Original Article

Studying the Increase in Portland Cement Quality Based on the Activated Micro-Silica

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Abstract - In this paper, in order to reduce the consumption of cement, which is considered important for the construction industry, and to increase its strength, we added 10% of the cement mass by activating the micro silica produced as a secondary product in the production of ferrosilicon alloys. Activation of micro silica in the presence of 10% triethanolamine at a temperature of 60 °C for 180 minutes is indicated as an optimal condition. In order to study the effect of activated microsilica on the strength of the cement sample, conventional cement and non-activated microsilica cement samples were compared. In addition to activated microsilica, calcium lactate and a new polycarboxylate superplasticizer were used. As a result of the hydration of C2S and C3S in cement, the formation of slightly weak Ca(OH)₂ crystals and its pozzolanic reaction with active microsilica, the good progress of secondary hydration, and the formation of CSHs were studied. Compressive strength was studied in 2, 7, and 28-day samples. Also, in order to study the microstructure, amount of elements, and thermal stability of the obtained samples, the samples were studied and compared using different analytical methods: IR spectroscopic, SEM, elemental analysis, x-ray diffractometry and thermogravimetric analysis TGA methods. It was determined that the cement sample with activated microsilica is 23.7% stronger than the conventional SEM II/A-I 32.5N cement sample and 30.1% compared to the cement sample with non-activated microsilica.

Keywords - Activated micro silica, Pozzolanic, Hydration, Modification, Polycarboxylate.

1. Introduction

We know that one of the most important products in the construction industry is cement. In the process of cement production, a lot of heat and electricity is required, as well as the release of CO_2 gas into the atmosphere. Due to the high demand for cement in the construction industry, in order to reduce the consumption of cement, adding various mineral additives reduces the consumption of cement. It increases the strength of the cement paste. These mineral additives have shown good results when added to 10-12% of blast furnace slag (GBBS) and micro silica (MS) [1]. Additive cement materials serve as a key resource in reducing environmental problems and reducing the cost of cement [2]. In order to improve the strength of cement stone, various industrial byproducts are also used; one such by-product is silica fume (SF), which is a by-product separated during melting in the silicon and ferrosilicon industry and is effective in the production of high-quality cement and concrete [3]. In order to reduce the consumption of cement and increase its strength, additional products with different pozzolanic properties are used. The use of natural mineral raw materials and various industrial waste products as pozzolanic additives is economically and ecologically effective. Pozzolanic substances are substances containing micro silica and aluminum. Together with cement, they hydrate with Ca(OH)₂ formed at normal temperature in moist conditions and form C-S-H. This ensures the high strength of cement [4,5]. When micro silica products such as micro silica, fly ash, and dominance slag are added to cement, the primary and secondary hydration process goes well. It has less porosity and water permeability than ordinary Portland cement paste [6]. It has been mentioned that microsilica increases the hydration of C3S and C3A in the first few hours of added cement [7]. Mechanical strength is evaluated based on compressive strength, tested by acid resistance, electrical resistance, and air permeability. Portland cements up to 40% fly ash, 15% silica fume, and 70% granular furnace ash mechanical strength is tested based on compressive strength. as its strength is higher compared to cement with additives [8]. Silica fume (silicate dust) affects the state of crystallization and the level of placement of CH on the concrete surface in the first days of cement hydration [9]. CSH formed by cement hydration plays an important role in cement strength and influencing its properties. Hydration of micro silica cement is faster in the first 3 and 7 days in terms of both Ca(OH)₂ content and nonevaporable water content. The hydration reaction with

microsilica mainly occurs in 3 and 7 days. In order to accelerate the cement hydration time, high temperature was also used to increase the secondary hydration of Ca(OH)2 with additives [10]. When treated with high temperature or pressure, the strength of added cement was observed to increase significantly [11]. The use of silica fume (SF) and fly ash (FA) was studied experimentally in order to improve the mechanical and durability properties of concrete. Mechanical properties (compressive and bending strength) and durability properties (permeability of water and chloride ions) were studied by replacing 15-45% fly ash (AF) with 5-15% silica fume (SF) for ordinary Portland cement [12]. The optimal amount of replacement of zeolite and silica fume with respect to cement mass is 20 and 7%, respectively. If the water absorption level of concrete with zeolite is 14%, the water absorption level of concrete with silica fume is 3% less than the original concrete [13]. In order to increase the strength of cement and reduce the demand for cement, methods of using activated microsilica have been considered. The activation of microsilica in an alkaline environment helps to improve the physicochemical properties of concrete and reduces cement consumption [14]. When the cement samples with micro silica are treated with steam at different temperatures of 65, 70, and 75°C, we can see that the bending and compressive strength of the cement samples at 70°C is higher than other samples [15]. The water/cement ratios of the prepared concrete (W / C = 0.33, 0.43, 0.53, and 0.63) and the cracking strength of the concrete with and without the addition of microsilica were tested. Reducing the water/cement ratio, the addition of micro silica reduces the porosity between the filler and the cement paste and increases the resistance to cracking [16].

