Effect of Additives and Installation Temperatures on Setting Behaviour and Mechanical Properties of Self-flowing Silica Bonded No-cement Castables

H. Peng, B. Myhre

Microsilica-gel bonded bauxite based no-cement refractory castables (NCCs) have been produced using two readily available types of dispersants. These are compared to NCC with SioxX-Zero, a purposely-developed product for microsilica-gel bonded no-cement castable systems to control flow properties and set characteristics. Three mixing and curing temperatures were applied: 5, 20 and 35 °C respectively. The results show that the setting-behaviour and mechanical properties strongly varies with the type of dispersant and the curing temperature. However, both the setting-behaviour and strength are less influenced by variation in curing temperature in the castables with SioxX-Zero. Since microsilica-gel bond system contains only a small amount of bound water, the castables can be fired at very high heating rates, once the free water has been removed.

1 Introduction

Silica-sol bonded no-cement castables (NCCs) is the current state-of-the-art. The main advantages are fast drying, volumetric stability, and good mechanical properties both at intermediate and high temperatures. The high temperature properties are obtained through mullite formation and no liquid formation from cement and silica [1–5].

However, the use has been limited due to the following challenges:

- inadequate green strength and long settime/complex setting behaviour at lower temperatures,
- frost sensitivity under installation, storage and transportation due to the liquid silica-sol,
- complex logistics and shelf-life issues due to the two-component system.

Naturally, a "dry-version" of a silica binder, using microsilica powder, is of great interest to develop NCCs further. Microsilica consists of spherical, amorphous silicon dioxide (SiO_2) particles with an average particle size of 150 nm. Recent reports disclose that a genuine bond based on microsilica coagulation is created, and that the setting of microsilica-gel bond castables is caused by cations [6–8], a similar set mechanism to colloidal silica. Microsilica-gel bond not only provide similar advantages as silica-sol but also eliminate some of the drawbacks of a two-component system (e.g. frost sensitivity and the shelf-life issues). Furthermore, due to the spherical shape of microsilica, closer packing and consequently enhanced castable flow/reduction in water demand can be achieved [6].

Based on our experience and understanding of the characteristics of microsilica and its performance in refractory castables during the last thirty years, a new speciality product (SioxX-Zero) has recently been developed for microsilica-gel bonded NCCs [9]. For ease of application and improved functionality, high-grade microsilica is used as a carrier in the product; and the recommended dosage is approximately 3 mass-%. The addition of SioxX-Zero will enhance green strength and control the set and hardening process. By using SioxX-Zero in combination with polyvalent cations, the set time can be controlled. Microsilica-gel bonded NCCs with SioxX-Zero perform excellent in terms of green strength and set-behaviour.

In this paper, the impact of dispersants and test temperatures on setting behaviour and mechanical strength of bauxite based NCCs with microsilica-gel bond have been studied in detail. Further, the microsilica-gel bonded bauxite based NCCs were investigated with respect to their drying behaviour.

2 Experimental

2.1 Mix design

The particle size distributions (PSDs) of microsilica-gel bonded NCCs were calculated using the EMMA program [10]. EMMA is based on the Andreassen model and is widely used to evaluate and optimise particle packing in castables. In the present study, the q-value was 0,25 for all mixes.

Tab. 1 shows the composition of the NCCs with different dispersants; the water addition was 5 mass-%. The optimised dosage of SioxX-Zero, dispersant B and C were 3, 0,05 and 0,23 mass-%, respectively. The composition with SioxX-Zero has been

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Keywords: dispersants, curing temperature, temperature dependence, no-cement castable (NCCs), microsilica-gel bond

Tab. 1 Composition of NCCs using either microsilica-gel or silica-sol as binders [mass-%]

	Microsilica-Gel			Silica-Sol
	A	B	C	D
Bauxite, 0–5 mm	78	78	78	78
Alumina fines	13	13,5	13,5	14,5
Elkem 971U	6	8	8	4,8
Silica-sol, 40/130 *				8
SioxX-Zero	3			
Disp. B		0,05		0,05
Disp. C			0,23	
70 % CAC	0,5	0,5	0,5	
Gelling agent				0,1
Water content	5			

