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## NamMTI ILMIY-TEXNIKA JURNALI TAHRIR HAY'ATI A'ZOLARI

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**RESISTANCE AND VOLTAGE ANOMALIES ABOVE 200K  
BSCCO SYNTHESIZED BY SOLAR TECHNOLOGY****GULAMOVA DILBARA**

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**Abstract:** The purpose of the research is to obtain room temperature superconducting phases in ceramics based on bismuth cuprates using solar technology. The synthesis of BSCCO was carried out using the “Solar Fast Alloys Quenchig” technology at the Big Solar Furnace (Parkent). Based on the results of microstructural and X-ray methods, the glass-crystalline composition of the precursors and the homo-phase composition of the nanolayered ceramics were determined. Resistance anomalies in the temperature range of 280-320 K have been established, interpreted in the assumption of room temperature superconducting phases. The Meissner effect in BSCCO precursors and ceramics under daylight and under the influence of light at room temperature and normal atmospheric pressure has been established. Resistance anomalies, the Meissner effect in the assumption of room temperature phases, are explained by the influence of the solar spectrum and technological conditions on the electronic structure of atoms. Different oxygen stoichiometry of homologous phases determines different potentials of adjacent layers, resulting in charge transport and increased charge density at intergranular boundaries.

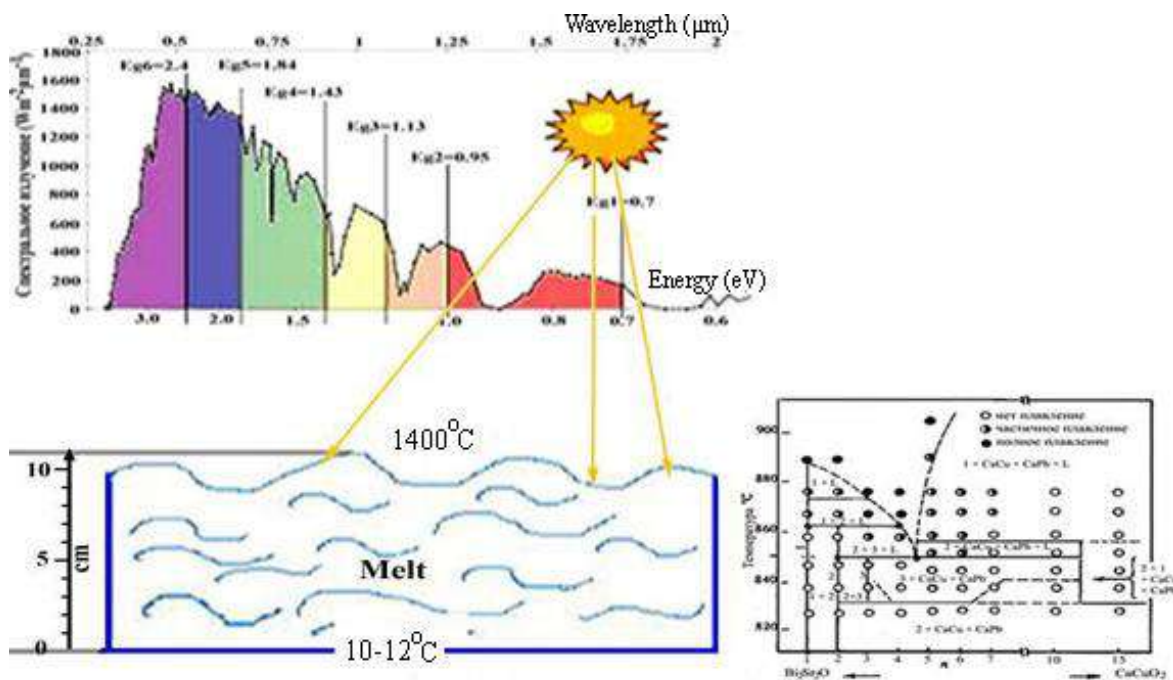
**Keywords:** “SFAQ-T” technology, glass-crystal precursors, nanolayered ceramics, 280-320K resistance anomalies, Meissner effect.

