Section A-Research paper

INCREASING THE STRENGTH OF PORTLAND CEMENT WITH THE USE OF ACTIVATED MICRO SILICA

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Abstract. Due to the high demand for cement in the construction industry, it is essential to produce high-quality cement. As we know, in the production of cement, a large amount of heat and electricity is consumed, as well as the release of CO_2 gas into the atmosphere. In our research, using activated micro-silica and a new polycarboxylate superplasticizer to reduce cement consumption and increase its strength gave effective results. Activation of micro-silica at a temperature of 60ºC for 180 minutes is indicated as an optimal condition. Due to its microstructure and activated micro-silica, it ensures good secondary hydration with $Ca(OH)_2$, produced as a result of C_3S hydration. And the superplasticizer improves the dispersion of microparticles and reduces water consumption. The compressive strength of the cement composite with micro-silica added between 5 and 10% of conventional cement and activated micro-silica has been studied in samples of 2, 7, and 28 days. Also, the obtained samples were analyzed and compared using IR spectroscopic, SEM, elemental analysis, X-ray diffractometry and thermogravimetric analysis TGA methods. We can see from results Which obtained, it has been found that the compressive strength of the cement composite sample with 10% activated micro silica is 20,6% higher than the initial cement sample.

Keywords: "Activated micro-silica; hydration; modification; polycarboxylate; IR spectroscopy; scanning electron microscope (SEM), thermogravimetric analysis (TGA), X-ray diffractometry".

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INTRODUCTION

As we know, nowadays the large amount of energy demand for the production of cement va a great deal amount of CO_2 gas is released into the atmosphere. Due to the high demand for cement, the addition of 10-12% of blast furnace slag (GBBS) and micro-silica (MS) has given good results to reduce cement consumption. It increases cement paste strength while reducing cement consumption [1-3]. In order to improve the properties of the cement composite, industrial by-products are used, one such by-product is silica fume (SF), which is a by-product of the melting process in the silicon and ferrosilicon industry, and is effective in the production of high-quality concrete [4]. Additives with pozzolanic properties are also added to reduce cement consumption. The use of natural minerals and industrial waste products as additives with properties is economically and ecologically effective. Pozzolanic substances are contain micro-silica and aluminum. Together with cement, they hydrate with $Ca(OH)_2$ formed at normal temperature in humidity conditions and form C-S-H. As a result, this helps ensures high cement strength[5].

In order to improve the properties of cement and also reduce the amount of cement consumption, studied many methods of using micro-silica and its activation were considered. When micro-silica activated in an alkaline environment is added to reduce cement consumption, it helps to improve the physical and chemical properties of concrete[6]. Concrete samples with micro-silica were treated with steam at temperatures of 65, 70, and 75ºC at atmospheric pressure. We can see that the bending and compressive strength of concrete samples with microkermensium is higher than other samples at a temperature of 70ºC[7]. When water/cement ratios (W/C = 0.33, 0.43, 0.53, and 0.63) of natural gravel concrete with and without silica fume were tested, the reduction of the water/cement ratio, the addition of silica fume reduces the porosity between the aggregate and the cement paste and increases the crack resistance [8-10]. An increase in the water-cement ratio increases the porosity between the filler and the cement paste, which leads to an increase in cracks and a decrease in strength.

Replacing 20% of silica fume with cement and adding a superplasticizer creates a higher interface zone than conventional cement paste. In this case, the SF of silica fume has a microstructure and ensures good secondary hydration, while the superplasticizer SP improves the deflocculation of microparticles and reduces water consumption [11]. All cement-based products have weak interactive zones in the filler and cement-based composites. Cracks in the cement composite spread along these weak zones, and over time, under the influence of various external factors, its strength decreases significantly. The addition of silica fume as a mineral additive to the mixture causes a significant change in the microstructure of the interactive zone and improves the physical and mechanical properties of the cement composite. Therefore, the interface zone should be as dense as possible for the cement composite to work for a long time. [12] Sufficient addition of silica fume to the cement mass improves the microstructure of the cement, i.e. increases the amount of CSH in the cement matrix, the amount of $Ca(OH)_2$, and the capillary porosity is much less due to the pozzolanic reaction compared to ordinary portland cement. . Usually, the excess amount of silica fume acts as an inert filler in the cement matrix, because there is not enough $Ca(OH)_2$ that reacts with Si during cement hydration [13,14]. In order to reduce the consumption of cement and improve its quality, silica fume (3, 6, 8, and 10%) and fly ash (10, 15, 20, and 25%) were added to the cement mass. The water/cement ratio (w/s) in all samples was added in the amount of 0.42. The obtained results showed that the cracking intensity of concrete decreased. The ideal ratio of silica fume to cement mass is 8%. Fly ash added to the mixture did not significantly affect the intensity of cracking, but it helped to increase the strength of the concrete [15,16].

