# "GREEN" CEMENT COMPOSITES WITH HYBRID ADDITIVES OF MICROSILICA AND ASH SLAG MIXTURE FROM THERMAL POWER PLANT

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*Received 21 June 2024*
*Accepted 14 July 2025* *DOI: 10.59957/jctm.v60.i6.2025.9*

## ABSTRACT

*The paper presents the results of studies on determination of composition of ultra dispersed microsilica (MS) and its derivatives in composition with active ash and slag mixture (ASM) of dry ash removal Thermal Power Plant. Their chemical and mineralogical compositions and suitability for use as an additive in cement were determined by their hydraulic activity according to the Student's t-criterion value and CaO absorption from saturated lime solution. It has been established that according to the t-criterion value, ASM meets the requirements of the Interstate standard (ISS) 31108 - 2020 for active mineral additives for cement (t - criterion - no less than 15), but MS does not correspond. Taking these data into account, the compositions of hybrid additives (HA) were formed, including active (ASM) and passive components (ASM), providing maximum replacement of the expensive clinker part in cement with the optimal ratios of the ingredients of hybrid additives and its optimal dosage found in the composition of composite Portland cements (CPC).The rate of chemical interaction of the ratio of mineral ingredients was found to be in the composition of hybrid additives: CPC exhibits high chemical activity upon contact with water with a content of 15 % ASM + 10 % MS (HA), introduced into cement in an amount of up to 35 %.*

*The achievement of hydraulic activity of cement composites is scientifically substantiated to be higher than that of additive-free cement, providing their belonging to the class 32.5 and 42.5 in strength due to the formation of a dense microstructure. Control tests of "green" CPC in the laboratories of two large cement plants of Uzbekistan confirmed the results of research on the possibility of replacing up to 35 % of expensive clinker with available and cheap hybrid additives from local techno genic waste to obtain high durability composite Portland cement.*

*Keywords: microsilica, ash and slag mixture of thermal power plants, hydraulic activity, hybrid additives, "green" cement composites, durability, microstructure.*

## INTRODUCTION

Active mineral pozzolanic additives have been purposefully used for many decades to modify Portland cement-based formulations to improve the strength, durability, impermeability and chemical resistance of the resulting materials and structures. One of the most active and widely used pozzolanic modifiers is ultra disperse microsilica (MS) - a waste product from the production of silicon-containing alloys, consisting of spherical particles 0.01 - 0.1 μm in size and containing up to 95 % of pure amorphous silica, capable of actively reacting with lime released by Portland cement during its hydration, with the formation of water-insoluble hydrate compounds. It is microscopic spherical particles of amorphous silica with an average specific surface area of about 20 $m^2$ $g^{-1}$. This highly active mineral additive consists of oxides of silicon, aluminium, iron, calcium, magnesium, potassium, carbon and sulphur. In terms of particle size distribution, the average particle size of microsilica is about 0.1 μm, i.e. 100 times smaller than the average cement grain size [1].

Researchers from the USA, UK, France, Australia, Germany, Netherlands and Bangladesh have published information on climate change in the journal Bioscience. According to their data, three key greenhouse gases (carbon dioxide, methane, nitrous oxide) set records for atmospheric concentrations in 2020. In 2021, atmospheric carbon dioxide reached the highest Monthly average of 416 parts per million. In the summer of 2022, scientists announced that the level of $CO_2$ in the atmosphere reached a record high for millions of years. Measurements from NOAA's Background Atmospheric Conditions Observatory recorded a carbon dioxide reading of 420.99 parts of gas per million parts of air, a 1.8 - millionth increase from 2021. The main carbon reduction measures highlighted in the ESG agenda ("green" agenda) include: the introduction of new technologies, investments in research and development, environmental initiatives, internal emission reduction plans, transition to carbon-free technology, which are still relevant today. The cement industry, which emits a huge amount of carbon dioxide, also makes a huge "contribution" to the atmospheric air, as the industry consumes carbonate-containing raw materials, mainly in the form of limestone, in the production of clinker, a semi-product for cement. During firing of "limestone-clay" raw material mixture at high temperature (1450 - 1500°C) about 0.8 - 0.9 tons of carbon dioxide per 1 tone of Portland cement clinker are emitted into the atmosphere [2, 3]. Based on the results of such studies, there is currently a worldwide struggle to reduce the impact of harmful dust and gas emissions from industrial enterprises into the atmosphere, against the formation of greenhouse gases and for a clean climate. For this purpose, the tendency to produce low-clinker cements by modification of Portland cement clinker by silica, thermo activated alumni silicate, carbonate-containing and other types of active mineral and composite additives of natural and anthropogenic origin is currently developing [4 - 7].