* The solid loading of silica-sol is 40 mass-%, with a surface area of 130 m^2/g; 4,8 mass-% water was introduced by the silica-sol and 0,2 mass-% additional water was added

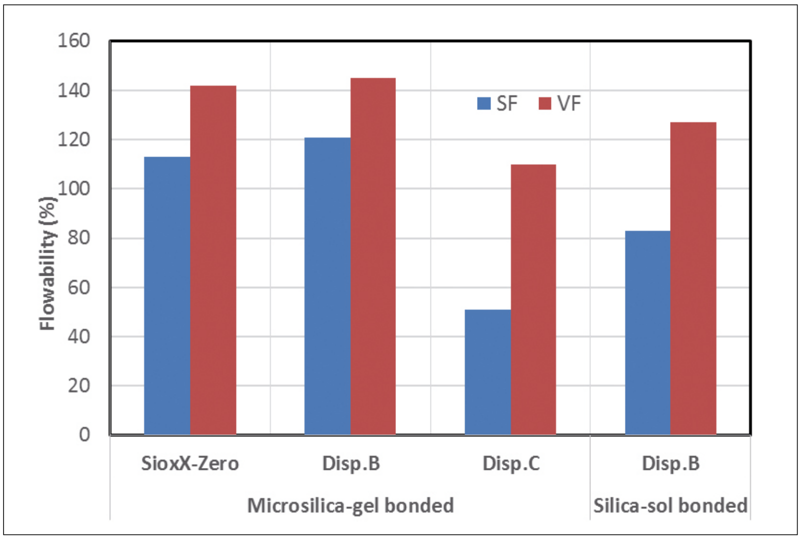

Fig. 1 Self- and vibration-flow of bauxite NCCs

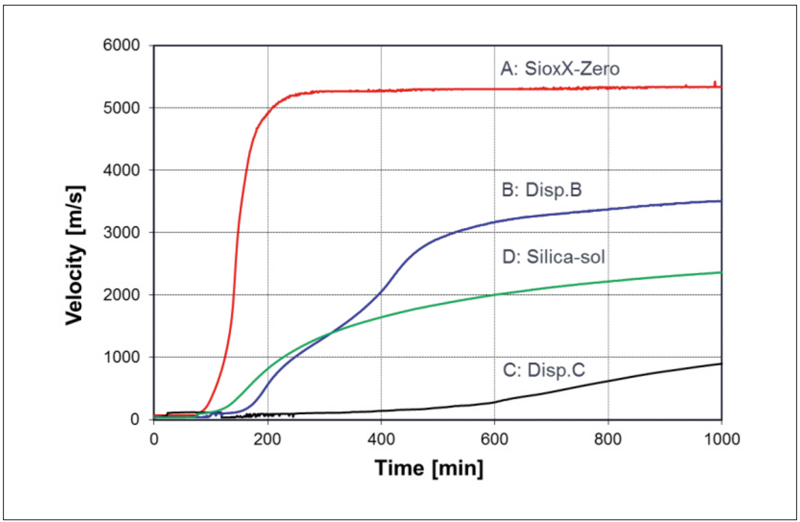

Fig. 2 Setting behaviour of bauxite NCCs with different dispersants and binders at 20 °C; ultrasonic velocity as a function of time

adjusted due to its microsilica content. A silica-sol bonded castable was included for reference.

2.2 Experimental procedure

Self-flow and vibration-flow after 4 min wet-mixing were measured using the flow-cone described in ASTM C230 (height 50 mm, not the 80 mm self-flow cone described in EN 1402-4:2003). The self-flow value is the percentage increase of the diameter measured 90 s after removing the cone.

Three different mixing and curing temperatures, 5, 20 and 35 °C respectively were applied for both microsilica-gel and silica-sol bonded NCCs in order to understand the effect of installation temperatures on set-behaviour and mechanical properties.