The effect of temperature on the hydration of cement composite is also essential, cement (w/c+sf) is 0.25 to 0.45, and silica fume is replaced by 10% to 30% of the cement mass. The hydration process was continuously monitored colorimetrically for 10 days at 20°C. The results showed that the hydration of the cement composite with silica fume is faster compared to the standard cement [17-19]. Improved mechanical properties and impact resistance of self-compacting concrete reinforced with silica fume and recycled steel fibers [20]. The rate of the Pouzalon reaction is proportional to the surface area available for the reaction. Therefore, it is desirable to add nano

SiO_2 particles to obtain concrete with high efficiency [21]. In order to improve the physical and mechanical properties of Portland cement, physical, chemical, and thermal methods of activation of mineral additives added to cement are used. In this study, we can see that the chemical activation of fly ash is more effective than other activation methods when studied [22]. In addition, in order to improve the pozzolanic properties of cement and reduce cement consumption, local kaolinite mineral was thermally activated. When cement is mixed with calcined clay, its mechanical properties depend on the fineness of calcined clay and the addition percentage [23, 24]. Adding 10% silica fume (SF) and 1% superplasticizer polycarboxylate (PC) to ordinary portland cement has a positive effect on the hydration and mechanical properties of cement paste [25]. In order to improve the properties of Portland cement, when modified with polyacrylic ether (PAE) emulsion and silica fume (SF), the porosity and density of the cement matrix are reduced, while the pozzolanic property of silica fume (SF) helps, the reduction of water consumption is due to the polyacrylic ether (PAE) polymer[26]. In addition, in order to increase the strength of the cement composite, 1.5% basalt fibers are also used with silica fume [27].

2. Research methods. The compressive strength of our samples obtained as a result of our research was tested on a 20-ton SERVO PRES device manufactured in Turkey. IR-spectroscopy was obtained and analyzed in the 400-4000 cm^{-1} range on a SHIMADZU IR-Fure spectrometer manufactured in Japan. The number of elements in the samples and the structure of this sample was studied with a MIRA 2 LMU scanning electron microscope (SEM) and the obtained results were analyzed using an X-ray diffractometer.

hermal thermal properties of the received cement composite sample were analyzed by differential-thermal and thermogravimetric methods in the device of the Japanese company SHIMADZU-DTG 60. The derivatogram was studied with the automatically obtained results at the speed of 10 degrees/min, T-900, TG-200, DTA - 1/10, DTG - 1/10 galvanometer sensitivity in the derivatograph.

EXPERIMENTAL PART

When activating micro-silica and taking its cement samples, the first 100 g of micro-silica is taken and added to 200 g of 5% triethanolamine solution and heated at 60°C for 3 hours. The resulting product is isolated on a vacuum filter and dried at 45°C for 1 day and crushed. When preparing cement samples, we added activated micro-silica in amounts of 5 and 10% of the cement mass.

When determining the cement brand, samples were prepared in the following order. First, 1350 g of standard poly fraction sand, 405 g of traditional SEM II/A-I 32.5N cement, 45 g of activated micro-silica, 1% new polycarboxylate superplasticizer by weight of cement, and 225 ml of water were mixed in a special mixer and poured into a 4x4x16 mold. The samples were stored in molds for 24 hours in a special climatic chamber with 98% humidity, and the samples taken from the molds were placed in containers filled with water in a climatic chamber with 98% humidity. Samples for testing were taken on 2, 7, and 28 days and tested in special presses.