For the most complete coverage of the demand of the intensively developing construction industry of the republic in cement, technologically, economically and environmentally favourable option to increase the volume of cement production is modification of clinker by active mineral additives and their derivatives with the use of local natural and man- made raw materials. In this aspect, the use of accumulated and current wastes of industrial productions that have passed a certain stage (mechanical, chemical, mechanical-chemical, thermal, etc.) of processing as additives for cement is ecologically and economically feasible. They also include wastes of mining and metallurgical production and thermal power engineering, which are overburden, host rocks, poor, off-balance, substandard ores, tailings, ash and slag from thermal power plants, slags and slimes of metallurgy, which have a significant resource potential and are a reserve of promising mineral raw materials to produce various construction products [8 - 13].

This highly reactive mineral component is primarily composed of silicon dioxide, with minor amounts of oxides of aluminium, iron, calcium, magnesium, potassium, carbon, and sulfur. When incorporated into Portland cement mixtures, it demonstrates a strong tendency to react with lime released during the hydration of tricalcium silicate ($C_3S$), contributing to the formation of calcium hydrosilicate (CSH (I)) — a water-insoluble hydrate compound. The reaction can be represented as follow:

$$SiO_2 + Ca(OH)_2 + nH_2O \rightarrow (1\text{-}1.5)\ CaO\cdot SiO_2\cdot(0.5\text{-}2.5)\ H_2O$$

This interaction leads to the development of additional calcium silicates within the cement–water matrix and creates internal structural tension due to the participation of the siliceous additive in cement stone formation. As a result, the microstructure of the hardened cement becomes denser and stronger. Furthermore, the use of ultrafine silica enhances the extent of cement hydration, resulting in improved processing characteristics and durability of the concrete.

The need to reduce carbon dioxide emissions into the atmosphere and save clinker component in cement production has determined the purpose of our research in terms of formation of effective types of hybrid additives based on active ash and slag mixture of TPPs of dry disposal and microsilica and development of technology of low clinker "green" cement composites with their use.

## EXPERIMENTAL

Portland cement clinker of JSC “Akhangarancement” PC clinker, gypsum stone of Bukhara deposit, ash and slag mixture of dry disposal (ASM) of Angren TPP and micro silica (MS) of JSC “Uzmetkombinat” served as objects in experimental work. Standard methods of physic-chemical research and physic-mechanical tests in accordance with the requirements of the following normative documents were applied in the work: chemical analysis of PC clinker of JSC “Ahangarancement”, gypsum stone of Bukhara deposit, ASBS of Angren TPP and MS of JSC “Uzmetkombinat” was carried out according to the methods of The Interstate standard 5382 - 91 “Cements and materials of cement production. Methods of chemical analysis”. Hydraulic activity of ASM and MS was determined according to the method of The Interstate standard 25094 - 94 “Active mineral additives for cements. Test methods”, the results obtained by Student’s criterion were evaluated according to The Interstate standard 31108 - 2020 “Cements for general construction. Technical conditions”. Physical and mechanical properties of laboratory samples of cements with hybrid additive (HA) were determined on small cube samples of 1.41×1.41×1.41 cm made of cement dough (composition 1:0). Hydraulic activity of the optimal cement composition was determined according to the method of The Interstate standard 310.4 - 81 “Cements. Methods of determination of bending and compressive strength”, the evaluation of the obtained results was carried out according to The Interstate 31108 - 2020 “Cements for general construction. Technical conditions”. Physic-chemical properties of “green” cement composites were investigated using X-ray phase (diffract meter XRO - 6100 of Shimadzu campaign), DTA (thermal analytical device Nietzsche Simultaneous Analyzer STA 409 PG), IR spectroscopic (Fourier spectrometer “IRTracer -100”, SHIMADZU CORP) and electron microscopic (scanning electron microscope JSM - 6490LV with systems of energy dispersive microanalysis INCA Energy and structural analysis HKL Basic) methods [14 - 17].

