



Research on the effect of microsilica on the properties of the cement-sand mixture

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Abstract. The article presents a study of changes in the mineralogical composition of cement when adding microsilica of different concentrations using diffractometer DRON-3 with CuCa-radiation, β -filter. X-ray phase analysis on a semi-quantitative basis was carried out on powder sample diffractograms using the method of equal weights and artificial mixtures. The work examines the effects of microsilica on the mineralogical composition of cement at the microstructural level. The aim of the study was to improve the physico-chemical properties of the cement. Solutions with different percentages of microsilica additives (0 %, 10 %, 20 %, 30 %) were made for the analysis. The conducted research makes it possible to determine the effect of microsilica in different concentrations on the properties of the cement composition. The introduction of additive into products is introduced to increase mechanical properties, reduce porosity, increase water resistance and durability of products, possibly due to binding of calcium hydroxide in the structure of hardening cement stone by dispersions of microsilica. In general it can be concluded from the results of studies that the additive contributes to increasing the strength of the mixture, accelerating hardening. Consequently, the additive is one of the most effective additives to increase the strength.

Keywords: modified additive, microsilica, mineralogical composition, different concentrations, concrete.

1. Introduction

In the last few years, data have been obtained and published by many researchers showing that the combination of additives with cement provides a synergistic effect in concrete, allowing the best strength results to be obtained. The use of microsilica, a waste product from ferroalloys production, in different types of concrete has aroused great interest among both builders and researchers today. The positive effects of microsilica as a fine-grained active mineral admixture and the need for its use in concrete were described in publications as early as 40 years ago.

Microsilica is a by-product of the metallurgical process of melting ferrosilicon and its alloys, produced by reducing high-purity quartz with carbon in electric furnaces. In the process of melting silicon alloys some part of silicon monoxide SiO goes into gaseous state and undergoes oxidation and condensation to form an extremely fine product in the form of spherical particles with high content of amorphous silica [1].

One of modern trends in production of high-strength concretes is modification of binding structure of construction composites with additives of various composition and morphology. Particularly effective in this regard are ultradisperse additives based on microsilica.

Ultra-dispersible additives generally do not exist in a ready-to-use form, but must be synthesised. Ultradisperse additives generally do not exist in a ready-made form, but must be synthesised and the most important of which are the properties of the stone to be synthesised. application, stability in time, a similar time stability, similar crystallochemical structure with synthesised material, comparability of its particle size with the particles of cement gel and gel

pores, etc. The effectiveness of the additives is assessed by measuring the mechanical, chemical and mechanical properties of the cement paste. The effectiveness of the use of additives is assessed by means of the improvement in the mechanical and physico-chemical properties of the materials being modified properties of the modified materials [2].

The particles of microsilica, whose size is 0.5 ... 0.05 microns, can fill the gaps between the particles of cement and aggregates. The microsilica particles create a tightly bound shell of water around themselves, which prevents water migration to the surface of the concrete and consequently reduces the delamination of the concrete mix and the shrinkage of the concrete. The addition of microsilica increases water resistance by 25-50 % and sulphate resistance by 90-100 %. Adding only 6 % of microsilica provides concrete frost resistance grade F300 at B/C = 0.45 [3].

The use of complex additives is now generally accepted as an effective way to improve the properties of cement concrete. In most cases additives are nowadays the obligatory component of concrete mixture. Analysis of scientific literature shows that additives that increase the rate of setting and hardening of cement are in demand, so the interest in developing new, competitively capable accelerating additives is not diminishing [4].

The creation and introduction into production of innovative materials is directly linked to research into their internal structure, which in turn determines the set of performance properties and their stability. Understanding the microstructure of a material is the key to understanding its properties and characteristics and how they relate to technological processes. Over the past few decades, tremendous progress has been made in methods for determining the microstructure of cement materials, especially in making these methods more quantitative. Quantification is very important because most cements have a roughly similar composition. Unfortunately, it is still not possible today to characterize the microstructure of a cement material with the same accuracy that can be obtained in mechanical characterization testing [5].

The aim of the study is to investigate the change in mineralogical composition of cement with the addition of microsilica in different concentrations.