RESULTS AND DISCUSSION

The compressive strength of cement samples containing 10% activated micro-silica and a new polycarboxylate superplasticizer is 9% higher than the conventional SEM II/A-I 32.5N cement sample when tested in the first 2 days. When these samples were tested for 7 days, we can see that the micro-silica cement composite is 14.7% stronger than the traditional SEM II/A-I 32.5N cement sample. The compressive strength of the micro-silica cement composite sample at 28 days was determined to be 20.6% stronger than the traditional SEM II/A-I 32.5N cement sample. It is known from the conducted experiments that 5% of the added micro-silica compared to the mass of cement addition of 10% was found to be more effective. (Table 1).

Table-1.

Compressive strength of cement samples
Таблица 1. Прочность на сжатие образцов цемента

№	Daily samples	Conventional SEM II/A-I 32.5N cement sample (N/mm^2)	Faollashtirilgan SEM II/A-I 32.5N brand cement composite with microsilica 5% (N/mm^2)	SEM II/A-I 32.5N brand cement composite with activated microsilica 10% (N/mm^2)
1	2 day	18,7	19,1	20,4
2	7 day	29,3	30,7	33,6
3	28 day	38,6	40,8	46,55

When we examined the setting time of the cement sample with activated micro-silica in the laboratory, we can see that the start of the setting is significantly more effective than the conventional SEM II/A-I 32.5N cement sample (Table 2).

Table-2.

Setting time of cement samples
Таблица 2.Время схватывания образцов цемента.

№	Daily samples	Conventional SEM II/A-I 32.5N cement sample (min)	SEM II/A-I 32.5N grade cement composite with activated microsilica 5% (min)	SEM II/A-I 32.5N grade cement composite with activated microsilica 10% (min)
1	Amount of water added	*121 ml*	*120 ml*	*119 ml*
2	Start of solidification	131	142	165
3	The end of hardening	180	195	215