An ultrasonic recorder has become an efficient tool to follow the set and hardening process of no-cement castables. The equipment used was an Ultratest IP-8 (Ultratest/DE). The propagation velocity of the ultrasonic wave through the samples is plotted vs. time from mixing. As stiffness and speed of sound are closely related, the onset of increasing velocity of the ultrasonic signal indicates end of working time and initial set.

Cold crushing strength (CCS) was also measured after demoulding and drying.

3 Results and discussion

3.1 Effect of dispersants on flow and setting-process at room temperature (20 °C)

Self- and vibration-flow values at 20 °C are summarised in Fig. 1. The water addition for all mixes is 5,0 mass-%. The NCCs with SioxX-Zero and dispersant B show similar self-flow values, some 110–120 %, while the one with dispersant C has the lowest self-flow and the silica-sol bonded NCC has a self-flow of approximately 80 %.

As shown in Fig. 2, the microsilica-gel bonded castable with SioxX-Zero has a much quicker and more “defined” set than the others. The significance of “defined” here is that once the castable starts to set, the strength development is fast and the time to reach its “final” (green-strength) value is short.

Time to final strength is in the order of 5 h for the SioxX-Zero containing castables as opposed to castable B, C, and D that have a much slower set- and strength development. Even though the time to final set of castable B and D seems acceptable, approximately 10 h, they do not have the same quick and well "defined" set behaviour and never reaches the strength level of the SioxX-Zero containing mix. The castable with dispersant C did not fully set even after curing for two days as shown in Fig. 3.

The results demonstrate that the type of dispersant and the binder system have a strong impact on the set and hardening process of NCCs. The exact mechanism of how the dispersants influence the set-behaviour is not totally understood yet, and further research is ongoing.

3.2 Green strength/strength after drying (20 °C)

Fig. 4 shows the green compressive strength (24 h at >90 % RH and 20 °C) and the strength after drying (24 h at 110 °C). The green strength of the castable with SioxX-Zero is more than double, up to 10 MPa, compared to the other castables. One could argue that the low strength of the mixes at least partly is a consequence of the slow set, but even with prolonged curing time, the results of the sonic velocity measurements indicate that the silica-sol containing composition never will reach the same level as "pure" microsilica-gel bond with SioxX-Zero.

After drying, the difference in strength is smaller. Even the castable with dispersant C that did not fully set after curing for two days, showed adequate strength after drying at 110 °C. Nevertheless, the castable with SioxX-Zero seems to be superior to the others, with a CCS of ~45 MPa.

3.3 Effect of the curing temperature on setting behaviour

Two more mixing and curing temperatures (5 and 35 °C respectively) were applied beside the room temperature of 20 °C. Fig. 5 shows the dramatic retarding effect of low temperature on the setting behaviour (Fig. 2 for corresponding setting behaviour at 20 °C). However, the degree of retardation varies with the different dispersants. The microsilica-gel bonded castable with SioxX-Zero can be demoulded after 24 h even though it only reaches approximately 60 % of its final strength. The other castables were still soft and could not be demoulded even after 48 h curing.

As shown in Fig. 6, the accelerating effect of a higher temperature on the setting behaviour is observed. For example, the mix containing Disp. C has set after curing less than 24 h at 35 °C, while it did not set at 20 °C for 48 h. At 35 °C, the setting behaviours of all mixes have been accelerated to various degrees. Yet, the microsilica-gel bonded castable with SioxX-Zero has a much quicker and more "defined" set than the others.

Fig. 3 Castable with dispersant C after curing for two days at 20 °C

3.4 Effect of the curing temperature and additives on mechanical strength

The compressive strength after 24 h at 5, 20 and 35 °C of the microsilica-gel bonded castable with SioxX-Zero and the silica-sol bonded castable were investigated further.

