Vibrocasting of Silicon Carbide Based Concrete Materials

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Abstract: In order to reduce the water demand of silicon carbide vibratory casting masses with high-alumina cement additives, the impact of various deflocculant of different natures to silicon carbide vibratory casting masses was investigated. The deflocculants used included polycarboxylate ether-based, sodium polyacrylate, high-molecular-weight poly-N-vinylpyrrolidone, and sodium salts of polymethylene-β-naphthalenesulfoxylic acid. Multifractional compositions of silicon carbide (2-3 mm, 1-2 mm, 0,5-1 mm, 0,2-0,5 mm, 0,063-0,12 mm fractions) with high-alumina cement and silicon additives, as well as with deflocculants, were studied. The firing of the materials was conducted in an oxygen atmosphere at temperatures between 1000 and 1400˚C. The adverse effect was demonstrated for deflocculant based on sodium polyacrylate and high-molecular-weight poly-N-vinylpyrrolidone, as the usage of these additives increases the water demand of the mix. A smaller amount of water used for the mass production allows the processing of more dense materials with reduced open and closed porosity. Using deflocculants, the moisture content of the material is reduced to 6.5%.

Keywords: Vibration casting, Silicon carbide, Deflocculants, Rheology, High alumina cement, Refractories.

1. INTRODUCTION

Materials based on silicon carbide (SiC) are well-known for their use as high-temperature structural and refractory materials [1-5]. The combination of high corrosion resistance, mechanical strength, thermal conductivity, and consequently, resistance to thermal shock is particularly relevant for metallurgical applications. The side lining of electrolytic cells in aluminum production is lined with silicon carbide [6]. The use of such materials is based on their enhanced corrosion resistance to molten metals and electrolytes. High temperature materials are also used as linings for solid waste incineration plants [7]. Large-sized silicon carbide products can be obtained using various forming technologies, such as vibro-pressing, hydrostatic pressing, as well as by vibratory casting technique [8-11].

Fractional compositions with reduced water cement and dense particle packing can enhance the strength of the final products.

An effective way to reduce the moisture content of the system is the use of various deflocculants [12-15]. These deflocculant are adsorbed onto the surface of cement particles, providing a reduction in water demand through the realization of electrostatic and steric repulsion. Deflocculant are actively used in concrete technology to improve the workability of mixes and to produce concretes with special casting properties. It is important to note that not every deflocculant ensures improved properties for a specific cement, and it is even possible to observe an increase in the water demand of the mix. The effectiveness of a deflocculant is influenced not only by its parameters, such as molecular weight and chain branching, but also by its inherent nature. The incompatibility of an admixture with cement can result in delayed or excessively rapid setting, along with a decline in properties.

The aim of this work is to improve the understanding of the influence of deflocculants on the casting properties of vibro-formed casting mixes, with the subsequent processing of silicon carbide-based materials through heat treatment in air.

2. MATERIALS AND METHODS

2.1. Materials

The samples were prepared using commercially available α-SiC powder (SiC > 98%, Fe < 0.3%, C < 0.4%), which was provided by Voljsky Abrasive Plant. KR-0 silicon powder (Si > 98%, Fe < 0.5%, Al < 0.4%, Ca < 0.4%, total impurities < 1.2%, average particle size of 40-50 μm) from RUSAL Bratsk, and commercially available aluminous cement CA-70 Kerui refractory. The casting process employed silicon carbide with five granulometric fractions: 2-3 mm, 1-2 mm, 0.5-1 mm, 0.2-0.5 mm, and 63-75 μm. The studied compounds were designated as 1 and 2 (Table **2**).

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The chemical composition of the aluminous cement was determined using Thermo Scientific ARL X-ray fluorescence analysis, and the results are presented in Table **1**. The specific surface area of the cement was measured using the Blaine air permeability method, yielding a value of 3500±100 cm²/g, the measurement was conducted on the installation Blaine Apparatus Humbold H-3062.3F. Distilled water, obtained through a single distillation process, was used to mix the cement. A frequency of 60 Hz and an amplitude of 0.4 millimeters were selected for the molding process. The firing was carried out in the Tula Thermal Furnace.