An increase in the water/cement ratio causes an increase in the pores between the filler and the cement paste. This leads to an increase in cracks and a decrease in strength. Cement paste made with a 20% addition of micro silica to cement mass and the presence of superplasticizer creates a higher interactive zone compared to ordinary Portland cement paste. In this case, micro silica ensures good secondary hydration due to its microstructure and pozzolanic properties, while superplasticizer SP improves the deflocculation of microparticles and reduces water consumption [17]. Suppose the additional mixing time of cement paste has the effect of improving the mechanical properties of cement. In that case, the activation of slag micro silica in a high-concentration alkaline solution accelerates the hydration process, reduces porosity, and increases mechanical properties [18]. All cement-based concrete products and cement composites have weak interface zones. The addition of silica fume as a mineral additive to concrete products and cement composites causes a significant change in the microstructure of the interactive zone. It improves the physical and mechanical properties of the cement composite [19]. Adding a sufficient amount of silica fume relative to the cement mass improves the microstructure of the cement composite. 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 [20, 21]. The effect of temperature on the hydration of cement composite is also important. It was found that the hydration of the cement composite with silica fume is higher than the results of the reference cement, which was continuously observed colorimetrically for 10 days at 20°C [22-24]. In order to reduce the consumption of cement and increase its strength, the addition of alkaline-activated microsilica has a good effect. According to the obtained results, it shows that this activator is better than activators such as sodium hydroxide, sodium carbonate, and liquid glass [25].

2. Materials

Activated microsilica, triethanolamine solutions, cement samples, Ca(OH)², polycarboxylate superplasticizer, GIPAN (hydrolyzed polyacrylonitrile), calcium lactate and sulfuric acid were used in this research. All substances were used "technically clean".

3. 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 amount of elements in the samples and the structure of this sample were studied with a MIRA 2 LMU Scanning Electron Microscope (SEM) and the obtained results were analyzed using an X-ray diffractometer. The thermal properties of the obtained 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, and DTG - 1/10 galvanometer sensitivity in the derivatograph.

4. Experimental Part

When activating microsilica with triethanolamine solutions and taking its cement samples, the first 100 g of microsilica is removed and added to a 5% triethanolamine solution and heated for 3 hours. The resulting product is isolated on a vacuum filter, dried at 45°C for 1 day and crushed. It was obtained by the method of neutralization of Ca(OH)₂ to obtain calcium lactate based on lactic acid. In this case, the reaction was carried out in a ratio of 2:1, and calcium lactate was isolated by crystallization of the resulting solution. The separated calcium lactate was dried at a temperature of 45-50 °C. To obtain a new polycarboxylate superplasticizer, GIPAN (hydrolyzed polyacrylonitrile) and sulfuric acid were added in a ratio of 1.45:1 and mixed at 60°C for 45 minutes. 230 ml of 35% formaldehyde solution was added to the prepared sample and heated at atmospheric pressure and temperature of 90°C for 4 hours with constant stirring. After that, the resulting product was dried in a drying oven at a temperature of 60°C and ground. When determining the strength of cement, samples were prepared based on the

requirements of GOST 197-(1,2,3)-2020. First, 1350 g of standard poly fraction sand, 405 g of traditional SEM II/A-I 32.5N cement, 10% of activated micro-silica, i.e. 45 g, 1% of calcium lactate and new polycarboxylate superplasticizer, 4.5 g, and 220 ml water was added, mixed, and samples were poured into a 4x4x16 mold. Samples for testing were taken on 2, 7, and 28 days and tested in special presses.

5. Results and Discussion

The compressive strength of the obtained cement samples was tested in a special press, "ATOM TEKNIK 20 t". We can see that the activated micro silica cement composite sample is 23.7% stronger than the conventional SEM II/A-I 32.5N cement sample and 30.1% stronger than the non-activated micro silica cement sample.In order to study the microstructure, amount of elements, and thermal stability of the obtained samples, the samples were studied and compared

different analysis methods [26]. When the using microstructure of cement samples is observed in SEM, we can see in Figure 2 that the cement sample with activated microsilica, the cement sample with non-activated microsilica, and the conventional cement samples have a complex structure. Small particles of activated micro silica fill the capillary spaces between cement and aggregates and increase the strength of the cement matrix. In this case, active SiO₂ contained in micro silica leads to a decrease in the amount of Ca(OH)2 and an increase in C-S-H due to the pozzolana reaction with Ca(OH)2, which is a weak product of hydration [26, 27]. The obtained SEM results were carried out on 28-day cement samples; in Figure 2(c) we can see that the cement sample with activated microsilica has a complex structure of crystals of C-S-H and calcium hydroaluminates compared to the rest of the cement sample.