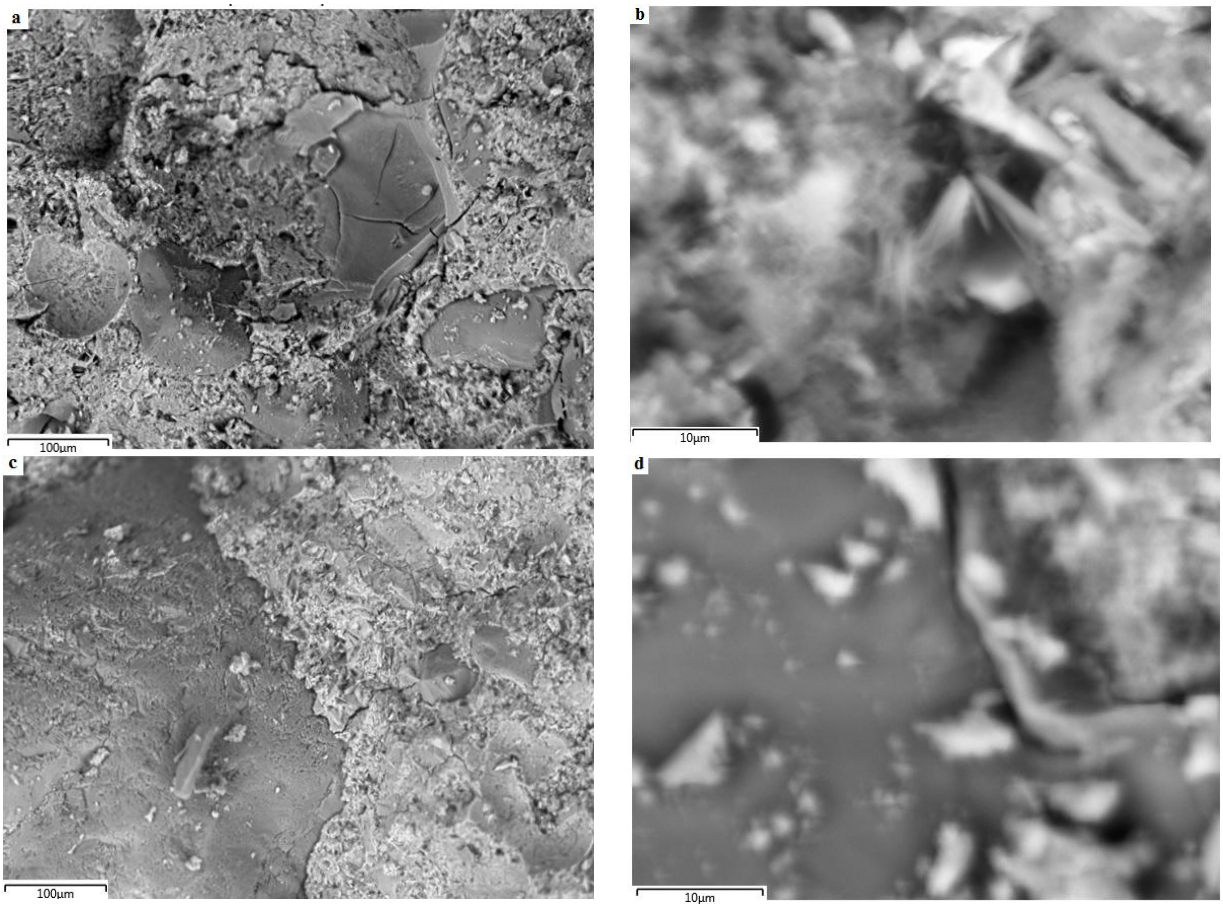


Fig. 1. SEM results. Microsilica cement composite (a, b), traditional cement sample (c, d).

Рис. 1. Результаты СЭМ. Цементный микрокремнеземный композит (а, б), образец традиционного цемента (c, d).

The strength of the cement matrix increases due to the fact that the crystals of CSH and calcium sulfoaluminate fill the spaces between the cement matrix. Activated micro silica actively participates in hydration reactions. In general, small particles of micro-silica enter the space between cement grains and fillers and enter into a pozzolanic reaction with $Ca(OH)_2$, which leads to a decrease in the capillary pores of the cement composite [28, 29]. In this case, $Ca(OH)_2$, mainly formed during the initial hydration, plays an important role in the formation of C-S-H and calcium hydro-aluminates (C-A-H) as a result of the pozzolanic reaction with active micro-silica.

In the obtained SEM results, we can see that the crystals of C-S-H and calcium hydro aluminates have a more perfect appearance compared to the conventional cement sample.