To evaluate the effect of different fractions on porosity and water demand, granulometric compositions 1 and 2 were calculated using Andreasen's equation with a coefficient n=0.3 (Table **2**).

$$Q_i = (d_i/d_{max})^n * 100 \quad (1),$$

where Q_i is the amount of a certain grain fraction, d_i is the volume-weighted diameter of the grain fraction, d_{max} is the maximum grain size dimension, respectively, n is the coefficient of shape. Coefficient n=0.3 is the most typical in calculation of optimal grain size compositions, although in may vary in the range 0,3-0,6 depending on the shape of the particles.

Investigation of the vibratory casting process for silicon carbide-based concrete mass it has been implemented, with the casting mixtures modified by deflocculants from three different manufacturers, hereafter referred to as Manufacturer 1, Manufacturer 2, and Manufacturer 3. The study was performed with deflocculants based on polycarboxylate deflocculant from two manufacturers, and based deflocculant a mixture of sodium salts of polymethylene β-naphthalene sulfonic acids supplied by Manufacturer 3 (Table **3**). The recommended content specified by the manufacturers ranged from 0.2% to 0.7% by weight of the dry substance.

2.2. Sample Preparation Methodology

Prepared dry mixture of silicon carbide and cement was mixed in a mixer for 10.0 minutes. Then, water mixed with the deflocculant (in excess of 100% of the dry mixture mass) was added to the prepared dry mixture. The casting mixture (Table **2**) suspension was further mixed for 5 minutes. Samples molded into 50*50*50 mm cubes were formed on a vibration table until flowability was achieved and water appeared on the surface. The cast samples were kept in plastic molds at room temperature for 24 hours, after which the molds were disassembled. The samples were then dried at 200°C in a drying oven.

To determine the porosity and mechanical characteristics near the edge and in the central part of the molds, bar-shaped samples with dimensions of

Table 1: Chemical Composition of High Alumina Cement CA-70

The Content of Oxides, Mass %					
CaO	Fe_2O_3	Al_2O_3	SiO_2	MgO	SO_3
28.0	0.5	70.0	0.5	0.5	0.5

Table 2: The Granulometric Composition of the Compounds Studied, in Mass Percent

	2-3 mm	1-2 mm	0,5 – 1 mm	0,2 – 0,5 mm	0,063 – 0,12 mm	Cement
Composition 1	15	30	10	10	25	10
Composition 2	0	40	15	25	10	10

Table 3: The Properties of the Deflocculates of Manufactures 1,2 and 3

Manufactures	The Active Component	pH	The Content of the Main Component, %
1	Polycarboxylic acid	6.0	38.0 – 40.0
2	Polycarboxylic acid	11.0	41.0 – 43.0
3	The sodium salt of polymethylene β naphthalenesulfonic acid	5.0	58.0 – 60.0

Figure 1: Assessment of the beginning of water separation during the molding process.

10*10*100 mm were cut from the mold center and at a distance of 10 mm from the mold edges by diamond instrument.

The prepared casting samples were subjected to heat treatment in air at 250, 1000, and 1400˚C.

A transparent glass mold (Figure **1**) was used for qualitative evaluation of the deflocculants' effects. The assessment included water release and the processability of the mixture depending on the content of the additive and the mixing water. The prepared cement and silicon carbide mixture was placed on a vibrating table, after which the flowability of the mass was evaluated by the speed at which it filled the mold. The use of a transparent mold allowed for precise determination of the initial moment of water release during the placement of the mass.

The open porosity and apparent density of materials were determined by water absorption in accordance with ISO 5017:2013-01. The structures were analyzed using a JEOL 6510LV scanning electron microscope.

3. RESULTS AND DISCUSSION

3.1. Experiment Procedure

Initially, to determine the optimal water demand of the cement without deflocculants, an assessment of the standard consistency was conducted. The normal consistency assessment was performed using a small Vicat apparatus, by free immersion of a needle without additional weights into a ring filled with cement paste (CAC paste). The optimum water-cement ratio characterizes the optimal moisture content of the mixture for forming.

