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Адрес редакции: 050100, г. Алматы, ул. Кунаева, 142, АО «Институт топлива, катализа и электрохимии им. Д.В. Сокольского», каб. 310, тел. 291-62-80, факс 291-57-22, e-mail: orgcat@nursat.kz

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G.A. Seitbekova, 2025.**

Taraz University named after M.Kh. Dulaty, Taraz, Kazakhstan.

E-mail: rm.kudajbergenova@dulaty.kz

STUDY OF PHYSICAL AND CHEMICAL CHARACTERISTICS OF HIGHLY DISPERSED ELECTROCORUNDUM

Kudaibergenova Rabiga Musaparovna — PhD, Department of Chemistry and Chemical Technology, M.Kh. Dulaty Taraz University, Tole bi str. 60, Taraz, Kazakhstan,

E-mail: rm.kudajbergenova@dulaty.kz, <https://orcid.org/0000-0003-0759-1539>;

Nurlybayeva Aisha Nurlybayevna — PhD, Associate Professor, Department of Chemistry and Chemical Technology, Taraz University named after M.Kh. Dulaty, Taraz, Kazakhstan,

E-mail: rustem_ergali@mail.ru, <https://orcid.org/0000-0001-9904-9979>;

Mateeva Sulushash Zijatbekovna — Candidate of Chemical Sciences, Department of Chemistry and Chemical Technology, Taraz University named after M.Kh. Dulaty, Taraz, Kazakhstan,

E-mail: mateeva73@mail.ru, <https://orcid.org/0000-0003-4416-1124M>;

Bulekbayeva Kamila Baltabaevna — Candidate of Technical Sciences, Taraz University named after M.Kh. Dulaty, Taraz, Kazakhstan,

E-mail: Nurhat2000@mail.ru, <https://orcid.org/0009-0003-1747-179X>;

Seitbekova Gulnaziya Atashbekovna — Candidate of Technical Sciences, Department of Chemistry and Chemical Technology, Taraz University named after M.Kh. Dulaty, Taraz, Kazakhstan,

E-mail: seitbekova@mail.ru, <https://orcid.org/0000-0001-7087-7180>.

Abstract. Electrocorundum is a refractory, chemically resistant, superhard material based on aluminum oxide. Electrocorundum is an artificially synthesized synthetic corundum obtained by melting bauxite in high-power electric arc furnaces with subsequent crystallization of the melt. Results. In this work, the main parameters of highly dispersed electrocorundum of the Rusal F1000 and Technoceramics F1000 were investigated. The particle sizes were determined, FTIR spectroscopic analyses were carried out, and the dependences of the ζ -potential on the pH value of the dispersion medium of the studied samples were measured. Based on the results of FTIR spectroscopy, it follows that the positions of the absorption bands at 452,50 – 647,48 cm^{-1} indicate the presence of stretching vibrations of the Al-O bond and belong to electrocorundum particles. The results of optical microscopy showed that the size of electrocorundum particles varies from 2-14 μm . Scientific novelty. Systematic research and comparison of production parameters of Rusal and Technoceramics allows not only to get an objective idea of

the quality of the produced material, but also to establish compliance with the declared characteristics. This is especially important in the conditions of increasing requirements for functional materials from high-tech industries. Practical value. The results of such analysis are of practical importance for choosing the optimal raw materials depending on the specifics of the technological process.

Keywords: Electrocorundum, Rusal, Tekhnokeramika, optical microscopy, ζ -potential

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М.Х. Дулати атындағы Тараз университеті, Тараз, Қазақстан.

