensional problems of technological processes	**285**
Mamaxonov A.	

Mathematical model of machine aggregate of tillage equipment process	293
Khudayberdiyev A. Technical and economic aspects of processing pyrolysis distillate into motor fuel	304
Abdurahmonov J. Research results on the selection of the mesh surface of a lint-cleaning device	311
Vohidov M. Development of a program for determining eccentricity by analyzing the magnetic field in the air gap of an asynchronous motor	319
Utaev S., Turaev A. Analysis of methods and prospects for application of optical methods for control of working surfaces of cylinder liners of internal combustion engines	327
Boltabayev B. Determination of seed damage in the pneumatic transport system by conducting experiments	335
Azizov Sh., Usmanov O. Simulation of equation of motion of the new construction gin machine	339
Sharibaev N., Homidov K. Theoretical analysis of the coefficient of friction induced by the pressure force of a vertical rope acting from above and below	347
Aliyev B., Shamshitdinov M. Improvement of the linter machine and development of its working scheme	356
Mukhametshina E. Analysis of cotton flow behavior in different pneumatic pipes	362
Yangiboev R., Allakulov B. Obtaining and analyzing correlational mathematical models of the sizing process	369
Mirzakarimov M. Efficient separation of fibers from saw teeth in the newly designed gin machine	379
Azambayev M. Measures to improve the quality of fluff	387
Abdullayev R. Scientific innovative development of cotton gining	392
Kholmirzaev F. Air flow control factors in pneumatic transport device	397
Sharibaev N., Makhmudov A. Separation of cotton from airflow in pneumatic transport systems of the cotton industry	404
Sharibaev N., Mirzabaev B.	

Effect of steam temperature on yarn moisture regulation in textile industry	**410**
Sultanov S., Salomova M., Mamatkulov O. Increasing the useful surface of the mesh surface	**415**
Muhammedova M. Kinematics of the foot in a healthy person's foot and ankle injury	**421**
ADVANCED PEDAGOGICAL TECHNOLOGIES IN EDUCATION	
Abdullayev H. Algorithm for creating structured diagrams of automatic control systems	**429**
Kodirov D., Ikromjonova N. On delayed technological objects and their characteristics	**437**
Uzokov F. Graphing circles, parabolas, and hyperbolas using second-order linear equations in excel	**444**
ECONOMICAL SCIENCES	
Zulfikarova D. Issues of developing women's entrepreneurship	**449**
Ergashev U., Djurabaev O. Methods for assessing the effectiveness of waste recycling business activities in the environmental sector	**455**