In order to achieve the objective the following tasks were carried out:

1. The composition with optimum ratio of components was selected;
2. A comparative mineralogical analysis of samples with the addition of components in different percentages and without the addition of component;
3. Selection of the optimum sample with addition of component in percentage ratio;

Microstructural analysis of the mineralogical composition of each sample with the added component (microsilica):

Type-1: Control sample without additives;

Type-2: Sample with added microsilica (10 %)

Type-3: Sample with added microsilica (20 %);

Type-4: Sample with microsilica additive (30 %)

2. Methods

Four samples with the addition of (microsilica) of different percentages were designed. The cement content varied with the addition of microsilica, depending on the percentage content. Microsilica was added to the mixture in the amounts of 10, 20, and 30 % of the cement mass [6].

The raw materials produced on an industrial scale were mainly used in laboratory tests. Their main characteristics as defined by the standards in force are given below [7].

Portland cement M400 brand CEM I 42,5 H (Karaganda (Central Asian Cement), corresponding to the requirements of GOST 31108-2016 [8].

A waste condensed microsilica MK-85, corresponding to the requirements of GOST 10178 [9], was used as an additive.

Tap water corresponding to the requirements of GOST 23732-2011 [10] was used as mixing water for concrete mixture.

Table 1 presents technological compositions of compared types of cement-sand samples for X-ray diffraction and X-ray phase analysis.

Type	Cement	Microsilica	Water/cement ratio
Type-1	100	0	0.3
Type-2	90	10	0.3
Type-3	80	20	0.3
Type-4	70	30	0.3

As the samples for XRF and XRF analysis are relatively small in size, the control of the water-cement ratio becomes particularly important. Therefore, all tools and appliances used for mixing the samples were treated with a damp sponge before mixing (Figure 1). The components were weighed on a high-precision analytical scale in order to determine the weight of the samples (Figure 1). Figure 2 shows the finished samples.