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**Introduction.** The progress of superconductor materials science (HTSC), aimed at finding phases with high superconducting transition temperatures, is largely related to their production technology. The most common methods for synthesizing HTSC are based on the diffusion principle of interaction between initial precursors (the solid-state reaction method), but the granular structure of the target material is a serious disadvantage that limits high current characteristics. Glass-ceramic technology, representing one of the modifications of the “melting method” is carried out by various technical means. Heating sources are resistance furnaces, laser, microwave heating, radiant flux from xenon and halogen lamps, solar flux, and others [1-10]. Melting is carried out in platinum, corundum, ZrO<sub>2</sub>, quartz crucibles, most often under isothermal conditions. Hardening of the melt is carried out between metal plates, by spinning, dispersion, centrifugal, and other methods. [11]. Using the recently developed Solar Fast

Alloys Quenching-T (SFAQ-T) technology [12, 13], long-term wear-resistant plate-textured superconductors of the  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{(n-1)}\text{Cu}_n\text{O}_y$  series ( $n=3-30$ ) with a critical transition temperature of  $T_c=150-295\text{K}$  were obtained [14, 15]. The technology for obtaining Bi/Pb HTSC using concentrated solar energy is based on the assumption of the influence of 2 factors. The first is the possibility of resonant excitation of each atom of the system as a result of the absorption of a certain wavelength of the solar spectrum, which coincides (or is close) to the period of oscillation of the valence electrons of this atom. Consequently, the change in the energy of interatomic bonds and the organization of bonds with "super-stechometric" oxygen occurs, because during the melting process with solar energy, an excess amount of oxygen dissolves in the melt compared to the stochometric amount [16]. Because each individual atom of the system cannot "organize" an additional bond with "super-stechometric" oxygen, this can be achieved by a group of atoms, each of which has additional energy as a result of interaction with the "corresponding" region of the solar spectrum. Therefore, the target material contains "excess" oxygen and high-valence states of cations, particularly  $\text{Cu}^{2+}$ , are possible, which is favorable for superconductivity [17]. The second factor is the sharp temperature gradient during melting and tempering. The gradient leads to the vector orientation of the germ crystals in the direction of the falling solar flux, and sharp quenching determines the formation of a glass-like phase, which is characteristic of glass-forming oxides. Precursors contain nano-sized crystallites-embryos as a base, on which, during subsequent heat treatment of ceramics, as a result of the reaction between the crystallites and the glass phase, HTSC phases are formed and a dense layered structure is formed [18]. As a result of the influence of concentrated solar radiation on ceramics of the series  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{(n-1)}\text{Cu}_n\text{O}_y$ ,  $n=3-20$ , high-temperature superconducting phases with  $T_c=200-295\text{K}$  were obtained [19]. Further work was dedicated to increasing the volume content of high-temperature superconducting phases in ceramics of this series, the results of which we present in this article.

**Preparation of samples and research methods.** Precursors of the nominal composition  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{29}\text{Cu}_{30}\text{O}_y$  were prepared from a mixture of powders:  $\text{Bi}_2\text{O}_3$ ,  $\text{PbO}$ ,  $\text{SrCO}_3$ ,  $\text{CaO}$ ,  $\text{CuO}$  of the "high-purity" brand using the "Super Fast Alloys Quenching technology (SFAQ-T)" technology at the Big Solar Furnace (Parkent) [16]. The diagram (Figure 1) shows the use of phase composition data for the  $\text{Bi}_2\text{Sr}_2\text{O}-\text{CaCuO}_2$  [20] phase diagram section to compare with the possible phase composition for the melt thickness of 5.4-5.8mm. This approach allowed for an approximate assessment of the phase composition based on the thickness of the ingot. The most probable formation of the 2201 phase was assumed in the upper part of the ingot crystallizing from the melt. The most favorable temperatures for the formation of embryos of the low-temperature phases 2201 and 2223 could be in the part adjacent to the water-cooled surface of the substrate (with subsequent peritectic reaction). The particles obtained by sharp cooling of the melt as a result of dispersion had different forms and thicknesses. Ceramic samples were made from precursors using solid-state technology.