## RESULTS AND DISCUSSION

According to the values of modular characteristics (saturation coefficient (SC), n, p) clinker of JSC “Akhangarancement” meets the requirements of Uzbekistan State Standard (UzSStan) 2801:2013 “Portland cement clinker. Technical conditions”, and gypsum stone by total content of gypsum and anhydride (93,44%) - 2 grade according to The Interstate standard 4013-2019 “Gypsum and gypsum-anhydrite stone for production of binding materials. Technical conditions” and can be used as a regulator of cement setting time (Table 1) [18,19].

MS is predominantly (90.9 %) composed of particles with grain sizes of 0.1 - 0.2 μm (32.2 %), 0.2 - 0.4 μm (34.1 %) and 0.4 - 1.0 μm (24.6 %). The remaining 9.1 % of its constituents are particles with grain sizes less than 0.1 μm (3.7 %), 1.0 - 2.5 μm (4.0 %) and greater than 2.5 μm (1.4 %). The $SiO_2$ content of is 90.84 % and impurities of other oxides are 7.29 %. At calculation of Student’s criterion, the value of $t = 13.47 < 15.0$ is received, that does not correspond to requirements of The Interstate standard 31108 - 2020 on active mineral additive for cement, i.e. MS is a mineral additive of average activity.

Table 1. Chemical compositions of components for the formation of hybrid additives and composite cements.

| Raw materials | Mass fraction of oxides, % | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Losses during calcination | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | Others |
| Clinker | 0.15 | 20.83 | 5.28 | 3.16 | 4.80 | 1.79 | 1.01 | 2.98 |
| | modular characteristics: SC = 0.94; n = 2.48; p = 1.67 | | | | | | | |
| Gypsum | 19.10 | 1.52 | 0.13 | 1.14 | 33.04 | 0.20 | 43.46 | 1.41 |
| | $\Sigma\ CaSO_4 - 2H_2O = 43.46 \times 2.15 = 93.44$ % | | | | | | | |
| ms | 2.79 | 90.84 | 1.51 | 1.59 | 0.56 | 1.00 | 0.23 | 1.48 |
| ASM | 3.0 | 62.02 | 23.55 | 4.32 | 3.0 | - | 1.28 | 0.80 |

ASM is a polydisperse mass of grey colour, including slag fraction with grain size not more than 15 mm and fine ash fraction. In the chemical composition of ash and slag mixture the content of $SiO_2$ oxides was 62.02 %, $Al_2O_3$ - 23,55 %. The diffract gram of ASM reveals quartz lines at d/n = (0.421; 0.331; 0.243; 0.226; 0.222; 0.212; 0.197; 0.181; 0.166; 0.153) nm, mullite (d/n = 0.346; 0.331; 0.251) nm, CaOсв (d/n = 0.239; 0.169) nm and $CaCO_3$ (d/n = 0.301) nm. Lines with d/n = (0.291; 0.285; 0.278) nm refer to low-basic silicates formed during coal combustion because of burning of its mineral part. The value of the Student's criterion of ASM t = 52.92 > 15.0, which characterizes it as a mineral additive of high activity. The compositions of HA "ASM + MS" were formed and their hydraulic activity was determined by the method of saturation of liquid phase, the essence of which is to determine their ability to absorb lime (CaO) formed during the hardening of cement samples, in the composition of which from 25 - 45 % of clinker part was replaced by HA containing 10 % MS and from 15 to 35 % ASM (Table 2). To compare the obtained data on the degree of lime saturation of the liquid and its alkalinity, samples of unblended Portland cement (PC 400 - A0) were also made (HA) "ASM + MS" exhibit high pozzolanic activity, which depends on the amount of ASM in the additive. At its content of 15 % in the composition with 10 % MS, the amount of CaO absorbed from the liquid phase was 0.78 %, which is 4.62 % less compared to its content in the liquid in which the samples of PC 400 - A0 were stored, and the total alkalinity of the solution decreased to 20 meq $L^{-1}$, which is more than 3.5 times less than the alkalinity of the liquid in which the samples of PC 400 - A0 were stored.