Figure 1 – Sample preparation for XRF and XRF analysis

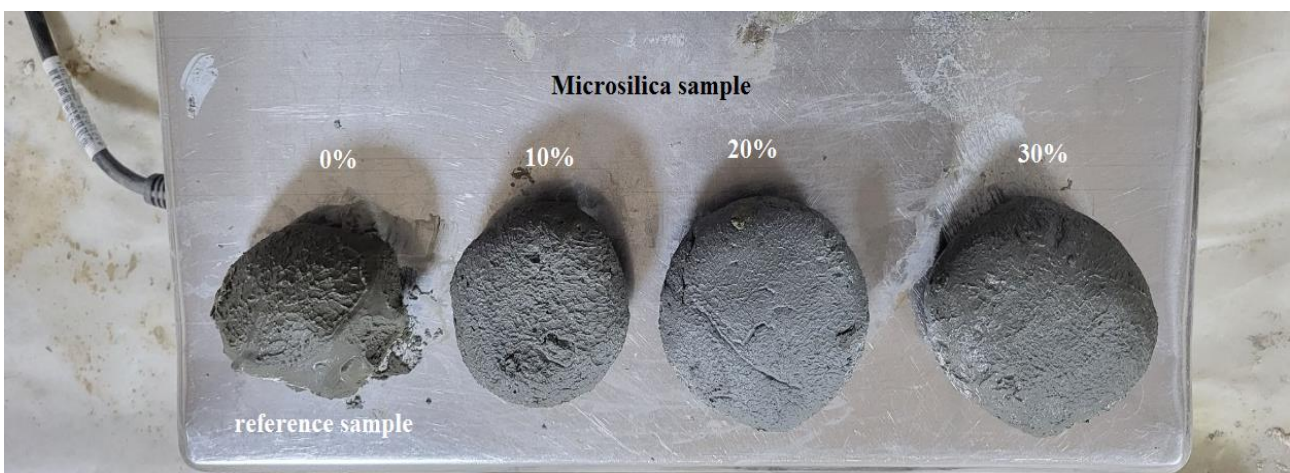


Figure 2 – Finished samples of different consistencies

3. Results and Discussion

The X-ray diffractometric analysis was carried out on an automated diffractometer DRON-3 with CuK α -radiation, β -filter. The diffractograms were taken with the following instrument settings: U=35 kV; I=20 mA; θ -2 θ survey; detector 2 deg/min. X-ray phase analysis on a semi-quantitative basis was carried out on the basis of diffractograms of powder samples using the method of equal weights and artificial mixtures. Quantitative ratios of crystalline phases were determined. We interpreted diffractograms using PDF2 (Powder Diffraction File) Release 2022 from the ICDD Powder Diffraction Database and clean mineral diffractograms. For the main phases a grade calculation was carried out. Possible impurities which cannot be identified unambiguously due to low grades and the presence of only 1-2 diffraction reflections or poor oxidation are listed in the table.

Table 2 – Results of semi-quantitative XRD analysis of crystalline phases

No	Type-1	Type-2	Type-3	Type-4
Hatrrurite (Ca ₃ SiO ₅)	62.1	64.7	56.3	41.90
Calcite (Ca(CO ₃))	7.1	9.8	10.9	18.10
Portlandite (Ca(OH) ₂)	15.7	25.6	17.6	11.70
Laihunite (Fe _{1.58} (SiO ₄))				10.30
Ilvaite (CaFe ₃ Si ₂ O ₈ (OH))	9.3		10.0	10.30
Quartz (SiO ₂)	5.8		5.10	7.80