E-mail: rm.kudajbergenova@dulaty.kz

ЖОҒАРЫ ДИСПЕРСТІ ЭЛЕКТРОКОРУНДТЫҢ ФИЗИКАЛЫҚ-ХИМИЯЛЫҚ СИПАТТАМАЛАРЫН ЗЕРТТЕУ

Кудайбергенова Рабига Мусапаровна — PhD, М.Х. Дулати атындағы Тараз университеті, Тараз, Қазақстан,

E-mail: rm.kudajbergenova@dulaty.kz, <https://orcid.org/0000-0003-0759-1539>;

Нурлыбаева Айша Нурлыбаевна — PhD, қауымдастырылған профессор, М.Х. Дулати атындағы Тараз университетінің «Химия және химиялық технология» кафедрасы, Тараз, Қазақстан,

E-mail: rustem_ergali@mail.ru, <https://orcid.org/0000-0001-9904-9979>;

Матеева Сұлшаш Зижатбековна — химия ғылымдарының кандидаты, М.Х. Дулати атындағы Тараз университетінің «Химия және химиялық технология» кафедрасы, Тараз, Қазақстан,

E-mail: mateeva73@mail.ru, <https://orcid.org/0000-0003-4416-1124>;

Булекбаева Камила Балтабаевна — техника ғылымдарының кандидаты, М.Х. Дулати атындағы Тараз университеті, Тараз, Қазақстан,

E-mail: Nurhat2000@mail.ru, <https://orcid.org/0009-0003-1747-179X>;

Сейтбекова Гүлназия Аташбековна — техника ғылымдарының кандидаты, М.Х. Дулати атындағы Тараз университетінің «Химия және химиялық технология» кафедрасы, Тараз, Қазақстан,

E-mail: seitbekova@mail.ru, <https://orcid.org/0000-0001-7087-7180>.

Аннотация. Электрокорунд алюминий оксидіне негіздегі отқа төзімді, химиялық төзімді, аса қатты материал. Сонымен қатар, электрокорунд бокситті қуатты электр доғалы пештерде балқытып, кейіннен кристалдану арқылы балқыту арқылы алынған жасанды синтезделген синтетикалық корунд. Өте жоғары беріктігінің арқасында ақ электрокорунд абразивті құрал ретінде, сондай-ақ абразивті құралдарды өндіруде қолданылады, ал жоғары температураға және төмен жылу және электр өткізгіштікке төзімділігіне байланысты – керамикалық және отқа төзімді бұйымдар өндірісінде қолданылады. Нәтижелері. Бұл зерттеу жұмысында Rusal F1000 және Technoceramics F1000 маркалы жоғары дисперсті электрокорундының негізгі параметрлері зерттелді. Яғни, бөлшектердің өлшемдері анықталды, ИҚ-спектроскопиялық талдаулар жүргізілді, ζ -потенциалдың зерттелетін үлгілердің дисперсиялық ортасының рН мәніне тәуелділігі өлшенді. ИҚ Фурье спектроскопиясының нәтижелеріне сүйене отырып, 452,50 - 647,48 cm^{-1} аралығындағы абсорбциялық жолақтардың

позициялары Al-O байланысының созылу тербелістерінің бар екендігін көрсетеді және бұл электрокорунд бөлшектеріне тән болып келеді. Үлгілердің оптикалық микроскопиялық нәтижелері электрокорунд бөлшектерінің өлшемі 2-ден 14 мкм-ге дейін өзгертінін көрсетті. Ғылыми жаңалығы. Русал және Технокерамика өнімдерінің параметрлерін жүйелі түрде зерттеу және салыстыру өндірілген материалдың сапасы туралы объективті түсінік алуға ғана емес, сонымен қатар мәлімделген сипаттамаларға сәйкестікті анықтауға мүмкіндік береді. Бұл әсіресе жоғары технологиялық өндірістердің функционалдық материалдарына қойылатын талаптардың артуы жағдайында маңызды. Практикалық құндылық. Мұндай талдау нәтижелерінің технологиялық процестің ерекшеліктеріне байланысты оңтайлы шикізатты таңдау үшін практикалық маңызы бар. Сонымен қатар, мұндай салыстырмалы зерттеулер отандық шикізат базасын дамытуға, импорттық аналогтарға тәуелділікті азайтуға және өндірушілердің ішкі және сыртқы нарықтағы бәсекеге қабілеттілігін арттыруға ықпал етеді.