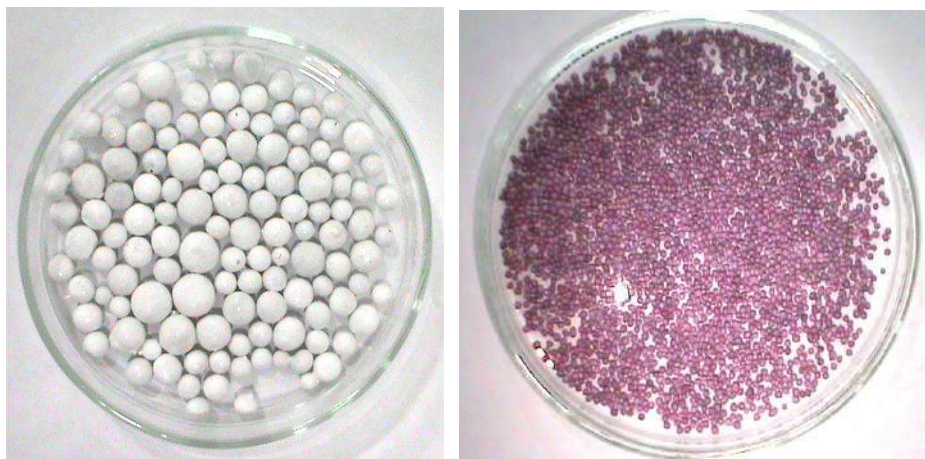
**Figure 1.** Diagram of comparing the temperature gradient in the melt bath with the phase composition of the Bi system  $2Sr_2O-CaCuO_2$

For firing, the pressed discs and beams were placed on corundum microspheres filling a 380ml corundum crucible, as shown in Figure 2. This method of “suspension” the sample eliminated the leakage of the liquid phase, which occurs when the presses are placed on the flat surface of the ceramic substrate.



**Figure 2.** The samples, pressed into a disk and beam, were placed in a crucible filled with corundum spheres

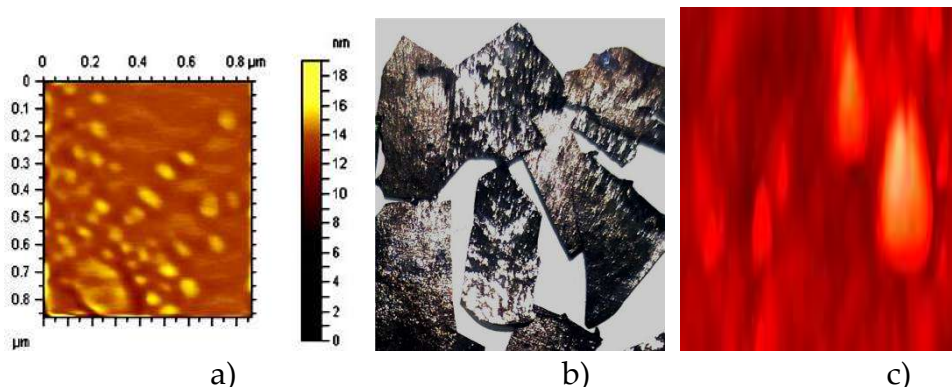
Furthermore, the possible influence of the radiation spectrum of corundum microspheres, different from the radiation of the insulation in the furnace chamber, and the heat flux directed perpendicular to the surface of the samples created a temperature gradient during the heat treatment of the samples (Figure 3).