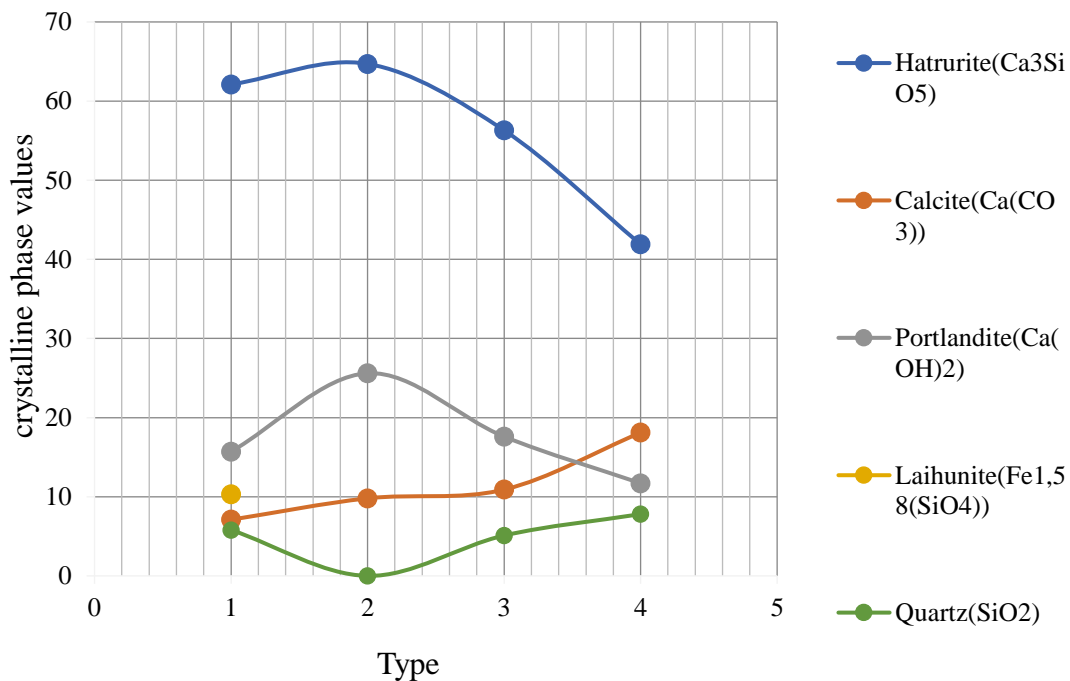
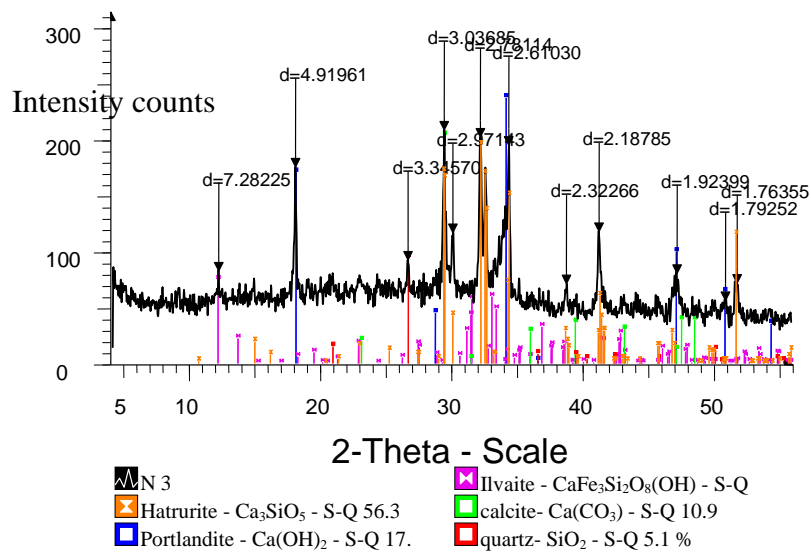
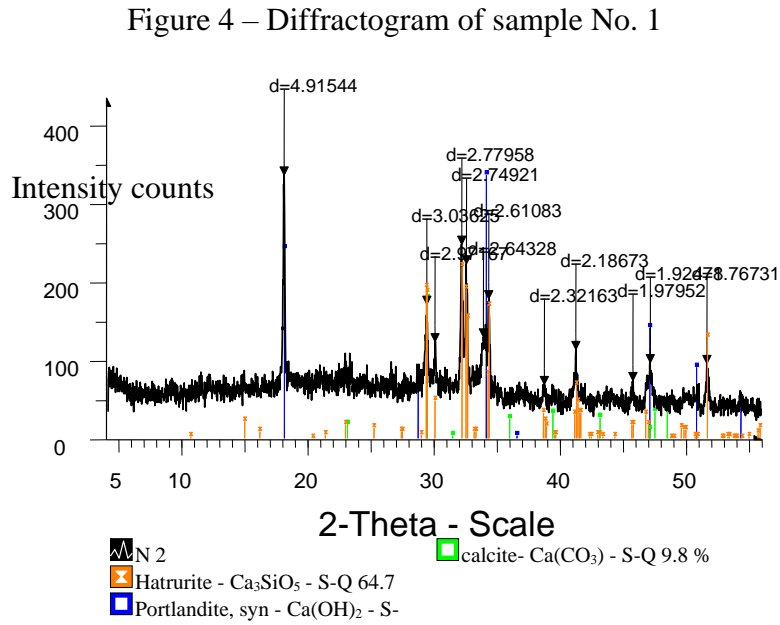
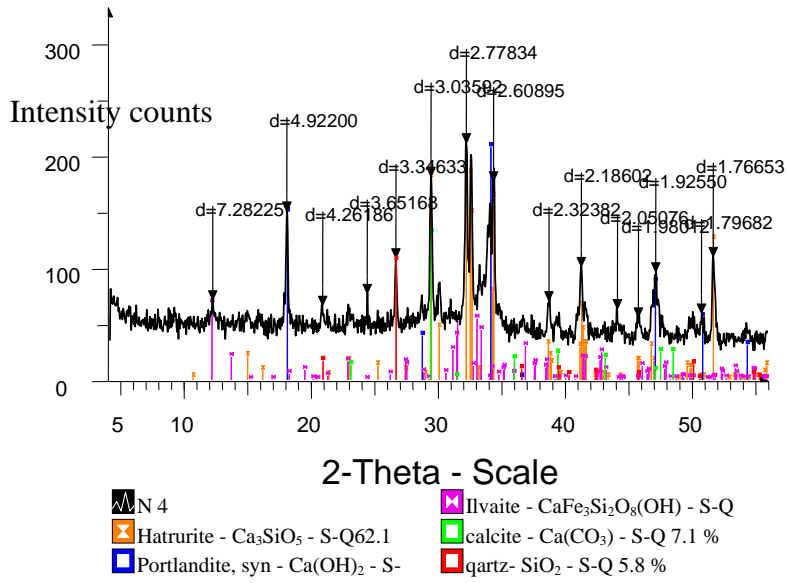


Figure 3 – Graph of crystalline phases

Table 2 presents the chemical composition of the studied samples, characterizing which it is necessary to note insignificant variations in the content of all oxides.