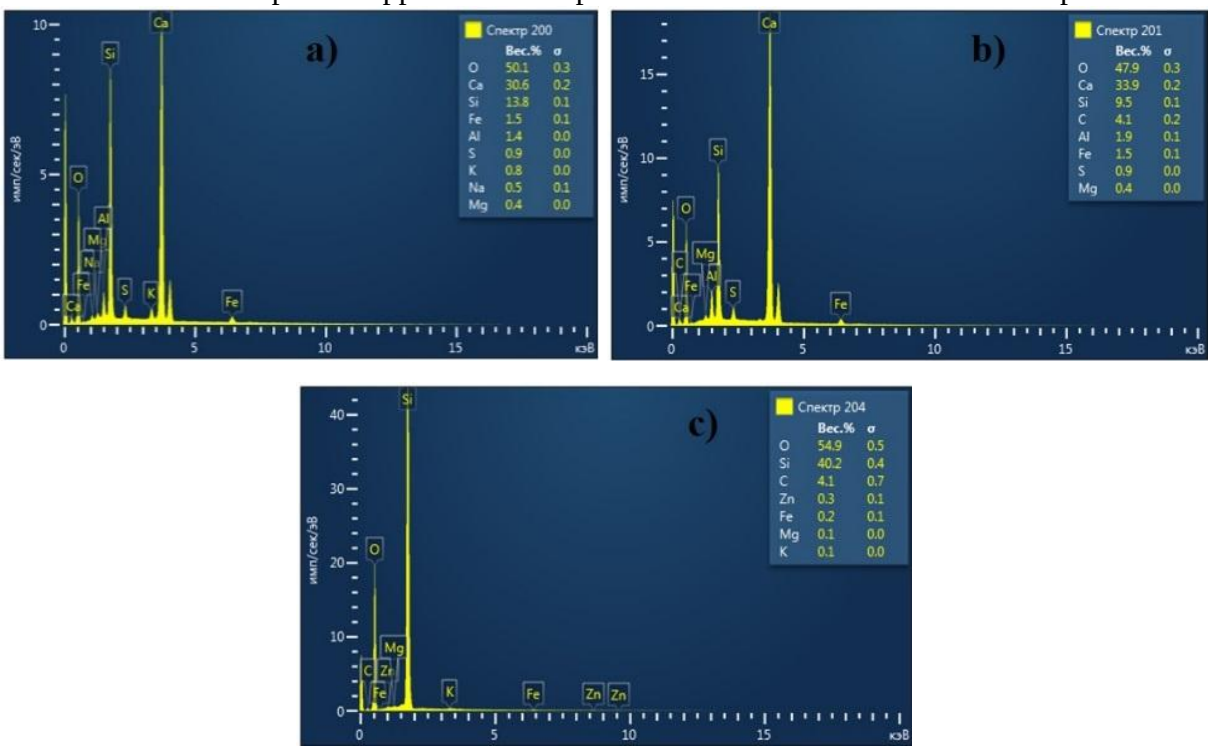
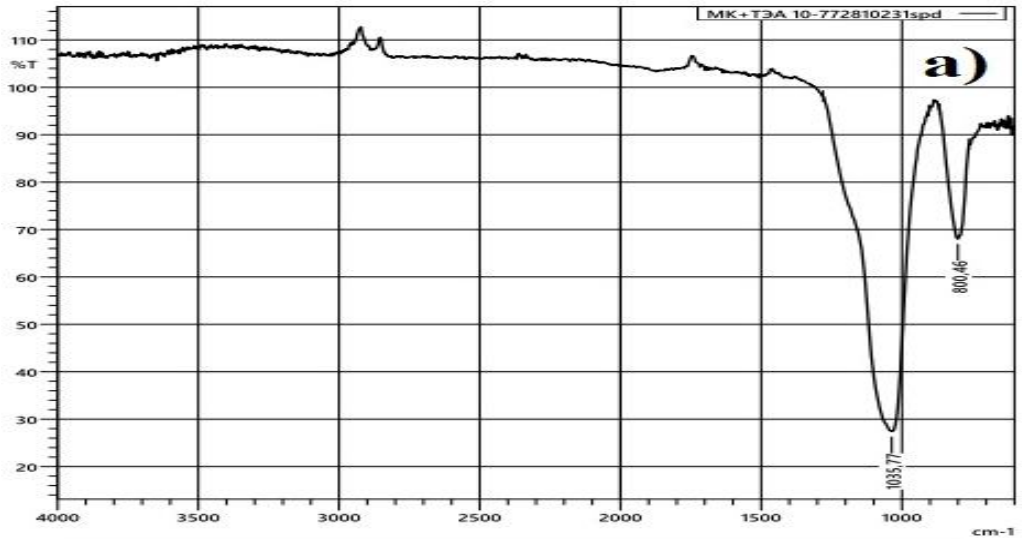


Fig. 2. Element analysis results. a) traditional cement sample. b) cement composite with micro-silica. c) activated micro-silica

Рис. 2. Результаты элементного анализа. а) образец традиционного цемента. б) цементный композит с микрокремнеземом. с) активированный микрокремнезем

When the obtained samples are analyzed for elements, we can see the amount of carbon element of 4.1% in the cement composite with micro-silica in Fig. 2-b and in the activated micro-silica in Fig. 2-c. This is due to triethanolamine used to activate micro calcium and superplasticizer added in the preparation of cement paste. Active SiO_2 in activated microkermensium forms C-S-H crystals due to high hydration with $Ca(OH)_2$ crystals formed from C_3S hydration. This ensures high cement strength.