Түйін сөздер: электрокорунд, rusal, технокерамика, оптикалық микроскопия, ζ -потенциал

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Таразский университет имени М.Х. Дулати, Тараз, Казахстан.

*E-mail: rm.kudajbergenova@dulaty.kz

ИССЛЕДОВАНИЕ ФИЗИКО-ХИМИЧЕСКИХ ХАРАКТЕРИСТИК ВЫСОКОДИСПЕРСНОГО ЭЛЕКТРОКОРУНДА

Кудайбергенова Рабига Мусанаровна — PhD, Таразский университет имени М.Х. Дулати, Тараз, Казахстан,

E-mail: rm.kudajbergenova@dulaty.kz, <https://orcid.org/0000-0003-0759-1539>;

Нурлыбаева Айша Нурлыбаевна — PhD, ассоциированный профессор кафедры химии и химической технологии, Таразский университет имени М.Х. Дулати, Тараз, Казахстан,

E-mail: rustem_ergali@mail.ru, <https://orcid.org/0000-0001-9904-9979>;

Матеева Сулушаш Зиятбековна — кандидат химических наук, кафедра химии и химической технологии, Таразский университет имени М.Х. Дулати, Тараз, Казахстан,

E-mail: mateeva73@mail.ru, <https://orcid.org/0000-0003-4416-1124>;

Булекбаева Камила Балтабаевна — кандидат технических наук, Таразский университет имени М.Х. Дулати, Тараз, Казахстан,

E-mail: Nurhat2000@mail.ru, <https://orcid.org/0009-0003-1747-179X>;

Сейтбекова Гульназия Аташбековна — кандидат технических наук, кафедра химии и химической технологии, Таразский университет имени М.Х. Дулати, Тараз, Казахстан,

E-mail: seitbekova@mail.ru, <https://orcid.org/0000-0001-7087-7180>.

Аннотация. Электрокорунд тугоплавкий, химически стойкий, сверхтвёрдый материал на основе оксида алюминия. Электрокорунд искусственно синтезированный синтетический корунд, получаемый плавкой боксита в мощных дуговых электропечах с последующей кристаллизацией расплава. Благодаря сверхвысокой прочности белый электрокорунд используется в качестве абразива,

применяется для производства абразивных инструментов, а за счет устойчивости к высоким температурам и низкой тепло- и электропроводности – в производстве керамической и огнеупорной продукции. Результаты. В данной работе исследованы основные параметры высокодисперсного электрокорунда марок Русал F1000 и Технокерамика F1000. Также, определены размеры частиц, проведены ИК-спектроскопические анализы, измерены зависимости ζ -потенциала от значения pH дисперсионной среды исследуемых образцов. На основании результатов ИК-Фурье спектроскопии следует, что положения полос поглощения в $452,50 \text{ см}^{-1}$ по $647,48 \text{ см}^{-1}$ указывают на наличие валентных колебаний связи Al-O и принадлежат частицам электрокорунда. Результаты оптической микроскопии показали, что размер частиц электрокорунда варьируется от 2 до 14 мкм. По результатам электрофоретического рассеяния света для суспензий электрокорунда с различными значениями pH дисперсионной среды установлено, что частицы электрокорунда имеют положительный знак заряда при pH от 1 до 6, и отрицательный знак при $\text{pH} > 6$. Научная новизна. Систематическое исследование и сравнение параметров продукции Русал и Технокерамики позволяет не только получить объективное представление о качестве выпускаемого материала, но и установить соответствие заявленным характеристикам. Это особенно важно в условиях повышения требований к функциональным материалам со стороны высокотехнологичных отраслей промышленности. Практическая ценность. Результаты такого анализа имеют практическую значимость для выбора оптимального сырья в зависимости от специфики технологического процесса.