With increasing the content of ASM up to 25 - 35 %, the concentration of CaO in the liquid sharply decreases (0.72 - 0.36) %, which emphasizes the special role of ASM in increasing the degree of CaO absorption from the reaction medium due to the acceleration of cement hydration and hydrolysis of high-calcium silicates $C_3S$ and $C_2S$ with intensive transition of $Ca^{2+}$ ions into the liquid phase and their absorption by components of HA, in particular ASM (Fig. 1). To determine the influence of HA "ASM + MS" on physical and mechanical properties of PC, the raw material charges with the specified dosages of HA were prepared (Table 2).

Addition of HA accelerates the clinker grinding process and increases the dispensability of cement powder, as for the same grinding time (45 min), the residue on sieve No. 008 of modified cements is 1 - 2 % less than that of PC - A0. Replacement of 25 - 45 % of clinker component in cements by hybrid additive, in the initial terms of hardening (up to 7 days) somewhat slows down the process of strength gain, which by 28 days

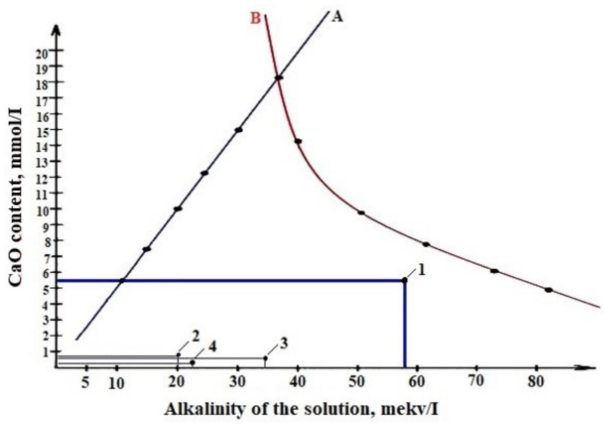


Fig. 1. Degree of pozzolanic activity of cements with HA "ASM + MS": A - lime solubility isotherm at 40°C; B - alkalinity attributable to the share of all components except lime. CaO content in solution: 1) PC 400 - A0, 2) HA 25 %, 3) HA 35 %, 4) HA 45 %.

Table 2. Indicators of hydraulic activity of hybrid additives.

| No | Component ratio, wt. % | | | | CaO content in liquid, % | Total alkalinity of the solution, meq $L^{-1}$ |
|---|---|---|---|---|---|---|
| | Clinker | ASM | MS | Gypsum | | |
| 1 | 95 | - | - | 5 | 5.4 | 58.8 |
| 2 | 70 | 15 | 10 | 5 | 0.78 | 20.0 |
| 3 | 60 | 25 | 10 | 5 | 0.72 | 35.2 |
| 4 | 50 | 35 | 10 | 5 | 0.36 | 23.2 |

accelerates, and the index of cement composite reaches the index of matrix PC 400 - A0 (Table 3). It should be noted that the strength indices of cements established by testing small cube specimens made of cement dough of 1:0 composition do not reflect the true hydraulic activity characterizing their grade or strength class.