Table 1.	Compressive	strength o	of cement	samples
Table I.	Compressive	sucugui	or coment	Sampics

No	Cement samples	SEM II/A-I 32,5N Poly	Polyfraction	Polyfraction Water sand (gr) (ml)	Microsilica (gr)	Activated microsilica (gr)	Calcium lactate (gr)	Calcium lactate (gr)	Compressive strength of cement samples		
		Cement	sand (gr)						2 day	7 day	28 day
		(gr)				ý	ý	ý	(N/mm^2)	(N/mm²)	(N/mm^2)
1	1	450	1350	225	-	-	-	-	14.2	27.4	33.6
2	2	405	1350	225	45	-	-	-	15.4	28.6	36.7
3	3	400.5	1350	220.5	-	45	4.5	4.5	24.1	37.1	48.1



Fig. 1 (a) The results of the compressive strength of the samples SEM II/A-I 32.5N cement sample, (b) Microsilica cement composite, (c) Activated microsilica cement composite



Fig. 2 (a) SEM results SEM II/A-I 32.5N cement sample, (b) Microsilica cement composite, (c) Activated micro silica cement composite

5.1. EDS analysis: The Surface Elemental Analysis of Activated Microsilica Cement Composite was Done to Identify the Elemental Compositions of the Selected Cement Composite

According to the results of the elemental analysis of the cement samples, we can see that the amount of Si element in the Activated micro silica cement composite is 2% higher than the Si element in the conventional SEM II/A-I 32.5N cement sample in Figures 3 and 4.

In this case, SiO_2 in activated microsilica forms CSH crystals as a result of a secondary hydration reaction, with $Ca(OH)_2$ formed as a result of primary hydration. In the traditional SEM II/A-I 32.5N cement sample, the low amount of Si element causes the incomplete pozzolanic reaction of $Ca(OH)_2$ with SiO₂.

A high content of $Ca(OH)_2$ in the cement matrix reduces the strength of the cement stone. Active SiO₂ contained in activated microkermensiom forms crystals of C-S-H and C-A-H due to the reduction of Ca(OH)₂ formed as a result of initial hydration of cement, i.e. pozzolan reaction. This increases the strength of the cement matrix.

6. IR Analysis

The structural analysis of activated microsilica cement composite was confirmed by the IR analysis. The obtained IR results of activated microsilica cement composite are indicated in Figure 4. The results of IR spectroscopic analysis of the samples show that the vibration of Si-O and Si-O-Si bonds at wavelengths of 669-694 and 775cm⁻¹ indicates the formation of CSHs. Vibrations of Si-O-Al bonds at this wavelength of 873-979 cm⁻¹ mean the presence of crystals of AFt, AFm, and calcium hydrosulfoaluminate. 1417-1471 and 1506 cm⁻¹ indicate the presence of CaCO₃, gypsum and sulfite groups [28, 29]. Members of this group form hydrocarboaluminates and hydrosulfocarbosilicates and contribute to the strength of the cement matrix. 2928-2931 cm⁻¹ wavelengths indicate the presence of free water molecules.

The results of IR spectroscopy analysis of activated micro silica cement samples and non-activated micro silica cement samples are similar. It differed from the traditional cement sample in that it differed in the wavelength range of 3373.5 cm⁻¹ with SiO₄ vibrations connected with OH-. It shows the presence of Si(OH)₄ hydrosilicates. This increases the strength of the cement matrix.





Fig. 3 EDS analysis of A-I 32.5N cement sample (a)



Ca-K Ti-K

Fe-K





🗖 100µm

N-K





 $5.0*10^{5} - 4.0*10^{5} - 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5} + 7.0^{5}$

🗖 100µm

Element	Line	Mass%	Atom%
С	Κ	9.72±0.02	15.78±0.03
Ν	Κ	nd	nd
0	Κ	48.91±0.04	59.62±0.05
Mg	Κ	0.43±0.00	0.34±0.00
Al	Κ	0.96 ± 0.00	0.69 ± 0.00
Si	Κ	20.33±0.02	14.12±0.01
S	Κ	0.40 ± 0.00	0.24±0.00
Ca	Κ	17.99±0.02	8.76±0.01
Ti	Κ	0.09 ± 0.00	0.04 ± 0.00
Fe	K	1.17 ± 0.01	0.41±0.00
Total		100.00	100.00

Fig. 4 Activated microsilica cement composite







Fig. 5 (a) IR spectroscopic analysis results. SEM II/A-I 32.5N cement sample, (b) Microsilica cement composite, (c) Activated microsilica cement composite

7. Thermogravimetric Analysis (TGA) Analysis

The thermogravimetric (TGA) analysis of activated microsilica cement composite was done to explore the thermal performances bestowed by the selected modification method. Thermogravimetric curves of activated microsilica cement composite Figure 5 and its thermogravimetric properties. The values in consumed energy ($\mu V^*s/mg$) weight loss percentage (%) and weight loss (mg) for activated microsilica cement composite were measured during the temperature changes, from 100-1000 °C for 90 min.