**Figure 3.** Corundum spheres obtained in a solar furnace

Microstructure analysis was performed using the “SOLVER NEXT” (NT-MDT) scanning probe microscope and the “ZEISS” electron microscope. The phase composition was determined by analyzing diffractograms obtained on the “DRON-UM1” diffractometer, XRD-6100; Cu,K $\alpha$ -radiation, Ni-filter). The resistance of the ceramics was investigated by the 4-contact method on samples - beams measuring 21x5.2x3mm. and samples-discs with a diameter of 12-26mm, thickness of 1.5-5mm. in the temperature range of 80-320K. The contacts were applied by grinding silver paste. The distance between the inner and outer contacts is 5mm, respectively. and 18mm. The connecting wires are silver. Electrical parameters were measured with a digital multimeter “RIGOL DM3058E” (U=±0.015%; I=±0.055%). The temperature was measured by the “chromel-drops” thermocouple, graduation in boiling and cold water, and the “chromel-alumel” control thermocouple. Temperature measurement accuracy is ±2.50° (GOST R8.585-2001). Resistance measurement device and diagram [21]. The voltage was measured according to the method [22].

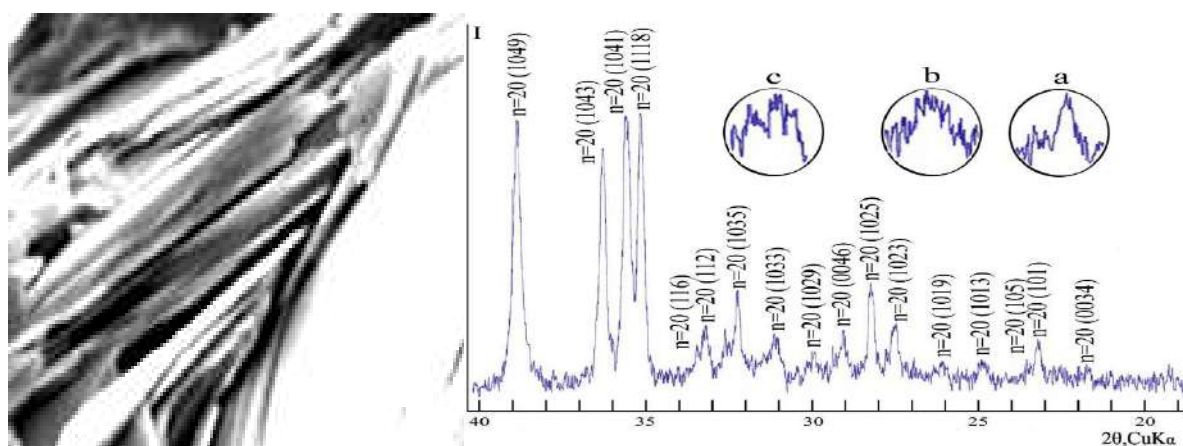
**II. Results and discussion.** Oriented distribution of crystallites in precursors, glass phase in the form of light inclusions, vector orientation of crystallites surrounded by glass phase are shown in Fig. 4a, b, c.



**Figure 4a, b, c.** Precursor plates with oriented distribution of nanocrystallites (a); glass phase inclusions (b); vector-oriented crystallites (c)

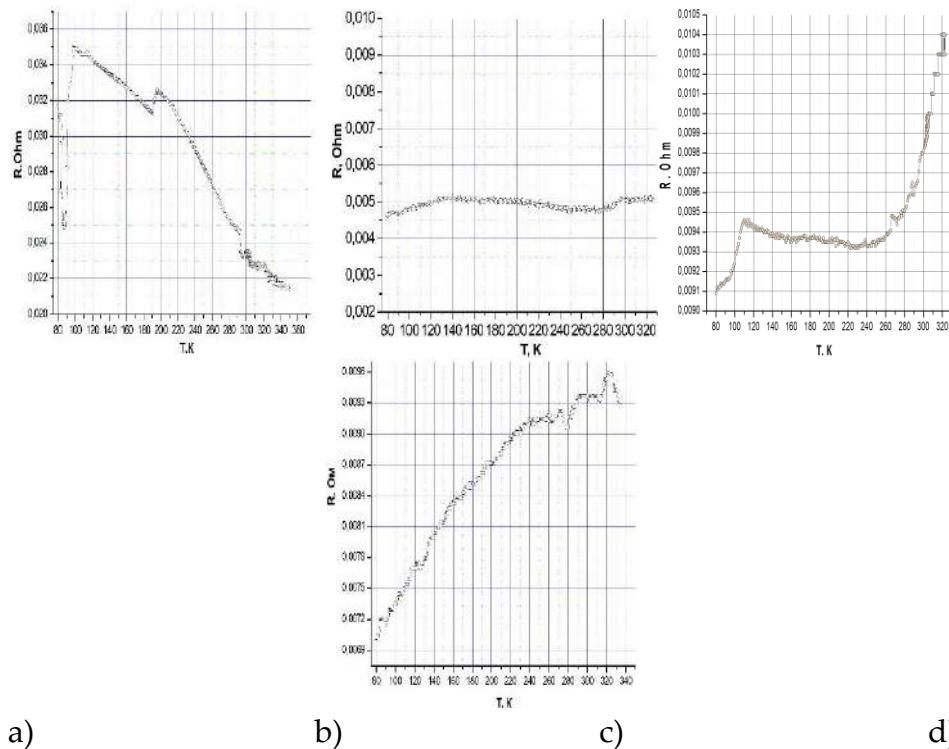
The vector orientation of the germ crystals is formed under the influence of a high temperature gradient ( $\approx 1400-12^\circ\text{C}$ ) between the melt surface and the opposite side located on the water-cooled substrate. The amorphous phase inhibits the growth of germ crystals, which contributes to the preservation of their nano-dimensional parameters.