Ключевые слова: электрокорунд, русал, технокерамика, оптическая микроскопия, ζ -потенциал

Introduction. Electrocorundum is a refractory and chemically resistant superhard material based on aluminum oxide (Al_2O_3). There are various modifications of aluminum oxide, but only corundum $\alpha\text{-Al}_2\text{O}_3$ has thermodynamic stability. Corundum is a white refractory powder with a density of 4 g/cm^3 , close to diamond in hardness. Chemically inert, it dissolves only in molten alkali (Angelescu, et al., 2010). The high stability of corundum is explained by the strength of its crystal structure, which is a two-layer dense spherical packing of oxygen ions, in the octahedral voids of which aluminum ions are located (Permikina, et al., 1986). The Al-O distance is almost the same throughout the crystal and is approximately 0,185 nm. Since the alpha modification of aluminum oxide is the only one present in nature, corundum can be obtained from natural rubies, bauxites, sapphires, etc. Corundum is obtained synthetically by thermal decomposition of aluminum hydroxide or alum ($(\text{NH}_4)\text{Al}(\text{SO}_4)_2$ at $1000\text{-}1200^\circ\text{C}$, or by thermal transformation of other forms into α .

Electrocorundum is an artificially synthesized synthetic corundum (88-99% Al_2O_3). It obtained by melting bauxite in electric furnaces with a reducing agent (anthracite, petroleum coke) and a precipitating agent (iron filings) (Teresa, et al., 2017). It used as an abrasive, refractory material for the manufacture of casting molds and rods, structural elements of radio tubes, abrasive wheels, etc. The study of the properties of highly

dispersed electrocorundum plays an important role in understanding its performance characteristics and selecting optimal conditions for its use. Particle size, morphology, phase composition and mechanical properties have a significant impact on the final properties of the material and its functionality in various technological processes (Drygalska, et al., 2012).

Features and advantages of this material: high resistance to chemical, temperature and climatic influences; preservation of crystal structure when heated during operation; the running size of crystals is 0,6-0,8 mm; homogeneity in mineralogical and chemical composition; minimal inclusion of foreign impurities (no more than 1%) (Ogrodnik, et al., 2010).

The classic color of electrocorundum is white. White electrocorundum is used for grinding cooled, high-carbon and high-speed steel. It is also widely used in the manufacture of ceramic and refractory parts. It can be used on catalyst carriers and special grinding machines (Mikhailov, et al., 2019).

Determination of the main physicochemical characteristics of highly dispersed electrocorundum produced by Rusal and Technoceramics is a relevant area of applied materials science research. Electrocorundum is widely used in the production of refractory, abrasive, ceramic and composite materials, where stability of properties, homogeneity of structure and high purity of phase composition are critical (Krasnyi, et al., 2009). At the same time, even minor differences in the parameters of the initial raw materials can significantly affect the quality of the final product. Despite the general classification according to state standards and technical conditions, brands of different manufacturers can vary in a number of indicators: average particle size, granule morphology (Krasnyi, et al., 2009), degree of agglomeration, specific surface area, composition and level of impurities (Komolikov, et al., 2018).

Therefore, a systematic study and comparison of the parameters of Rusal and Technoceramics products allows not only to obtain an objective idea of the quality of the manufactured material, but also to establish compliance with the declared characteristics (Gasik, et al., 2020). This is especially important in the context of increasing requirements for functional materials from high-tech industries. The results of such analysis are of practical importance for selecting the optimal raw material depending on the specifics of the technological process. For example, the production of technical ceramics requires materials with high phase purity and a narrow particle size distribution, while in the abrasive industry; preference may be given to more durable and heat-resistant fractions (Anisimov, et al., 2022). In addition, such comparative studies contribute to the development of the domestic raw material base, reducing dependence on imported analogues and increasing the competitiveness of manufacturers in the domestic and foreign markets. Rusal F1000 is one of the world's largest manufacturers of white electrocorundum. It is produced by melting pure alumina in electric arc furnaces. Due to its ultra-high strength, white electrocorundum is used as an abrasive, applied in the production of abrasive tools, and due to its resistance to high temperatures and low thermal and electrical conductivity – in the production of ceramic and refractory products (Tsyv'yan, 2003).