The normal density was calculated using the formula: (2)

$$normal\ density = \frac{m_W}{m_C} \bullet 100 \quad (1)$$

Where: m_w – weight of suspended water, g

m_c – weight of suspended cement, g

The desired normal consistency was found to be 23%. At this moisture level, the necessary consistency of the workable cement mix is ensured. At this moisture level, the cement paste did not exhibit self-leveling properties.

The introduction of deflocculants based on polycarboxylates in amounts less than 0.3% did not have a noticeable effect on the rheological properties. However, in the concentration range of the additive to 0.3-0.4%, the cement paste became self-leveling (Figure **2**), indicating complete adsorption of the polymer molecules on the surface of the cement particles and the realization of the barrier effect (repulsion effect). When using the sodium salt of polymethylene β-naphthalene sulfonic acid, a slightly larger amount of the additive was required compared to polycarboxylate. The use of non-polycarboxylate additives required 0.5%. Deflocculant based on β-naphthalene sulfonic acids, although they have a small steric effect due to bulky sulfonic groups, act by electrostatic repulsion between cement particles and make their surfaces negatively charged, which reduces the adhesion forces between the particles. Polycarboxylic deflocculant provide a dual action: they create spatial obstacles around the cement particles that prevent their agglomeration, and also provide electrostatic repulsion. Their molecules adsorb on the cement particles and "branch out," which enhances the dispersing effect (Figure **3**).

Thus, it was found that the minimum necessary concentration of deflocculants to achieve an improvement in the rheological properties of the cement paste was 0.3 - 0.5%.

Figure 2: Self-spreading cement dough after the introduction of 0.3% polycarboxylate additive.

The granulometric composition also affects water demand. For instance, to mold samples of composition 1, a water percentage exceeding 10.0% of the dry mix's mass was required, whereas for composition 2, only 7.2% was necessary.

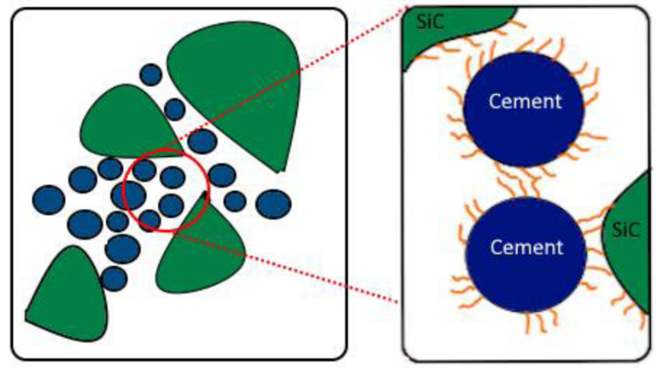

Figure 3: Schematic diagram of the implementation of the steric barrier.

The experiment had shown that the minimum water amount for molding significantly depends on the parameters of the selected additive. The use of deflocculants based on polycarboxylic acids helped to reduce water demand to 6.8% and 7.0% for deflocculant of manufacturers 1 and 2, respectively. The additive from manufacturer 3 reduced water demand to 6.5%, demonstrating superior interaction with silicon carbide compared to polycarboxylates (Figure **4**). This efficiency is attributed to the structure of the polymer composition, which ensures strong interaction with negatively charged silicon particles, promoting their uniform repulsion. Further increasing the concentration of the additive beyond 0.4% did not reduce water demand, as additional water was introduced with the additive, leaving the resulting water content unchanged.