The CCS after demoulding and drying at 110 °C are shown in Fig. 7. At 5 °C, the silica-sol specimen did not harden after 48 h curing, but it set when it was dried at 110 °C. Obviously, at installation temperatures of 5 and 20 °C, the castable containing SioxX-Zero outperforms the silica-sol bonded one. At 35 °C, both castables

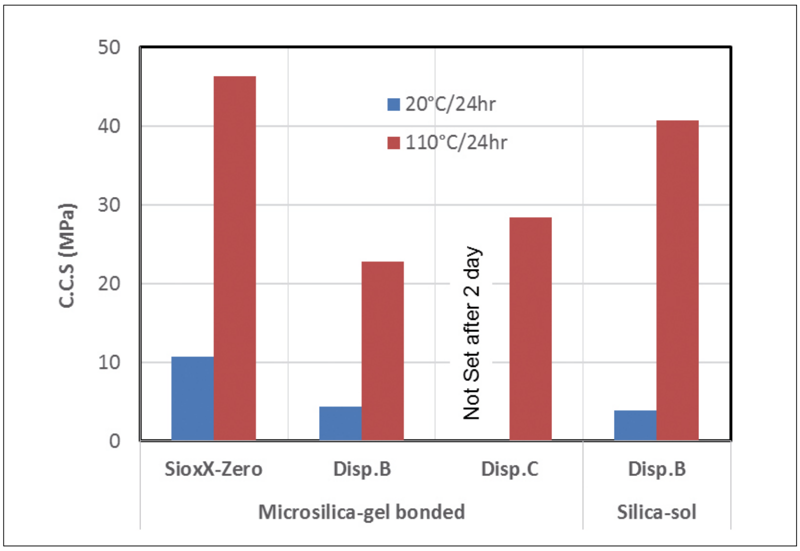

Fig. 4 Cold crushing strength (CCS) of bauxite NCCs with various dispersants and binders

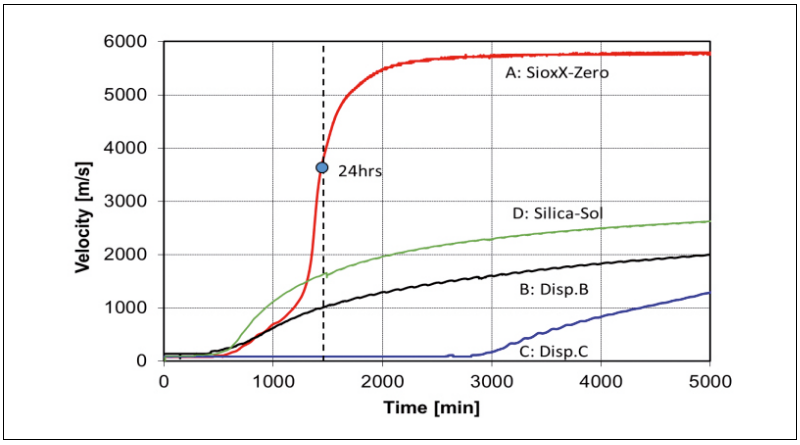

Fig. 5 Setting behaviour of bauxite NCCs with different dispersants and binders at 5 °C

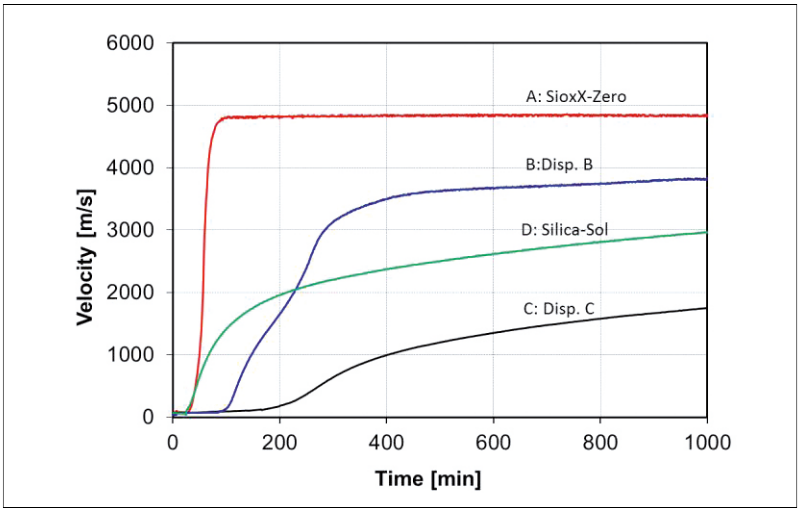

Fig. 6 Setting behaviour of bauxite NCCs with different dispersants and binders at 35 °C