Fig. 5 (a) Thermogravimetric (TGA) and differential thermal analysis (DTA) SEM II/A-I 32.5N grade cement sample, (b) Activated microsilica cement composite



The analysis of thermogravimetric analysis of cement samples shows that the experiment was conducted at a temperature of 800 °C for 90 minutes. During this time, mass loss was observed in 4 intense peaks in the TGA of the conventional cement sample. 2.397% mass loss due to excess moisture and water in gypsum was observed in intensive peak 1 at a temperature of 31.95-189.77°C. 1.341% mass was lost due to dehydration of CSH in the 2nd intensive peak at a temperature of 189.05-417.78°C, decomposition of Ca(OH)₂ in the third intensive peak at a temperature of 417.78-455.23°C we can see that 0.832% mass is lost due to In intensive peak 4, we can see 1.523% mass loss due to the decomposition of CaCO₃ at 456.69-710.51°C. Energy absorption was observed in the range C, and its highest heat of absorption was observed at a temperature of 455.22°C. (Figure 5a). According to the thermogravimetric analysis of the cement sample with activated micro silica, mass loss was observed in 3 intense peaks. In the first intensive peak, 5.526% mass loss is observed due to the loss of chemically and mechanically combined water in the temperature range of 29.84-178.36°C. In the second intense peak at a temperature of 177.62-450.82°C, 2.303% mass loss is observed due to dehydration of C-S-H crystals and decomposition of Ca(OH)₂. In the 3rd intensive peak, 1.425% mass loss is observed due to the decomposition of CaCO₃ at a temperature of 450.09-792.23°C. In the differential thermal analysis, energy absorption was observed in the range of 113.81, 456.11, and 584.14°C, and its highest heat of absorption was observed at 456.11°C. (Figure 5b).

8. X-ray Diffractometer Analysis

According to the results of the X-ray diffractometer analysis of cement samples, the results of the hydration of conventional cement and cement samples with activated microsilica were studied (Figure 6). We can see that the amount of Ca(OH)₂ formed by the hydration of the cement sample with activated micro silica is 1.7% less than the amount of Ca(OH)₂ formed by the hydration of the conventional cement sample possible Ca(OH)₂ with weak crystals is also formed during cement hydration. High Ca(OH)₂ content in cement matrix reduces cement strength. As a result of the secondary hydration reaction of activated micro silica with Ca(OH)₂, it forms C-S-H crystals and increases cement strength. (Figures 6a and 6b).

9. Conclusion

In this scientific study, 10% of non-activated microsilica and activated microsilica were added to the mass of conventional cement, and the compressive strength of the samples was compared with the conventional cement sample of SEM II/A-I 32.5N brand. According to the obtained results, it was determined that the cement samples with activated microsilica are 23.7% stronger compared to the traditional SEM II/A-I 32.5N cement sample and 30.1% compared to the non-activated microsilica cement sample. When studying the microstructure of the samples using SEM analysis, we can see that the cement sample with activated microsilica, the cement sample with non-activated microsilica, and the traditional cement samples have a complex structure. It was found that the crystals of C-S-H and calcium hydro aluminates in the cement sample with the addition of activated micro silica have a more perfect appearance compared to the conventional cement sample. According to the IR spectroscopic analysis of the obtained cement samples, in contrast to the conventional cement sample, the activated microsilica cement sample differed in the 3373.5 cm⁻¹ wavelength region with OHbonded SiO₄ vibrations. Si it means that the content of (OH)₄ hydrosilicates is high. This caused the strength of the cement sample to be high. X-ray diffractometric analysis of SEM II/A-I 32.5N traditional cement sample and cement sample with activated microsilica was performed. In this case, it was proved that the amount of Ca(OH)₂, which is somewhat weak, in the activated microsilica cement sample is less than that of the traditional cement sample. This is due to the formation of aluminosilicates and calcium hydrosilicates CSH due to the pozzolanic reaction of Ca(OH)₂ with active silicon dioxide.

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Authors' Contribution Statement

Mukimov A.S: Writing – Original Draft. Turaev Kh.Kh: Reviewing and editing paper. Tojiev P.J: Reviewing and editing paper. Karimov M.U: Software, Validation.: Mukimov A.S: Writing – Original Draft, Conceptualization, Investigation, Visualisation.

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