The purpose of this study is to determine the main parameters of various brands of highly dispersed electrocorundum of the Rusal F1000 brand, electrocorundum of the Technoceramics F1000 brand.

Materials and methods. Materials: During the work, the following substances were used as initial reagents: electrocorundum Rusal F1000 and electrocorundum Technoceramics F1000, artificially synthesized synthetic corundum (88-99% Al_2O_3). Rusal F1000 is one of the world's largest producers of white electrocorundum. It is produced by melting pure alumina in electric arc furnaces at the Boksitogorsk Alumina Plant. Technoceramics F1000 is a Russian research and production company. Since 2010, we have been actively engaged in the development and production of high-tech and science-intensive refractory and abrasive materials made of white electrocorundum 25A, mullite, as well as zirconium dioxide and other materials. Methods: FTIR analysis was carried out to identify and study the structure. FTIR absorption spectra were recorded at room temperature in the frequency range 4000–400 cm^{-1} (mid-IR region) on a Thermo Fisher Scientific Inc (USA) Fourier-transform IR spectrometer using the KBr method (transmission).

The electrophoretic light scattering method was used to measure the ζ -potential. The ζ -potential of the samples was determined using a Photocor Compact-Z device (Photocor LLC, Russia). This method is based on the dynamic light scattering method in the configuration of a laser Doppler anemometer, which is used to measure the velocities of liquid and gas flows. The contact liquid for electrophoresis was prepared using distilled water and nitric acid. The velocity of the particles in the field, calculated from the phase function, allows one to determine the electrophoretic mobility of the particles.

$$\mu_E = \frac{v}{E}$$

Where, v is the velocity of charged particles in an electric field with strength E .

The electrophoretic mobility of particles is recalculated using the Helmholtz-Smoluchowski equation and applying corrections for different thicknesses of the double electric layer.

$$\mu_E = \frac{2\varepsilon\xi}{3\eta},$$

Where, ξ is the zeta potential, mV; μ_E is the electrophoretic mobility, $\text{m}^2/(\text{V}\cdot\text{s})$; ε is the permittivity, F/m; η is the viscosity, Pa·s.

The particle sizes were determined using optical microscopy. An optical microscope designed for laboratory research is used for the analysis, and a micrometer grid is inserted into the eyepiece.

Results and discussion. FTIR analysis.

The infrared spectroscopy method makes it possible to obtain information about the relative positions of molecules over very short periods of time, as well as to evaluate the nature of the bonds between them, which is fundamentally important when studying the structural and informational properties of various substances. Absorption bands appear as a result of transitions between vibrational levels of the ground electronic state of

the system being studied. The spectral characteristics (positions of band maxima, their half-width, intensity) of an individual molecule depend on the masses of its constituent atoms, the geometry of the structure, the features of interatomic forces, the charge distribution, etc., therefore infrared spectra are distinguished by great individuality, which determines their value in identifying and studying the structure of compounds.

For alumina materials, infrared absorption spectra were obtained, which are presented in Figures 1 and 2.

As can be seen, several absorption bands are observed in the FTIR spectra in the range of 400 – 650 cm^{-1} . The results of the FTIR spectrum identification of the Rusal F1000 sample showed absorption spectra at frequencies of 452,50 cm^{-1} , 605,88 cm^{-1} and 647,48 cm^{-1} , which are related to the stretching vibrations of the Al-O bond. Also, peaks at 453,17 cm^{-1} , 601,53 cm^{-1} and 640,57 cm^{-1} , which are characteristic of the stretching vibrations of the Al-O bond, were also revealed in the spectra of the Technoceramics F1000 sample. Based on the FTIR spectroscopy results, it follows that the positions of the absorption bands indicate the presence of stretching vibrations of the Al-O bond and belong to the particles of electrocorundum (Wei, et al., 2020).