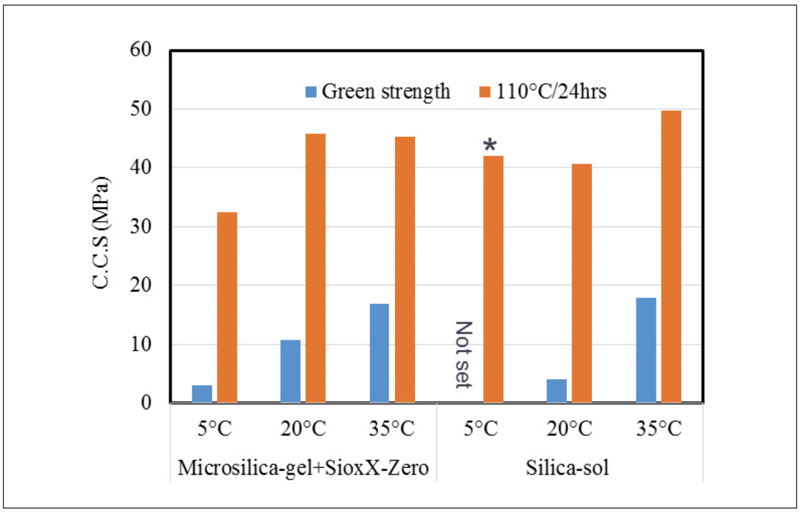

Fig. 7 CCS after demoulding and drying at 110 °C of bauxite NCCs that were cured at 5, 20 and 35 °C respectively for 24 h
*the specimen was cured for 48 h before drying at 110 °C

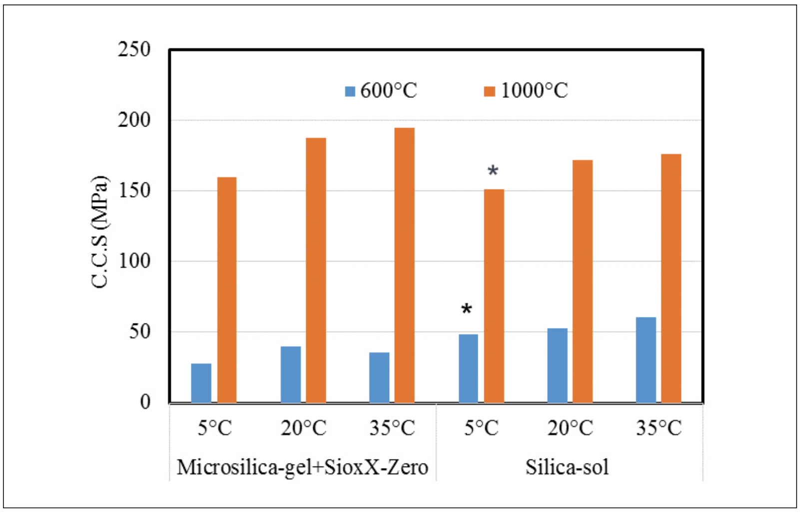

Fig. 8 CCS after prefiring at 600 and 1000 °C respectively of bauxite NCCs that were cured at 5, 20 and 35 °C respectively for 24 h
*the specimen was cured for 48 h

exhibit similar green strength as well as strength after drying.

Fig. 8 shows the CCS of the bauxite NCCs after pre-firing at 600 and 1000 °C respectively. At 600 °C, the CCS of microsilica-gel bonded NCCs is slightly lower than the silica-sol bonded one after curing at various temperature. At the higher temperature of 1000 °C, the former series of NCCs catch up, showing higher CCS than the latter one at each curing temperature.