In this connection the hydraulic activity of 2 compositions of composite cement containing 25 and 45 % of HA “ASM+MS” was determined, for what standard samples were made according to the method of The Interstate 310.4 - 81 (Table 4). In accordance with the data of Table 4, composite Portland cement with 25 - 45 % of HA “ASM + MS”, on hydraulic activity by one mark exceed the indicators of matrix PC 400 - A0, which according to The Interstate 31108 - 2020 correspond to the class 42.5 on durability.

The rate of hydration of binding systems is determined by the removal of various forms of water from cement stone during drying. The amount of water removed from crystalline hydrates characterizes the rate of transformation of anhydrous cement minerals into hydrate compounds and it varies depending on the duration of their interaction with water.

To find out the rate of chemical transformation in the system “ground clinker-additive- gypsum-water”, the samples of optimal compositions of CPC with hybrid additive “ASM + MS”, hardening in water for (1 - 28) days, were subjected to heat treatment at (950 - 1000$^{o}$C), after which their weight loss was determined. The difference in the weight of the initial and heat - treated sample is the amount of water chemically bound into hydrate products, and the change in its amount over time, allows us to judge about the rate of the bonding process and, indirectly - about the number of new formations.

It is established that in the process of hardening of PWC containing 25 % of HA composition (15 % ASM + 10 % MS), the process of hydration and water binding into hydrate products both in the initial and later periods proceeds faster, as evidenced by a higher content of chemically bound water (by 2.04 - 7.38 %) in PC samples than in hardened samples of PC 400 - A0 in the period of 1 - 28 days. Increase of ASM content in HA composition up to 35 % (25 % ASM + 10 % MS) (composition No 4) sharply slows down the process of hydration and the amount of chemically bound water in all periods of its hardening by 4 - 5 % less compared to the previous composition of PC. This is probably a consequence of the fact that when mixing with water, a large number of dispersed particles of ASM and MS in HA, absorbing water swell and forming colloidal sub microcrystalline formations in the initial terms cover the surface of clinker grains and prevent the deepening of the process of their hydration, which somewhat slows down the binding of water in hydrate formations.

According to the data of X - ray phase analysis, the primary products of hydration of PDA of this composition are $Ca(OH)_2$, some lines of which (d/n = 0.193; 0.176 nm) merge (except d/n = 0.491 nm) with the

Table 3. Change in strength of cements depending on the dose of HA “ASM + MS”.

| No | Designation of cements | Water/ cement | Compressive durability of samples, MPa after days | | | |
|---|---|---|---|---|---|---|
| | | | 1 | 3 | 7 | 28 |
| 1 | PC-A 0 | 0.24 | 13.3 | 35.8 | 52.9 | 54.0 |
| 2 | PC-HA 25 | 0.24 | 8.30 | 27.9 | 40.0 | 52.5 |
| 3 | PC-HA 35 | 0.24 | 11.6 | 32.0 | 42.0 | 50.8 |
| 4 | PC-HA 45 | 0.24 | 12.0 | 24.5 | 45.0 | 50.0 |

Table 4. Hydraulic activity of “green” cement composites.

| No. of p.p.s | Designation of cements | Water/ cement | Compressive durability of Samples, MPa, after days: | | | | Grade/strength class |
|---|---|---|---|---|---|---|---|
| | | | 1 | 3 | 7 | 8 | |
| 1 | PC-A0 | 0.24 | 13.3 | 35.8 | 52.9 | 43.2 | 400/32.5 |
| 2 | PC-HA25 | 0.24 | 8.30 | 27.9 | 40.0 | 52.5 | 500/42.5 |
| 4 | PC-HA45 | 0.24 | 12.0 | 24.5 | 45.0 | 50.0 | 500/42.5 |