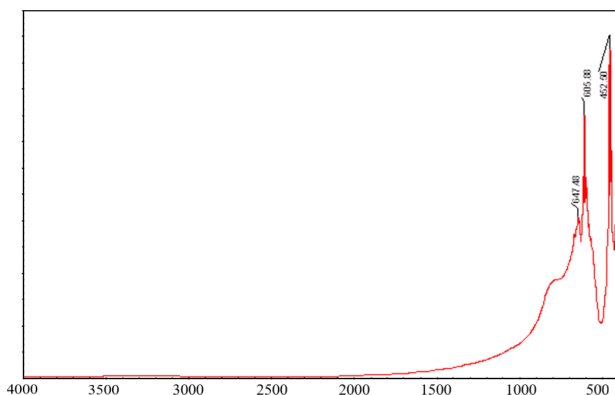


Figure 1 - FTIR spectrum of the Rusal F1000 electrocorundum sample

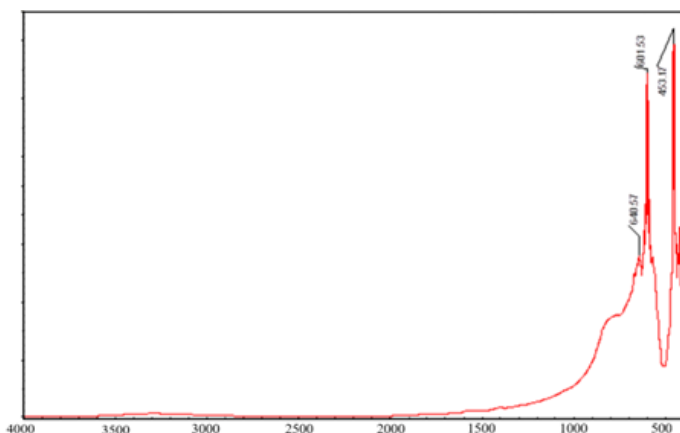


Figure 2 - FTIR spectrum of the Technoceramics F1000 electrocorundum sample

Electrosurface properties of electrocorundum samples.

The electrophoretic light scattering method was used to measure the zeta potential. The ζ -potential value allows us to judge the potential stability of the system. Figure 8 shows the dependences of the ζ -potential on the pH values of the medium for all the studied samples.

For each sample, the sign of the ζ -potential value was found at a certain pH of the dispersion medium, and the pH values at which the minimum and maximum pH values would be observed were determined.

For the electrocorundum sample - Rusal F1000, it was found that in the range from pH = 1 to pH = 6, the Al_2O_3 particles are positively charged, and in the range from pH = 6 to pH = 10, the Al_2O_3 particles are negatively charged. The isoelectric point of the sample is observed at pH = 6. The minimum value of the ζ -potential is 10 mV, the maximum value of the ζ -potential is 35 mV at pH = 2.

For the electrocorundum sample - Tekhnokeramika F1000, it was found that in the range from pH = 1 to pH = 7, the Al_2O_3 particles are positively charged, and in the range from pH = 7 to pH = 10, the Al_2O_3 particles are negatively charged. The isoelectric point of this sample is observed at pH = 7. The minimum value of ζ -potential is 0 mV, the maximum value of ζ -potential is 10 mV at pH=5.

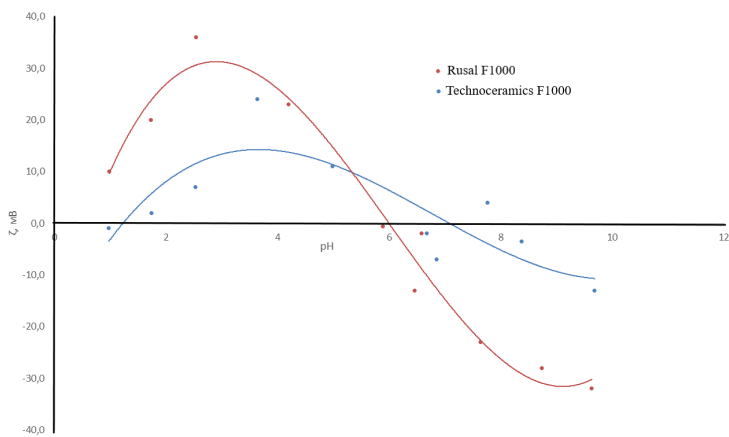


Figure 3 - Dependence of the ζ -potential on the pH value of the dispersion medium for the studied samples