The firing and cooling of ceramic samples in the temperature gradient field contributed to the formation of an oriented microstructure. The “ZEISS” analysis of the cracking of a ceramic sample - a tablet - perpendicular to the sample plane showed a layered structure consisting of parallel nano-sized plates (Figure 4). Comparison of the oriented microstructure of germ crystals in precursors (Fig. 3c) and ceramics (Fig. 4) shows their relationship. The crystallites of the precursor nano-embryos were centers where, during roasting, reactions between the crystallites and the amorphous phase resulted in the formation of superconducting phase layers [23]. Figure 5 shows a disruption in the parallel arrangement of the layers due to the fragmentation of the sample, but areas with oriented layers are clearly visible. XRD analysis showed the formation of  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{29}\text{Cu}_{30}\text{O}_y$  phase homologues,  $n=2-21$ ,  $n=2-21$ .



**Figure 5.** Microstructure of cracks (a) and XRD data (b) of ceramics with nominal composition  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{29}\text{Cu}_{30}\text{O}_y$

The resistance of ceramic samples obtained at different firing temperatures and times showed a significant dependence of  $R(\text{Ohm})$  and  $U(\text{mV})$  on the firing temperature and time. Samples obtained in the temperature range of  $840-848\text{K}$ , firing time 1-10 hours, had a decrease in  $R(\text{Ohm})$  with increasing temperature, which is characteristic of semiconductors (Fig. 6a). Effects near  $200\text{K}$ ,  $300\text{K}$ ,  $320\text{K}$  can be associated with superconducting phases. An almost linear relationship of  $R(\text{Ohm}) - \text{TK}$  was obtained in ceramics after 44 hours of firing (Figure 5b). After 54 hours of firing, weak effects above  $220\text{K}$  appeared on the curve. The effects at  $270\text{K}$  and  $320\text{K}$  in the sample obtained after 62 hours of firing are of interest (Figure 6d). Comparison of the graphs (6c, d) shows that the “higher phases” are formed in the temperature range of  $840-848^\circ\text{C}$ ,  $\geq 54$  hours.



a) b) c) d)

**Figure 6 a,b,c,d.** Dependence of the resistance of ceramics with the nominal composition  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{29}\text{Cu}_{30}\text{O}_y$ , obtained at  $846^\circ\text{C}$ , 10h (a); 44 hours (b); 54 hours (c); 62 hours (d).