Vibrations in the 800.46-1035.77 cm-1 range were observed in the IK spectroscopic analysis of activated micro-silica. As we know from the literature, it corresponds to symmetric and asymmetric vibrations of Si-O-Si and Si-O bonds as well as Si-O-Al bonds in the range of 1100-750 cm-1 [30, 31].

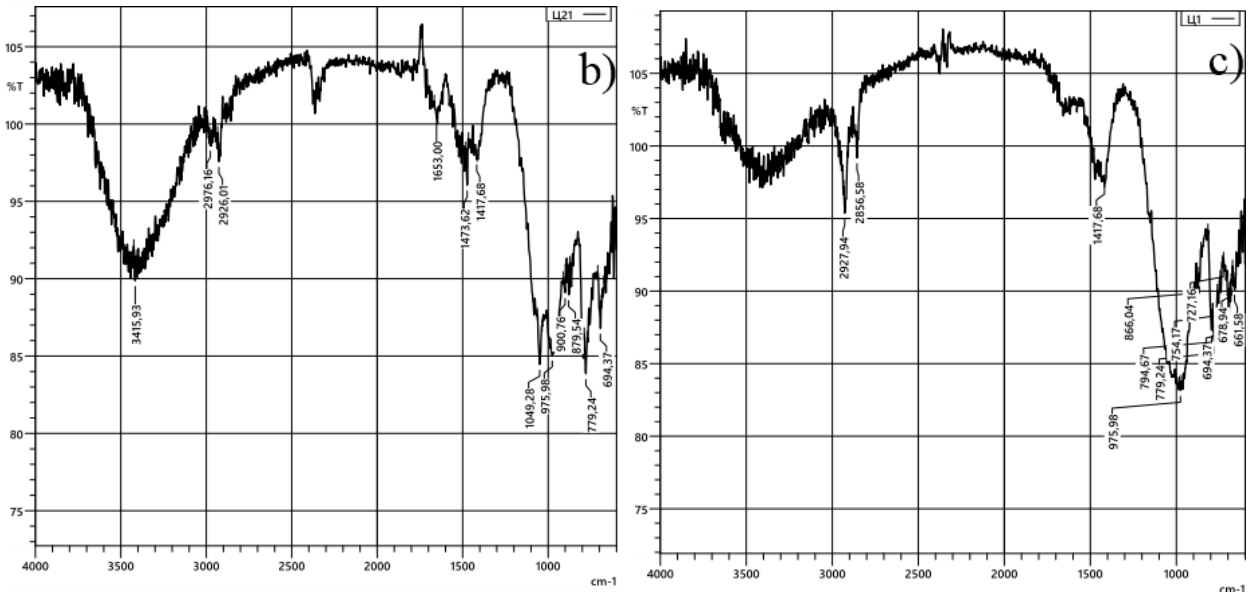


Fig. 3. IR spectroscopic analysis results. a) activated micro silica and b) cement composite with micro-silica. c) traditional cement sample

Рис. 3. Результаты ИК-спектроскопического анализа. а) активированный микрокремнезем и б) цементный композит с микрокремнеземом. с) образец традиционного цемента.

According to the IR spectrum analysis of activated micro silica cement composite and conventional cement sample, wavelengths in the 694-779 and 879 cm^{-1} regions are full of symmetric and asymmetric vibrations of Si-O-Si and Si-O bonds. fainting. At this wavelength of 900-975 and 1049 cm^{-1}, we can see the vibrations of Si-O-Al bonds and better crystallization of calcium hydro sulfoaluminate. Wavelengths of 1417-1473 cm^{-1} indicate the presence of carbonate, calcium bicarbonate, and sulfite groups, as a result of which hydrocarboaluminates and hydrosulfocarbosilicates are formed, which increases the strength of cement stone. 2926-2976 cm^{-1} wavelengths indicate the presence of free water molecules. In contrast to the conventional cement sample, it is related to the vibrations of the SiO_4 tetrahedron bonded to OH- at a wavelength of

3415.93 cm^{-1}, indicating a high amount of $Si(OH)_4$ hydro silicates and sub-microcrystals. This increases the strength and water resistance of the cement composite.