Determination of particle sizes by optical microscopy

An optical microscope designed for laboratory research is used for the analysis, into the eyepiece of which a micrometer grid is inserted. The scale of the micrometer grid divides the field of view of the microscope into 100 squares, the side of each of which is 5 grid divisions. The value of a grid division of 24 depends on the magnification of the eyepiece and objective and is determined using an eyepiece micrometer. A glass slide with a sample of emulsion or powder is placed under the microscope objective and, by moving the microscope tube vertically, the best sharpness of the particle image is achieved. To obtain the best magnification for counting particles, a sample is examined

with different eyepieces and one is selected so that no more than 30-40 and no less than 15 particles are within the micrometer grid.

A micrograph of the Rusal F1000 electrocorundum sample was obtained using an optical microscope and is shown in Figure 4.

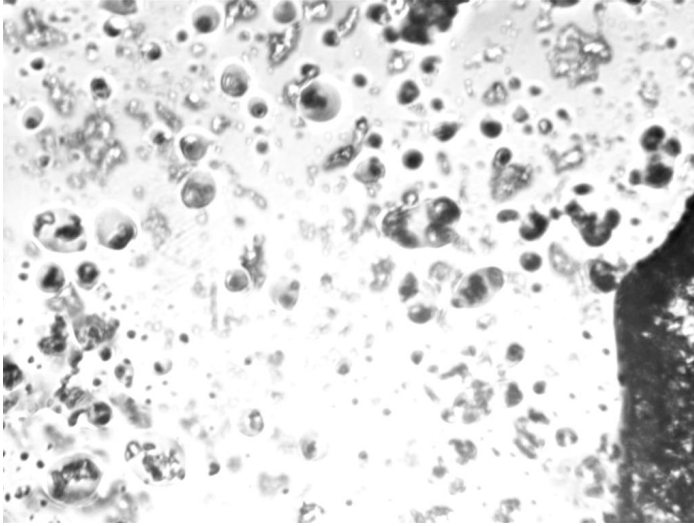


Figure 4 - Micrograph of a sample of electrocorundum (Rusal F1000)

As can be seen, the dispersed material is represented by irregularly shaped particles, and particle aggregation is observed [Kryuchkov, 2020]. The results of dispersion analysis obtained by optical microscopy are presented in Table 1.

Table 1. Results of dispersion analysis

$r_{i-1}; r_i$	1,73-3,03	3,03-4,33	4,33-5,63	5,63-6,93	6,93-8,23	8,23-9,53	9,53-10,83	10,83-12,13	12,13-13,43	13,43-14,69
$r_i, \text{ med}$	2,4	3,7	5,0	6,3	7,6	9,0	10,2	11,5	13,4	14,06
Number of particles	10	19	8	6	8	7	3	1	1	1
$Q_i \%$	15,6	29,6	12,5	9,3	12,5	10,4	4,6	3,1	1,5	1,5

Based on the obtained results, a histogram of the distribution of sample particles by size was constructed (Zhu, et al., 2001). The presented histogram shows 1 predominant size – 3,7 μm . The particle sizes vary from 2,4 to 14 μm .