**III. Conclusion.** The presented results confirm the determining influence of the technology on the properties of superconducting materials. The SFAQ-T technology is carried out under conditions of a sharp temperature gradient at all stages from the obtaining of precursors to the synthesis of massive ceramic samples. In the initial stage, during the melting and hardening processes, the conditions-basis for the formation of vector-oriented crystallites-embryos and the amorphous phase are created. The synthesis of ceramics from glass-crystal precursors, also carried out under temperature gradient conditions, determines the formation of superconducting phases-homologues and a layered microstructure with nano-sized intergranular boundaries [24, 25]. Adjacent homologous phases have different compositions, which determines the potential difference between them and, accordingly, the possibility of charge transfer and their concentration at the intergranular boundaries. Such quasi-heterophase ceramics retains properties similar to semiconductors (resistance decrease with increasing temperature) until a series of high-temperature superconducting phases-homologues are formed. After the formation of a multitude of superconducting phases - homologs, the “R-T” relationship changes in the opposite way and corresponds to the superconducting system. In such a system, with an increased content of phases-homologues, the charge concentration at the intergranular boundaries and the current density respectively increase. Resistance anomalies in the room temperature range are presumed to be related to the formation of RTSC phases.

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## C O N T E N T S

---

### TECHNICAL SCIENCES: COTTON, TEXTILE AND LIGHT INDUSTRY

---

<b>Saloxiddinova M.</b>	<b>3</b>
Improving the separator design to prevent cotton fiber loss.	
<b>Juraeva G.</b>	<b>9</b>
Optimizing cotton fiber quality during the production process.	
<b>Mamadaliyev F.</b>	<b>16</b>
Analysis of problem in the aerodynamic system of cottonseed linting equipment in cotton processing plants.	
<b>Kozokov S.</b>	<b>23</b>
Conducting experiments with newly designed saw gin ribs in the cotton cleaning process for different cotton varieties.	
<b>Usmonov I., Abdullajonov S.</b>	<b>30</b>
Methods and results for determining the parameters and operating modes of irradiating watermelon seeds with ultraviolet rays.	
<b>Majidov A.</b>	<b>36</b>
Theoretical foundations of the technological parameters of a straight-flow fiber separation device.	
<b>Rahmatova S.</b>	<b>44</b>
Scientific approach to considering properties in the design of garments made from knitted fabrics.	
<b>Rahmatova S.</b>	<b>48</b>
Technology for obtaining knitted fabrics from various raw materials.	
<b>Turaboyev G.</b>	<b>54</b>
Methodology for determining the tribotechnical properties of structural materials interacting with raw cotton.	

---

### TECHNICAL SCIENCES: AGRICULTURE AND FOOD TECHNOLOGIES

---

<b>Khurmamatov A., Boyturayev S.</b>	<b>58</b>
Results of industrial water treatment from mechanical impurities.	
<b>Khurmamatov A., Alimardonov Kh., Akhmedova K.</b>	<b>65</b>
Two-stage installation for deep air purification from fine-dispersed solid particles.	
<b>Mamatusmonova D., Mamatov Sh.</b>	<b>73</b>
Technical characteristics of the use of vibrating conveyors for drying rosa caninas.	
<b>Toshboyeva S., Dadamirzayev M.</b>	<b>79</b>
Physicochemical properties of a functional sauce for fish canned products.	

---

<b>Saribayeva D., Maxmudova D.</b>	
Study of protein–lipid composition in food products.	<b>83</b>
<b>Gulomkhojaeva N., Zokirova M.</b>	
Study of polyphenolic compounds in jujube ( <i>Ziziphus jujuba</i> mill.) grown in Uzbekistan.	<b>88</b>
<b>Gulomkhojaeva N., Zokirova M.</b>	
Investigation of the amino acid composition in black and white mulberry ( <i>Morus nigra</i> l. and <i>Morus multicaulis</i> perr.) varieties.	<b>94</b>
<b>Kadirov A., Vokosov Z.</b>	
New technology for growing microorganisms of the <i>Bacillus</i> sp, <i>Rhizobium</i> sp, <i>Azotobacter</i> sp.	<b>101</b>
<b>Rakhimova G.</b>	
Development of an effective technology for producing soy milk from local soy raw materials, studying its composition and physical and chemical properties	<b>107</b>