It is worth noting that compositions 1 and 2, which had different amounts of mixing water without the

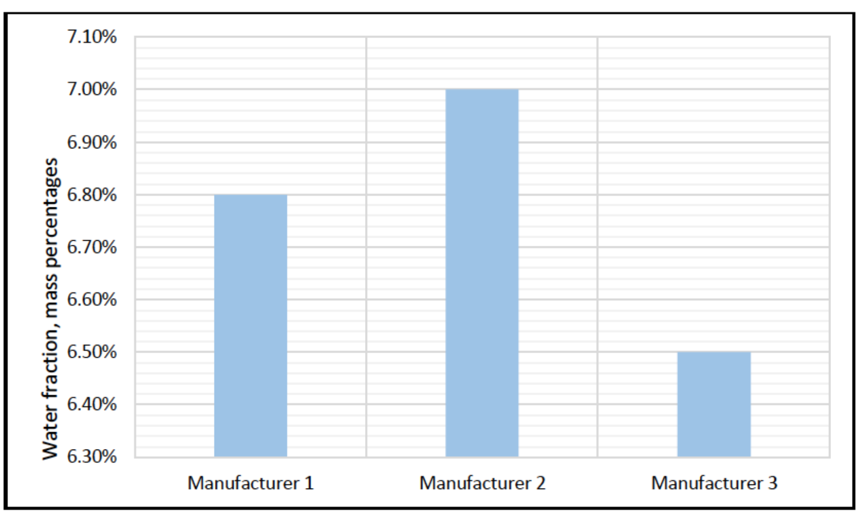

Figure 4: The minimum water requirement for forming concrete based on silicon carbide by vibration casting in the case of using deflocculants from manufacturers 1,2,3.

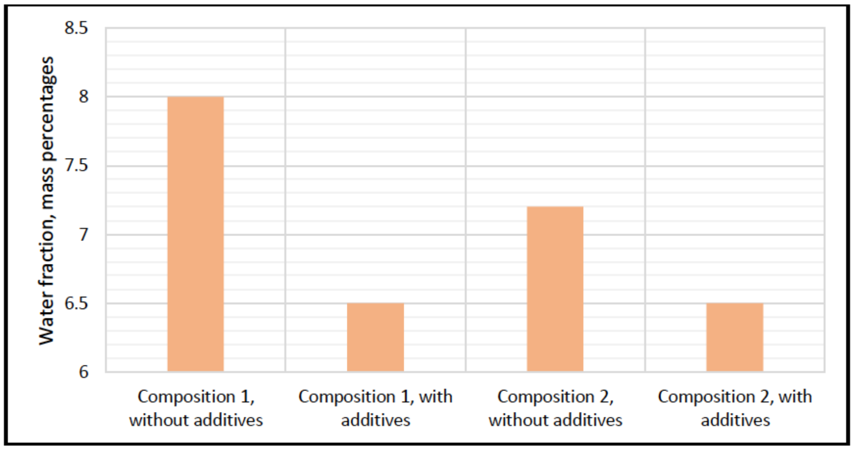

Figure 5: Water demand depends on the fractional composition and the presence of a deflocculant.

additive, exhibited the same water demand when the deflocculant was introduced (Figure **5**). This indicates that the water demand of the final concrete mass is largely determined not only by the type of filler, its dispersion, and the number of fractions, but also by the cement.

Well-chosen deflocculants enhance the density of castables and reduce their porosity. The results confirm the necessity of using deflocculants in the technology of vibrated castable refractories.

3.2. Physico-Chemical Changes in Vibro-Cast Concretes Based on Silicon Carbide and Alumina Cement During Heat Treatment

During cement hydration, a structure forms consisting of interwoven conglomerates of crystallohydrates. Amorphous crystallohydrates grow on the surface of individual silicon carbide grains in the untreated material.

The primary phase of the alumina cement used in the study is calcium monoaluminate ($CaO \cdot Al_2O_3$), with a small amount of aluminum oxide (Figure **6**). The formation process of hydrate phases based on calcium monoaluminate involves several stages. In contact with water calcium monoaluminate hydrates transforms into calcium aluminate decahydrate. This aluminate is an unstable compound and converts to calcium hydroaluminate octahydrate according to the reaction (3)

$$2(CaO{\bullet}A1_2O_3)+11H_2O \rightarrow 2CaO{\bullet}A1_2O_3{\bullet}8H_2O+2A1(OH)_3 \quad (3)$$

Hydration is accompanied by an increase in molecular volume (Table **4**). The cast shape contains hydrated monocalcium aluminate, physically bound water, and pores (Figure **7**).

