

Influence of Different Matrix Components on the Rheological and Mechanical Properties of High-Alumina LC Castables

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The development of high-quality low-cement refractory castables is the result of several aspects that influence the performance of the final product. The selection of suitable raw materials both for the coarse grains and for the matrix has a strong impact on the parameters. The coarse grains are responsible for the formation of the final structure, while the matrix components (cement, reactive aluminas and chemical additives including deflocculants, retardants or setting accelerators, etc.) enhance the characteristics of castables in application. In this paper, the focus is, therefore, just on high-alumina LC castable with different matrix components and the effects of these components on the rheological and mechanical properties important for the production and installation process.

This study focuses on the workability of the test products. Standard quality control procedures during sample preparation and testing (DIN EN 1402) were applied. The following tests were performed: flowability test, CCS, MOR, density. The flowability tests enabled further selection of materials according to the best flow values achieved. Another objective of this study was to determine the sensitivity and measurable impact on the properties of LC castables as a result of the change in reactive alumina, deflocculant and consequently increased water content.

1 Introduction

In recent years, in the aftermath of the world economic crisis followed by very fierce competition in the refractory market, a strong tendency has been observed towards the development of more cost- and lifetime-efficient refractory solutions. The use of high-grade raw materials has become important not in the overall structure of the product but particularly in the matrix. Experience has proven that using or replacing even small amounts of dif-

ferent components can strongly influence the performance of the final castable. That is the reason why the appropriate selection of reactive aluminas, deflocculants and cements has become an important issue in designing LC castables and their performance.

2 Experimental procedure and results

To analyse the influence of different matrix components, their effect on rheological and mechanical properties and to avoid any impact coming from natural raw materials or their reprocessing, tabular alumina was selected as the main raw material for the test samples. In the matrix, the following reactive aluminas were used respectively: two mono-modal (MR42 from *Martinswerk* and

Tab. 1 Zschimmer & Schwarz's formula used during the investigation (reactive aluminas and deflocculants have been taken respectively)

| Raw material: | D ₅₀ [µm] | PSD | Specific surface [m ² /g] | addition [%] |
|--|----------------------|------------|--------------------------------------|--------------|
| Tabular alumina (1–6mm) | | | | 55 |
| Tabular alumina (0–1mm) | | | | 10 |
| Tabular alumina (<44 µm) | | | | 25 |
| <i>Reactive alumina (respectively):</i> | | | | |
| MR 42 (<i>Martinswerk</i>) | 2 | Mono-modal | 2,5 | 5 |
| CT 3000 SG (<i>Almatis</i>) | 0,8 | Mono-modal | 7,5 | 5 |
| CL 370 C (<i>Almatis</i>) | 2,6 | Bi-modal | 3 | 5 |
| <i>Deflocculating agents</i> – all Polymer type (respectively): | | | | |
| DOLAPIX FF 26 (<i>Zschimmer & Schwarz</i>) | | | | 0,15 |
| Product "F" | | | | 0,15 |
| Product "A" | | | | 0,15 |
| <i>Cement:</i> | | | | |
| SECAR 71 (<i>Kerneos</i>) | | | | 5 |

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CT3000SG from *Almatis*) and one bi-modal (CL370C from *Almatis* – a standard option for many refractory producers). Also three deflocculants, from different suppliers (DOLAPIX FF 26 is manufactured by *Zschimmer & Schwarz*), were selected for the purpose of the tests. The formula chosen was *Zschimmer & Schwarz's* standard laboratory recipe.

2.1 First stage of the investigation

At the first stage of the investigation several samples were prepared to evaluate the behaviour and workability of the LC castable depending on the water content. The following water additions were considered for testing: 4,7 %, 4,8 %, 4,9 %, 5,0 %.

The tests performed on the cast specimens:

- Flow test depending on the amount of water added (Fig. 1 left)
- Cold crushing strength (CCS); also green CCS on selected samples (DIN EN 1402) (Fig. 1 right)

The following results were obtained during this part of the investigation:

As shown in Fig. 1A for the reactive alumina MR42, the amount of water necessary for the proper flow of DOLAPIX FF26 and "F" is in

the range between 4,7 and 5 %. Considering the results from the "best flow range" (190–210 mm) ("proper consistency" [1]), the following was observed:

- DOLAPIX FF26 showed much more stabilized CCS results with different water additions. As it seems, small differences in the amount of water do not impact too strongly on the final strength level. Such behaviour could be an advantage during installation at the job site.
- Product "F" also achieved high strength values (in the best flow range, it showed an about 25 % lower value compared to Dolapix). CCS results fell slightly with increased water additions. A secondary flow effect was marginally visible.
- Product "A" has a very strong effect on secondary flow (bleeding) about 20 min after casting into the mould (Fig. 2). After 24 h, it was still difficult to demould it without causing any damage to the sample. In the tests setting accelerators were not used. That is the reason why this option was not considered for further investigation with MR42 reactive alumina. Further tests on this reactive alumina were not necessary, owing to the fact that the "best flow" val-

ues were already achieved during the first stage of this investigation.

For reactive alumina CT3000SG, as Fig. 1B shows, the behaviour of deflocculants was very different. DOLAPIX FF26 requires a higher content of water for proper deflocculation (>5 %); the products "F" and "A" started effective deflocculation already with <4,7 % water addition. Recorded strength values were also high in all cases. The decision was taken to follow up with tests to achieve flowability within 190–210 mm in all cases. A secondary flow effect was not observed this time.