As demonstrated above, the curing temperatures and type of dispersant have strong impact on the performance of silica-bonded NCCs. The dry microsilica binder in combination with SioxX-Zero exhibits improved setting behaviour and higher green strength at lower temperatures as compared to the silica-sol bonded system.

3.5 Drying behaviour of microsilica-gel bonded NCCs

The drying behaviour of industrial-scale specimens of the microsilica-gel bonded bauxite NCC using SioxX-Zero cured at 20 °C was further investigated. Larger specimens, 800 mm × 600 mm × 200 mm blocks (~300 kg), were produced and dried at various temperatures and holding times. Two different rapid drying schedules (as shown in Fig. 9 c) were applied.

Fig. 9 a shows the remainder of a block heated according to schedule A, holding at 160 °C for 6 h before continuing to 850 °C at a rate of 75 °C/h. The block still contained more than 40 % of the water, and during the rapid heat-up from 160 °C, the pressure of water vapour caused complete disintegration. Fig. 9 b shows a perfect block after heating according to schedule B where the block was kept at 220 °C for 10 h before continuing to 850 °C at a rapid rate of 100 °C/h. Approximately 97 % of the free water is removed at 220 °C. This block was essentially dry when the rapid heat-up started.

Unlike low-cement castables (LCC), the microsilica-gel bonded NCCs do not use cement as binder and contain only small amounts of bound water, thus are well suited for fast firing as soon as the free water has been removed.

4 Conclusions

The performance of microsilica-gel bonded NCCs varies when different types of dispersants and curing temperatures are applied. The castable containing SioxX-Zero has a controlled setting behaviour and highest green strength.

Compared to the silica-sol bond system, the microsilica-gel bonded NCCs in combination with SioxX-Zero not only give easier handling, storage and transportation thanks to the "all-in-the-bag" solution, but also exhibit improved setting behaviour, adequate green strength and good robustness to temperatures.

Furthermore, the heating temperature and holding time has strong impact on the drying behaviour of the NCCs. The microsilica-gel bond system contains only small amounts of bound water. Once the free water is removed, the castables can be fired at very high heating rates.

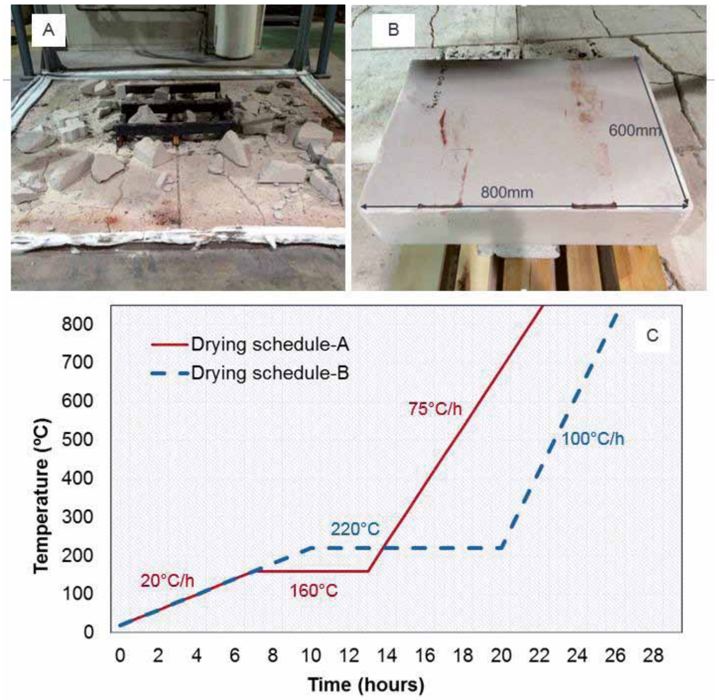

Fig. 9 a–c **a) Remainder of block after drying according to schedule A; b) perfect block after drying according to schedule B; and c) drying schedules**

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