## CHEMICAL SCIENCES

<b>Khabibullaev J., Shomurotov Sh.</b>	
Oxidation of various cellulose containing materials using the $\text{HNO}_3/\text{H}_3\text{PO}_4\text{-NaNO}_2$ system.	<b>112</b>
<b>Nuritdinov A., Abdullaev O.</b>	
Technical parameters and energy efficiency of an oil sludge processing unit	<b>122</b>
<b>Okhundadaev A.</b>	
Study of the effect of various factors on the synthesis of vinyl esters of wine acids	<b>127</b>
<b>Usmonova Z.</b>	
Effectiveness analysis of thermally and steam activated plum seed adsorbents	<b>133</b>
<b>Kaxarova M.</b>	
Technological scheme for extracting naphthalene from pyrolysis oil by the extraction (phase separation) method	<b>139</b>
<b>Oribzhonov M., Bektemirov A., Arislanov A., Azizov V.</b>	
Method for producing biosuperphosphate fertilizers containing humic compounds	<b>143</b>
<b>Erkinov R., Soliyev M., Arislanov A.</b>	
Synthesis of sulfur containing organic compounds by reaction of thiol-en and thiol-in	<b>151</b>
<b>Yusupov M., Nuritdinov A.</b>	
Elemental analysis of carboxyl-modified copper phthalocyanine pigment	<b>156</b>

---

<b>Nuritdinov A.</b>	
Thermal analysis of carboxyl-modified cobalt and calcium metal phthalocyanine pigments	<b>162</b>

---

<b>Isakov B.</b>	
Development and study of an anti-caking additive to improve the physico-mechanical properties of ammonium nitrate	<b>168</b>

---

### TECHNICAL SCIENCES: MECHANICS AND MECHANICAL ENGINEERING

---

<b>Gulamova D., Bobokulov S., Eshonkulov E.</b>	
Resistance and voltage anomalies above 200k bscco synthesized by solar technology	<b>173</b>

---

<b>Kutbidinov O., Abdullabekov D., Usmonov D., Xushbakov M.</b>	
Analytical and experimental model for assessing the depreciation rate of transformer oil based on physicochemical factors	<b>182</b>

---

<b>Obidov A., Abdurasulov A.</b>	
Basis of implementation of resource-effective shaft production	<b>188</b>

---

<b>Utaev S.</b>	
Calculation of oil change intervals in diesel-based gas engines	<b>193</b>

---

<b>Isomiddinov A.</b>	
Derivation of differential equations for spindle oscillation in a system of rectangular coordinates	<b>200</b>

---

<b>Dedakhanov A.</b>	
Determination of fuel consumption for drying cotton raw materials	<b>209</b>

---

<b>Atambaev D.</b>	
Difference of the individual yarns in the composition of a wrapped yar on the quality of the yar and determination of acceptable values of the main factors affecting their production	<b>215</b>

---

<b>Rokhmonov D., Sulaymonov J.</b>	
Development of a control algorithm for a smart irrigation system based on soil moisture and meteorological data	<b>224</b>

---

<b>Mamakhonov A., Khikmatillaev I.</b>	
Modeling of a vibratory cleaning device with cosinoidal and sinusoidal shapes in matching the longitudinal and transverse cutting surface	<b>227</b>

---

<b>Soliyev A.</b>	
Theoretical study and characteristics of yarns in the production of circular knit fabrics	<b>239</b>

---

---

**Nomanov M.**

With improved blade mixer results of research work on the development of the 5lp linter **246**

---

**Lastochkin P.**

The influence of carding parameters optimization on the useful time coefficient of a rotor spinning machine **259**

---

**Mirzaakbarov A.**

Improving the efficiency of the ginning process to enhance fiber quality **260**

---

## ADVANCED PEDAGOGICAL TECHNOLOGIES IN EDUCATION

---

**Abdumanonov A.**

Enhancing the methodology for applying intelligent control systems in the teaching of technical sciences **265**

---

**Makhmudov Z.**

Increasing students' activity and knowledge level using test assignments **271**

---

## ECONOMICAL SCIENCES

---

**Sarimsakov B., Mirzabdullayev R.**

The role of contemporary HR technologies in improving business performance **275**

---