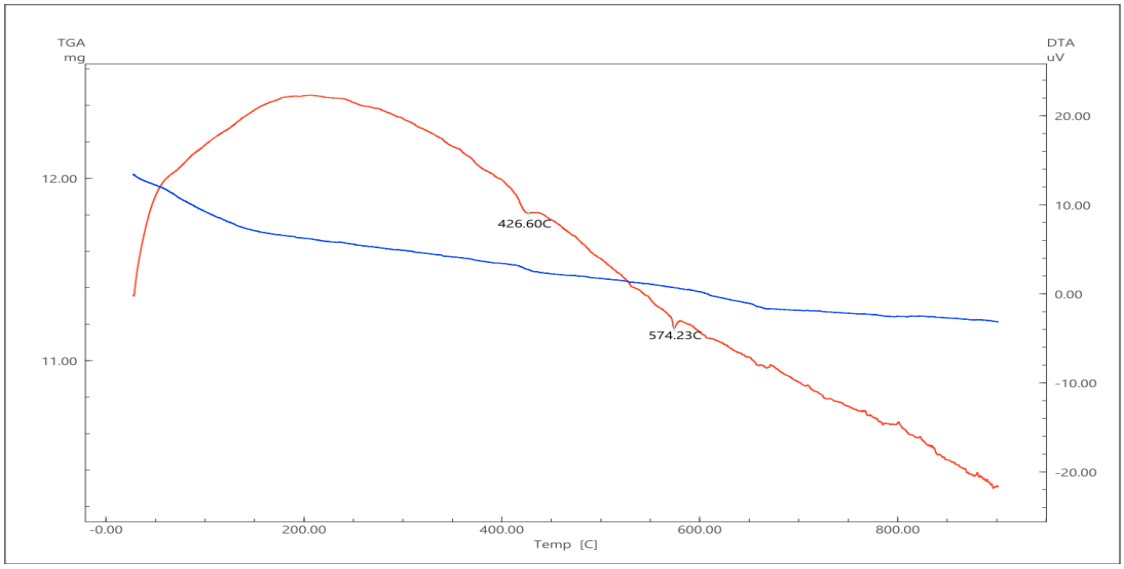
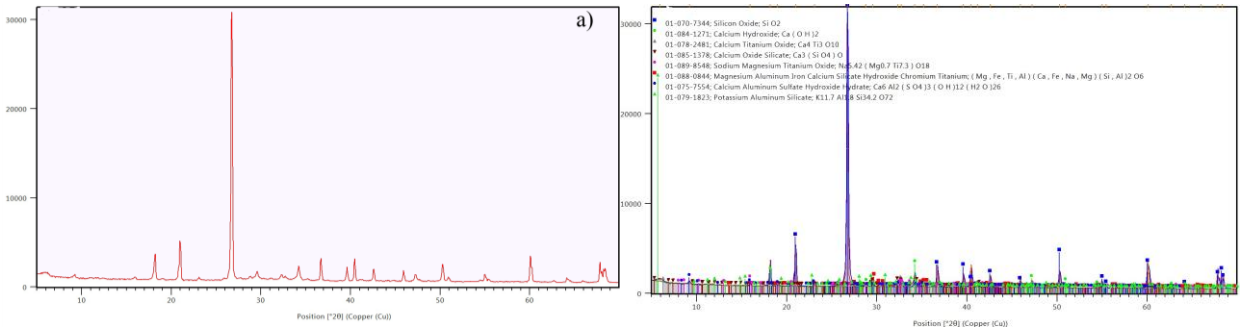


Fig. 4. Thermogravimetric (TGA) and differential thermal analysis (DTA) of microsilica cement composite

Рис. 4. Термогравиметрический (ТГА) и дифференциальный термический анализ (ДТА) микросиликацементного композита

The thermogravimetric analysis of the micro-silica cement composite shows that the experiment was conducted at a temperature of 900 °C for 90 minutes. During this time, mass loss was observed in 3 intense peaks in TGA. In the 1st intensive peak at a temperature of 27.47-239.14°C, excess moisture and 3.111% mass loss due to dehydration of CSH were observed. 1.855% of the mass was lost in the 2nd intensive peak at a temperature of 239.14-540.36°C due to partial dehydration of CSH and decomposition of $Ca(OH)_2$, in the 3rd intensive peak 540.36-901.65°C At C temperature we can see that 1.764% of the mass is lost mainly due to the decomposition of $CaCO_3$(Figure 4).