After heat treatment at 250°C, the structure becomes crystalline with clearly visible boundaries. The physically bound water evaporates, leaving behind hydrated monocalcium aluminate and pores. During heat treatment, dehydration occurs, resulting in monocalcium aluminate ($CaO^*Al_2O_3$); however, it is noticeable that the dehydration is not complete.

Treatment at 1000°C leads to the decomposition of the crystallohydrates to the original oxides. This reaction occurs with a negative volume effect, resulting in a slight increase in porosity. This increase in porosity was experimentally confirmed. Calcination at 1000°C ensures complete dehydration, and the particles of monocalcium aluminate, which bind the silicon carbide grains, become sharper (Figure **9**, **10**).

Table 4: Density, Molecular Weight and Molecular Volume of Calcium Monoaluminate and Hydrated Calcium Aluminate

	density, g/cm^3	mol. Weight	molar volume
CA	2.98	158	53.02
CAH_{10}	1.72	338	196.51

The molar volume Vm, of a substance is equal to the molar mass (M) divided by the mass density (ρ):

$Vm = M / \rho$ (4)

The molar volume of calcium aluminate is (cm^3/mol)

$Vm = M / \rho = 158 / 2{,}98 = 53{,}02$ (5)

The molar volume of calcium aluminate hydrate is (cm^3/mol)

$Vm = M / \rho = 338 / 1{,}72 = 53{,}02$ (6)

Respectively, at dehydration of calcium hydroaluminate to calcium monoaluminate is negative.

Volumetric effect of dehydration of calcium hydroaluminate

$$\Delta V/V = (196{,}51-53{,}02)/196{,}51 = 0{,}73 \qquad (4)$$

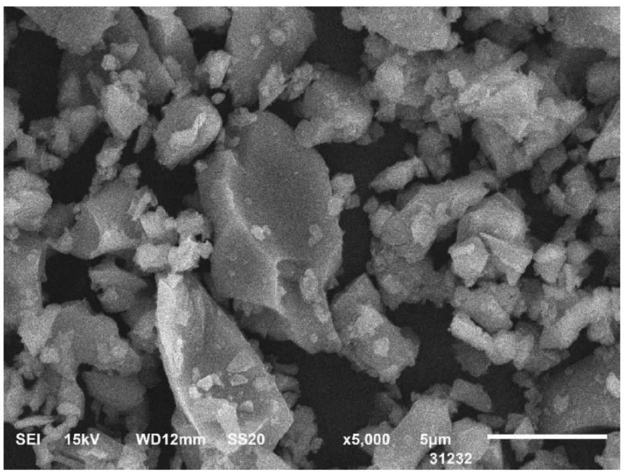

Figure 6: Alumina cement, based on calcium aluminate.

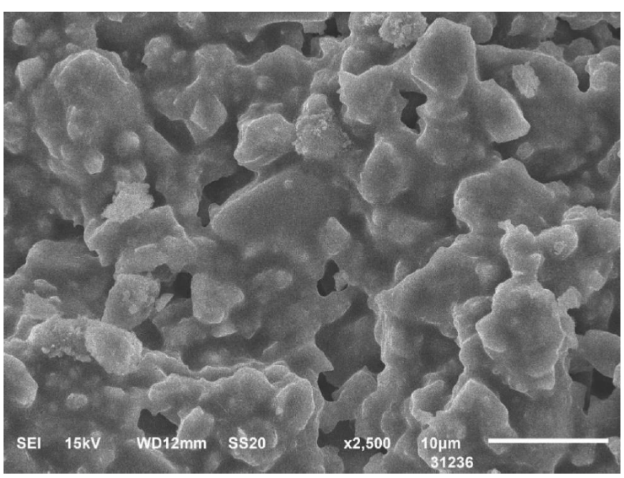

Figure 7: Hydrated calcium monoaluminate.

Thus, the reduction in molecular volume will be minus 27.0%. In this study, silicon carbide compositions with 10.0% calcium aluminate were used. Accordingly, the increase in porosity during the dehydration of hydrated calcium monoaluminate should be 2.7%. During the heat treatment process, dehydration occurs. The removal of water increases the porosity of the samples (Figure **11**).