lines of initial clinker minerals. The appearance of the line of ettringite (d/n = 0.972; 0.560; 0.387 nm), which constitutes the crystalline framework of the solidifying mass, was noted. The line found at d/n = (0.304; 0.191) nm indicates the partial carbonation of $Ca(OH)_2$, released into the liquid phase with the formation of $CaCO_3$, and the line with d/n = 0.387 nm - the introduction of carbonate ions into the structure of calcium hydro aluminates and the formation of the carbonate analogue of ettringite $3CaO.A1_2O_3.3CaCO_3.31H_2O$, which also, like ettringite, participates in the formation of the crystalline framework of the cement composite. By 7 days the picture of hydrate products formation changes significantly: the intensities of clinker minerals and $Ca(OH)_2$ lines decrease strongly, which indicates the acceleration of the process of hydrolysis of clinker silicate minerals and binding of $Ca(OH)_2$ into hydrate products. The level of diffraction reflections of ettringite and its carbonate analogue also slightly decreases, apparently due to recrystallization of hydration products during this period, the amount of which increases again by 28 days of age.

According to Shpynova and Franus et al., time-hardening cement paste consists mainly of hydrated calcium silicates, the so-called C-S-H phase, accompanied by calcium hydroxide (portlandite) and calcium aluminate hydration products, i.e. ettringite [20, 21]. The initial stages of hydration of this phase are characterized by a radial concentration of fibers or needles, often tapered at the ends. These fibers grow from the surface of the cement grains. The increasing degree of orientation of the C-S-H structure is manifested by the formation of a lattice of fibers, sometimes three-dimensional plates, so-called "honeycombs", which are transformed into densely packed, isometric grains.

In the process of further growth crystalline products begin to overlap each other and splicing, in some areas of the solidifying mass by 7 days form parallel orientated blocks (Fig. 3).

The relief of the surface of a fresh CPC after a day of curing is represented by a fine-grained plane with many pores of different sizes, from the walls of which grow the smallest fibrous and needle crystals, which gradually increasing in size and chaotically arranged in the form of crystal meshes by 3 days, begin to fill the pore spaces (Fig. 2).

By 28-day period the relief of the composite spall surface acquires more ordered character. Both parallel and perpendicular growth of crystalline neoplasms on the surface is noted, which relates to the fact that the acceleration of hydrolysis of silicates under the layers of hydration products. As the process of hydration of the system "ground clinker - additive - gypsum - water" deepens, the degree of contact between clinker grains and water increases, and the process of hydration is accompanied by the removal of crystalline products to the surface of the reaction medium, i.e. there is a met somatic removal of matter [22]. As a result, these crystals in the volume of hydrated medium are located perpendicularly from the surface of blocks towards free spaces (Fig. 4).

The amount of dissolved hydro silicates on the surface of hardening stone becomes more and they more densely packed, reduce structural porosity and strengthen the forming cement composite, which, despite

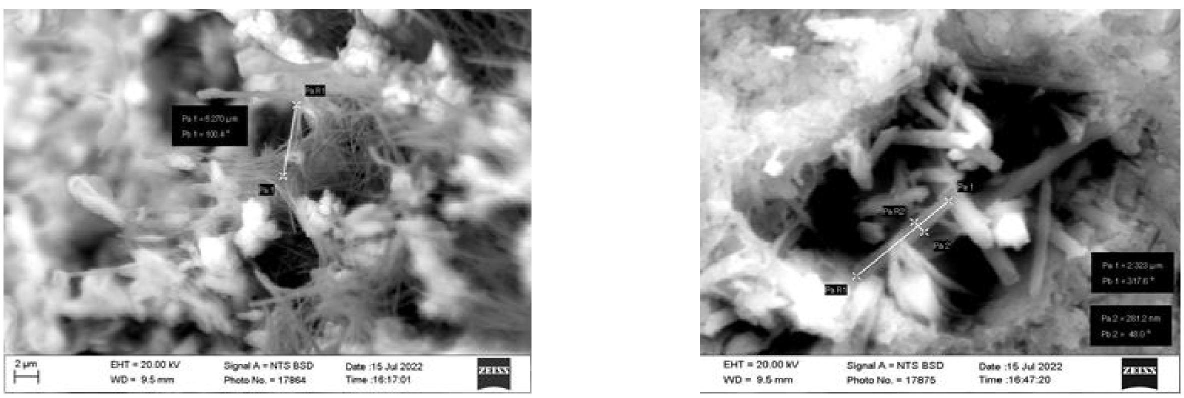


Fig. 2. Relief of the surface of the CPC cleavage after 1- and 3-days hardening.