It is known from the differential thermal analysis of the micro-silica cement composite that energy absorption was observed in the range of 426.60-574.23 °C, and its highest heat of absorption was observed at a temperature of 574.23 °C. (Fig. 4)



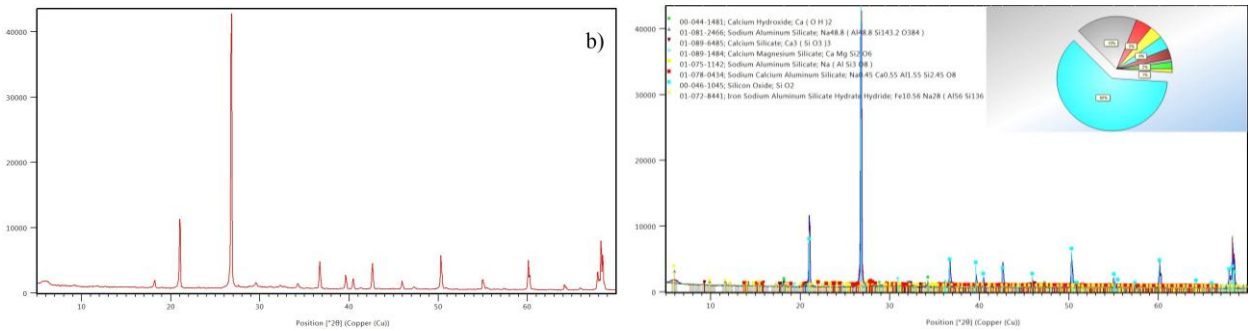


Fig. 5. X-ray diffractometer analysis results. a) traditional cement sample b) cement composite with micro-silica.

Рис. 5. Результаты рентгенодифрактометрического анализа. а) образец традиционного цемента б) цементный композит с микрокремнеземом.

Diffraction peaks of $Ca(OH)_2$ in the 18°, 28° and 34° areas of activated micro silica cement composite (Fig. 5-b), $Ca(OH)_2$ in the 18°, 34° and 47° areas of the conventional cement sample (Fig. 5a) we can see that is weak from the diffraction peaks it. As a result of the hydration of elite C_3S in cement, $Ca(OH)_2$ crystals are formed, but the diffraction peaks are weak due to active SiO2 combining with Ca^{+2} ion during hydration to form C-S-H [32]. Over time, the increase of C-S-H and calcium sulfoaluminate hydration products in the cement matrix ensures an increase in cement strength. Ca(OH)2 crystals that remain unhydrated have a negative effect on cement strength. As a result of the conducted research, it was found that the cement composite with the addition of activated micro silica is 20.6% stronger than the traditional cement sample.

CONCLUSION

As a result of the conducted research, 5 and 10% activated micro-silica were added to the cement mass, and its compressive strength was increased by 4.1 and 20.6%, respectively. When the cement composite with micro-silica was studied using SEM analysis, it was found that the crystals of C-S-H and calcium hydro aluminates have a more perfect appearance compared to the traditional cement sample, and it was proved that the added micro silica increases the composite strength.

The results obtained according to the IR spectrum analyses of the activated micro silica cement composite show that the amount of $Si(OH)_4$ hydro silicates and sub-microcrystals are higher than that of the traditional cement sample. This increases the strength and water resistance of the cement composite. Also, the results of x-ray diffractometric analysis showed the formation of aluminosilicates and calcium hydro silicates CSH at the expense of $Ca(OH)_2$ and active silicon dioxide in the matrix of the cement composite with the addition of activated micro-silica.

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