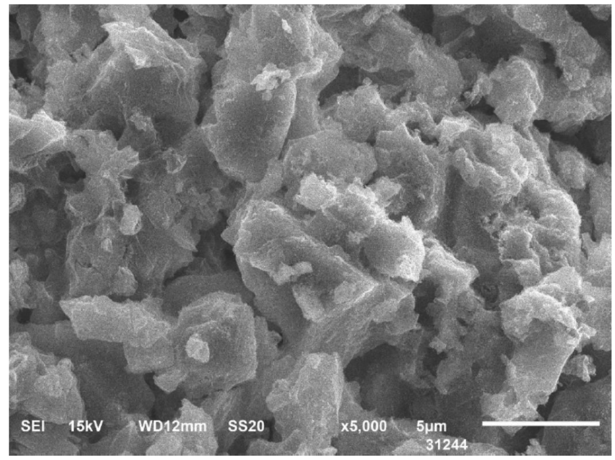

Figure 8: Hydrated calcium monoaluminate after calcination 250˚C.

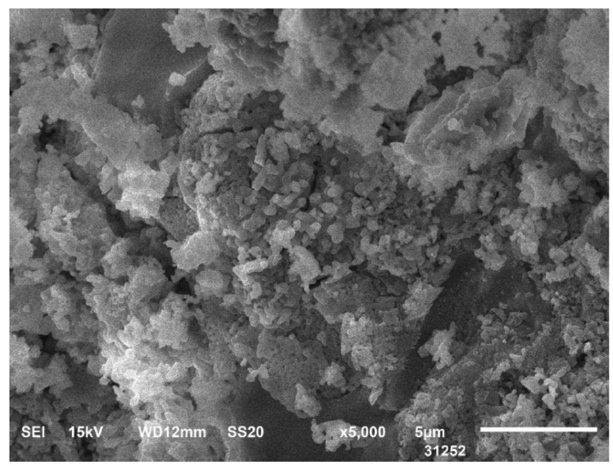

Figure 9: Hydrated calcium monoaluminate after calcination 1000˚C.

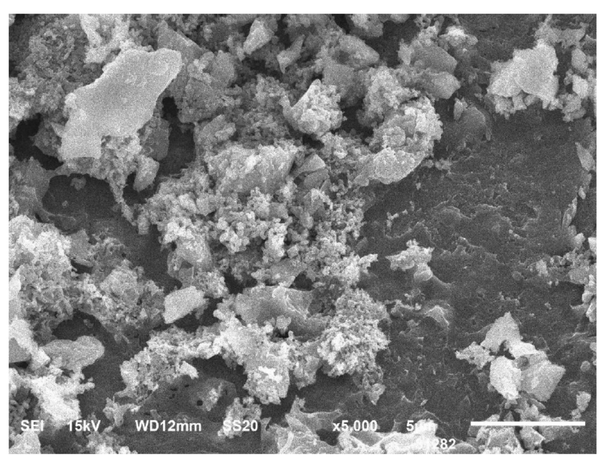

Figure 10: Structure of silicon carbide concrete calcined at 1000˚C.