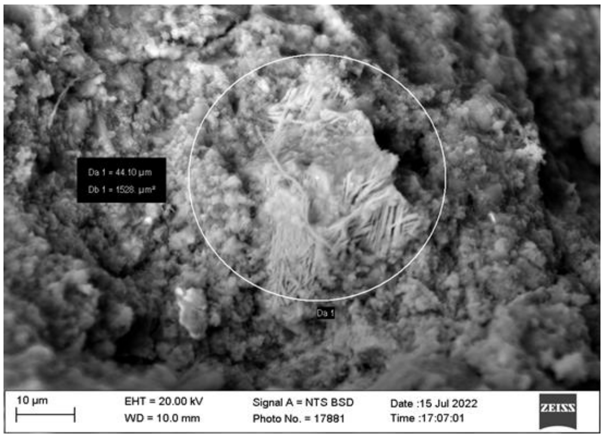


Fig. 3. Relief of the surface of the CPC after 7 days hardening.

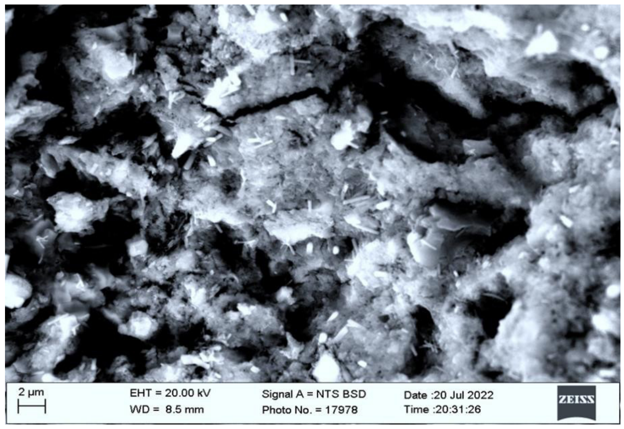


Fig. 4. Relief of the surface of the CPC chip after 28 days hardening.

the reduction of the clinker part in the cement to 35 % due to the replacement of HA composition “25 % ASM + 10 % MS”, in terms of hydraulic activity corresponds to grade 500 according to The Interstate standard 10178 - 85 and 42.5 class of strength according to The Interstate standard 31108 - 2020 [23].

## CONCLUSIONS

The efficiency of the choice of microsilica and ash and slag from dry removal TPP mixtures as ingredients of hybrid additives has been established, which successfully replace 25 - 35 % of clinker part in cements with increase of their hydraulic activity up to mark 500, which is confirmed by the patent of Rep of Uz on useful model No. FAP 02387 dated 29.12. 2023.

The possibility of achieving high durability values of composite Portland cement in the presence of new types of “green” hybrid additives consisting exclusively of techno genic waste, compensating the lack of active clinker part in them due to a favourable combination of constituent minerals and their ability to actively absorb lime with the formation of the same hydrate products arising during the hardening of additive-free Portland cement, which provides compaction of hydrate structure and the formation of a strong cohesive structure.

***Funding***

The work was carried out within the framework of the joint Uzbek-Chinese project A- FA-2021-31 “Joint development of “green” cement composites using ash and slag mixture of power engineering and metallurgy slags”, financed by the Ministry of Innovative Development of the Republic of Uzbekistan. The authors declare that there is no conflict of interest required in this article.

***Acknowledgement***

*The authors express their gratitude to the staff of the accredited testing laboratories of JSC Akhangarancement and JSC Bekabadcement for their assistance in conducting control tests of low-clinker “green” Portland cements with new types of hybrid additives.*

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