Comparing the results of semi-quantitative X-ray diffraction analysis of crystalline phases with addition of microsilica we see the following results: the content of Hatrrurite (Ca₃SiO₅)-(62.1 - 41.90) decreases significantly, the content of Calcite (Ca(CO₃)-(7.1-18.10) increases, the content of Portlandite (Ca(OH)₂) – in all four cases has different value. Ilvaite (CaFe₃Si₂O₈(OH)) and Quartz (SiO₂) – are characterized by close values (9.3-10.30) and (5.8-7.80).



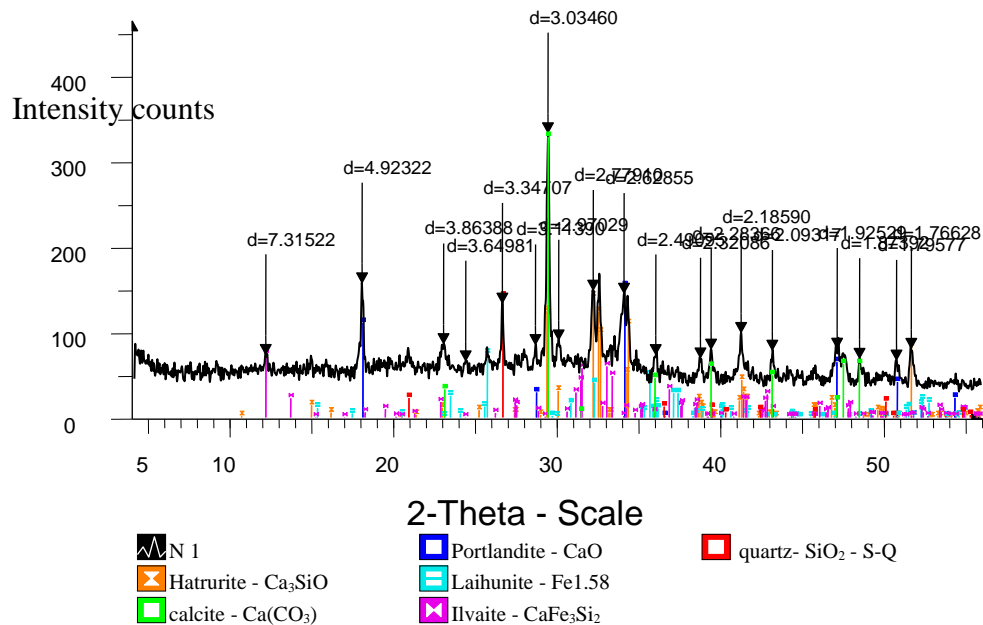


Figure 7 – Diffractogram of sample No. 4

4. Conclusions

The following conclusions can be drawn on the basis of the research carried out:

1. The mixture of microsilica additive with cement has been studied and the chemical and phase composition of the raw materials has been determined.

X-ray phase analysis revealed the presence of silicon oxide in the form of coesite or coesite in microsilica. This gives it a high chemical activity in aqueous medium. It is a highly baric modification of silica, chemical formula: SiO_2 . Average density is 2.95...3 g/cm³, hardness 7.5...8 on Mohs scale. On lowering of pressure it transforms into quartz. Therefore, the presence of coesite in microsilica is unlikely.

2. The analysis of control samples showed higher amounts of calcium carbonate. This is probably due to the carbonation of free calcium hydroxide produced during cement hydration. Increasing the content of microsilica to 30 % of the cement weight was noted to increase the total volume of calcium hydrosilicates ($\text{Ca}(\text{CO}_3)$).

3. Microsilica is introduced into products to increase mechanical properties, decrease porosity, increase water resistance and durability of products, possibly due to binding of calcium hydroxide in the structure of hardening cement stone by dispersions of microsilica. Microsilica in this case forms a structure with calcium hydrosilicates.

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