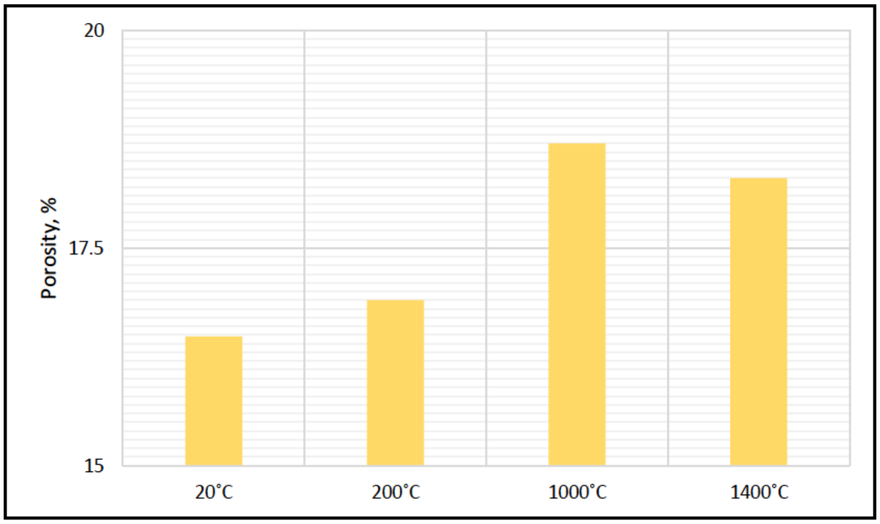

Figure 11: Porosity of silicon carbide concrete during drying and calcination.

3.3. The Effect of Silicon Additives on Water Demand, Liquefaction, Porosity of Vibro-Cast Concretes Based on Silicon Carbide

It is well known in the technology of producing materials from silicon carbide with a silicon nitride binder that silicon powder is added to the charge, which transforms into silicon nitride during nitriding firing (Figure **12**)[17-20]. An evaluation of the influence of silicon on the rheology of the system and pre-firing porosity and density was performed.

To evaluate the effect of silicon, we introduced it into Composition 2. The rebatching was done by replacing the finest fraction of 0.063 - 0.12 microns. Free silicon increased water demand, most significantly after 10% (Figure **13**). Up to 10%, the increase in water demand was insignificant and remained below the levels without the introduction of a deflocculant. When 15% free silicon was introduced, a significant increase in the required amount of water for molding was observed. The introduction of 20% silicon did not require a substantial increase in water compared to 15% silicon. The next jump was with a silicon content of 25%, for molding this composition required 10% more than a hundred.

Considering the essential presence of silicon in the system, which leads to an increased water demand, it is necessary to evaluate the porosity and density of the obtained samples.

Increasing the volumetric fraction of water in the composition leads to an increase in porosity. Increasing the silicon content to 25% leads to a significant increase in porosity, as much more water is required (Figure **14**).

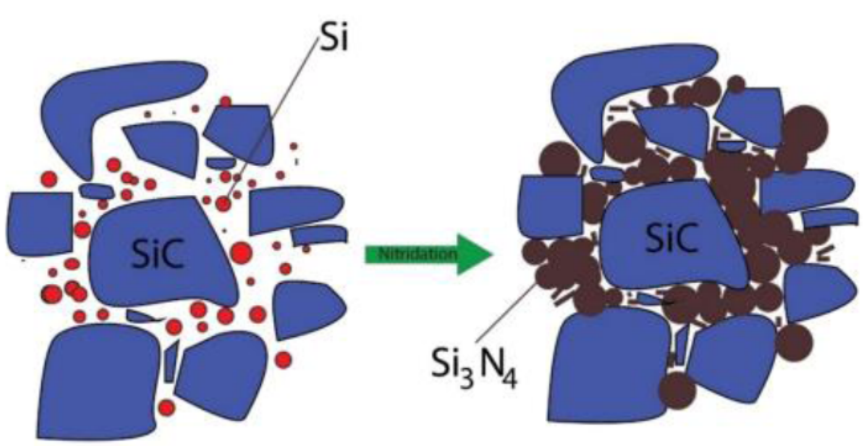

Figure 12: Schematic of the densification mechanism due to Si_3N_4 formation [17].