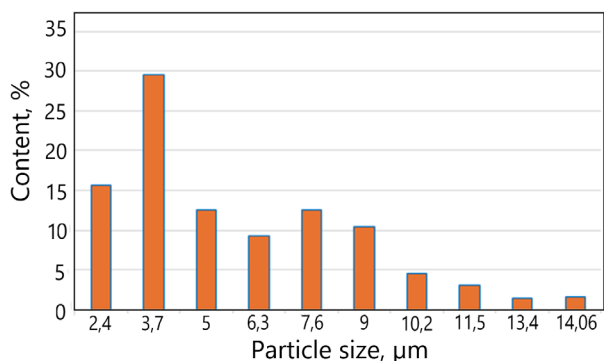


Figure 5 - Histogram of distribution of electrocorundum (Rusal F1000) by sizes

Also, a micrograph of an electrocorundum sample (Technoceramics F1000 brand) was obtained using a microscope, shown in Figure 6.

In this micrograph obtained by the optical method, it can also be seen that the material is represented by particles of different shapes, and aggregation of particles is observed (Kryuchkov, 2020).

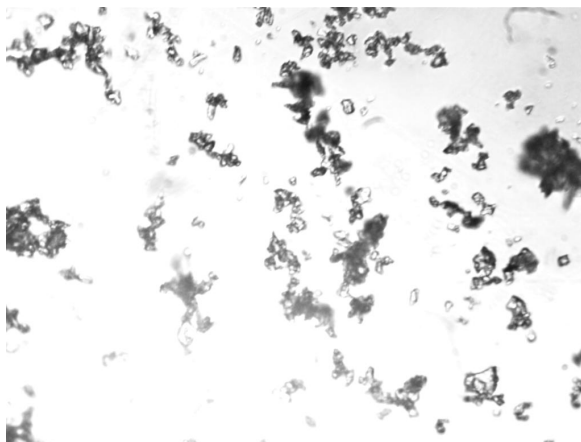


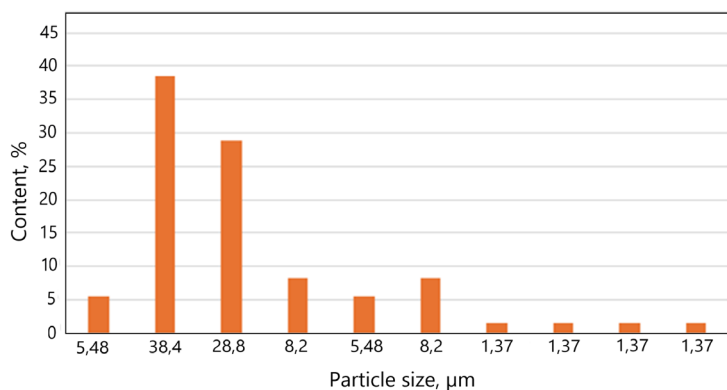
Figure 6 - Micrographs of a sample of electrocorundum (Technoceramics F1000)

The results of the dispersion analysis obtained by optical microscopy are presented in Table 2.

Table 2. Results of dispersion analysis

$r_{i-1}; r_i$	1,16-2,42	2,42-3,68	3,68-4,94	4,94-6,2	6,2-7,46	7,46-8,72	8,72-9,98	9,98-11,24	11,24-12,5	12,5-13,8
r_i , med	1,8	3,05	4,3	5,6	6,8	8,09	9,3	10,6	12,0	13,1
Number of particles	4	28	21	6	4	6	1	1	1	1
Q_i , %	5,48	38,4	28,8	8,2	5,48	8,2	1,37	1,37	1,37	1,37

Based on the obtained results, a histogram of the distribution of material particles by size was constructed (Figure 7). The presented histogram shows one predominant size – 3,8 μm . The particle sizes vary from 1,2 to 14,0 μm .



Conclusion. In the context of increasing requirements for technical materials, control and precise characterization of the physical and chemical parameters of electrocorundum, especially in its dispersed state, is of particular importance. In this work, the main parameters of electrocorundum of different grades (Rusal F1000, Technoceramics F1000) were determined. The results of optical microscopy showed that the size of electrocorundum particles varies from 2 to 14 μm . According to the results of electrophoretic light scattering for electrocorundum suspensions with different pH values of the dispersion medium, it was determined that the electrocorundum particles have a positive charge sign at pH from 1 to 6, and a negative sign at pH > 6.

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