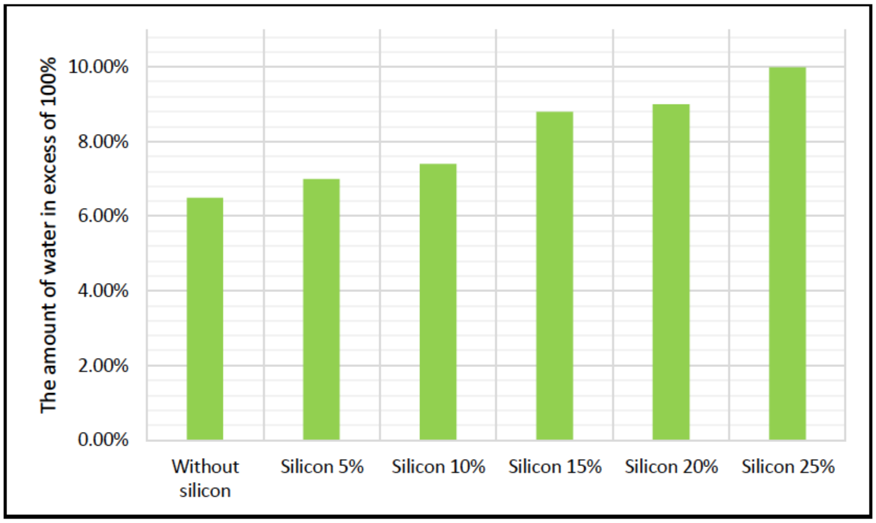

Figure 13: Water demand depending on the amount of silicon.

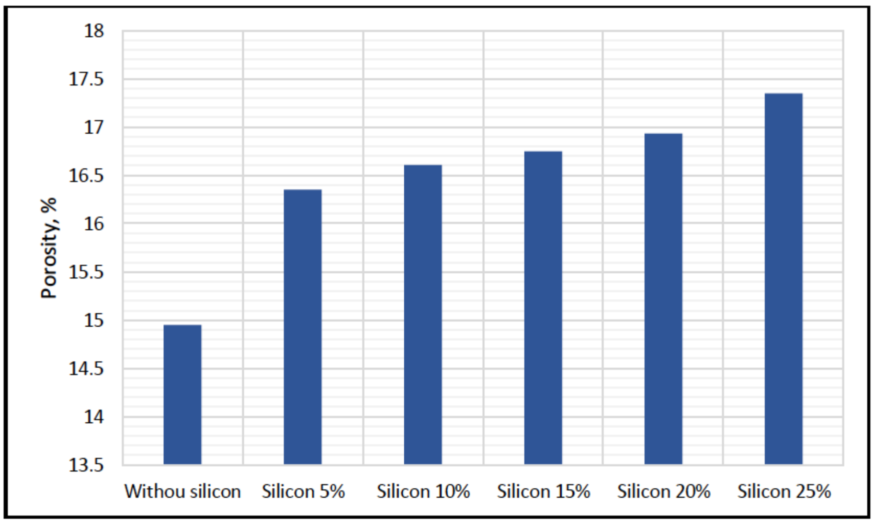

Figure 14: Cold porosity of samples without and with the introduction of silicon.

Thus, the introduction of free silicon negatively affects the rheology of the mass and negative impacts the packing density [21]. The volumetric effect of Si_3N_4 formation compensates for the negative volumetric effect of crystallohydrate decomposition.

The reduction in density is primarily due to the increase in the mass fraction of silicon in the batch composition. The increased amount of water also contributes to the increase in porosity.

CONCLUSION

1. Deflocculants are very helpful for the vibrocasting process of silicon carbide concrete.

2. The proper selection of pH, molecular weight, and the length and branching of the side chains of additives can reduce the water demand and residual porosity in the final product.

3. Defloculants containing polycarboxylic acids are more effective at reducing water demand compared to those containing sodium salts of polymethylene beta naphthalene sulfonic acid.

4. Silicon powder added to silicon carbide vibrocast concrete significantly increases the water requirement for casting.

5. The analysis of porosity during the processing of these concrete shapes agrees well with the measured values of porosity, indicating a successful process.

DATA AVAILABILITY

All data generated or analyzed during this study will be made available on request.

DECLARATIONS

Research Involving Human Participants and Animals

This article does not contain any studies with human or animal subjects.

STATEMENT ON THE WELFARE OF ANIMALS

This article does not contain any studies with human participants or animals performed by any authors.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

We comply to the ethical standards. We provide our consent to take part.

The microstructure images were taken at the D.I. Mendeleev Central Research Center.

CONSENT FOR PUBLICATION

All the authors are giving consent to publish.

COMPETING INTERESTS

The attracted financing was not used in the performance of the work. The authors declare no competing interests.

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