- Using DOLAPIX FF26 – within the range of 4,7–5 % water addition – the product achieved a good flow value but not the "best flow" value (190–210 mm) within the recorded results, but the CCS was high. The decision was taken to follow up the result with a further test with an increase in the water level up to 5,2 %.
- Product "F" exhibited very good flowability already with 4,7 % water addition. The decision was taken to follow up with tests applying 4,5–4,6 % water. The CCS values obtained were high, but with 4,8–5 % water content, it led to an about 35-% reduction

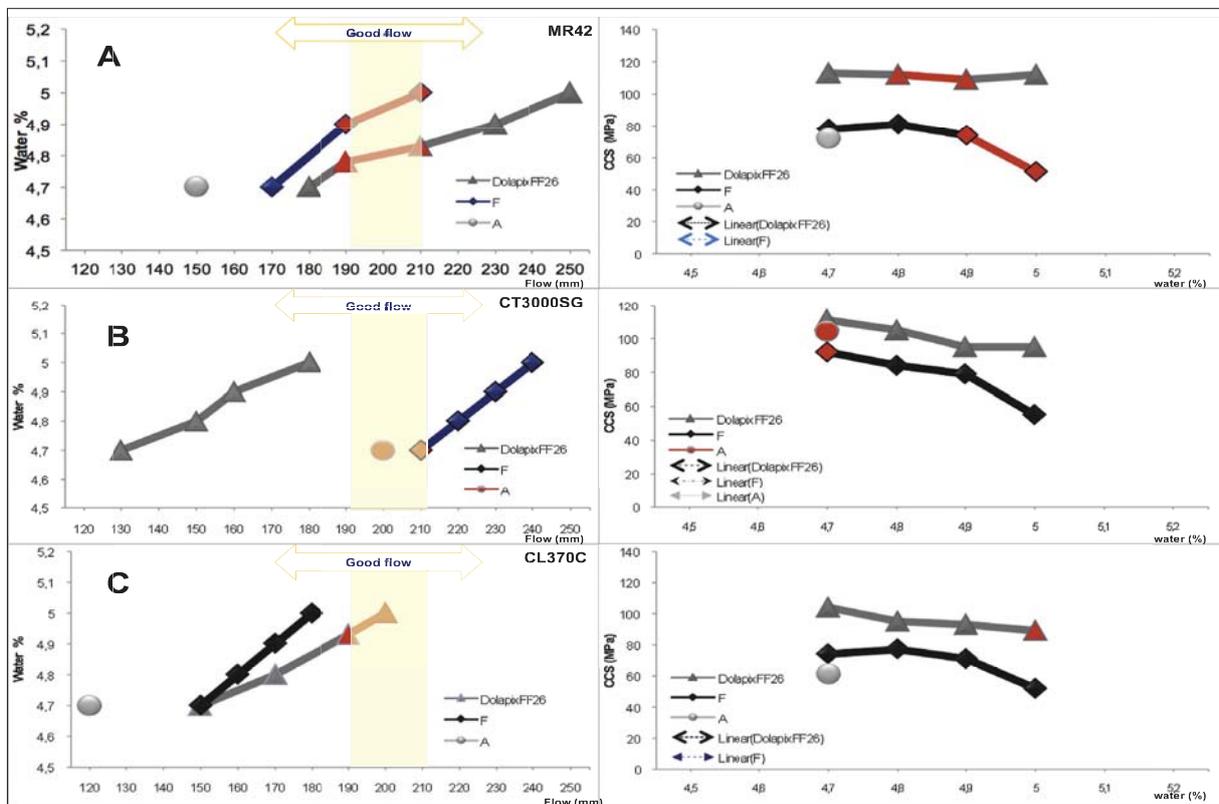


Fig. 1 Flow results of castables with each reactive alumina and performance of the deflocculants used (left); CCS results of the castables achieved during testing (right). The values within the "best flow range" (190–210 mm) [1] are marked red



Fig. 2 The product was cast directly into the mould (flowability value about 190 mm); within the first 10 min the consistency of the castable changed (small holes were visible on the surface); after about 20 min the surface of the castable became very rough with "cheese"-like holes within the complete structure. Such behaviour may be difficult to control on the job site during the installation of the product

in castable strength. This product was quite sensitive to changes in the water addition.

- Product "A" was tested with only 4,7 % water, achieving a very good flow value at this level. Further tests were conducted with this additive.

As shown in Fig. 1C for reactive alumina CL370C, according to the test results, the water demand of the LC castable was much higher. To achieve proper flow values and correct deflocculation, it was necessary to increase the water addition up to 5,2 % in the case of DOLAPIX FF26 and product "F". In the case of Product "A", it was difficult to achieve a "best flow" value, owing to its strong secondary flow effect already reached with 120 mm of flow.

- DOLAPIX FF26 obtained very good flow values with 5 % water addition. Further tests have to be followed up.
- Product "F" did not achieve the "best flow" range, so the decision was taken to increase the water addition in further tests.

Owing to the fact that with additive "A" a very strong "secondary flow" behaviour of the castable with MR42 and CT370C reactive aluminas was reached, the decision was taken not to conduct any further tests (Fig. 2). The "A" deflocculant was considered for further tests with CT300SG alumina.

Conclusion of the first results:

- With regard to the types of reactive aluminas used in the tests, it was clear that each of them influences the water demand of the castable and strongly impacts the behaviour of the chemical additive applied.
- The chemical additives perform differently depending on the matrix components and require different amounts of water for proper deflocculation.

- In some cases, the mechanical parameters (after drying) of the castable were strongly dependent on the reactive alumina used and chemical additive applied (for example: Product "F" showed a strong dependence on the water demand and CCS values of the castable).

- Sensitivity of the products with different chemical additives was established as shown in Fig. 2. Some of the additives can lead to a "secondary flow" (bleeding effect), which may lead to unexpected results during installation of the castable at the job site.

2.2 Second stage of the investigation

The decision was taken to follow up with further tests to achieve products with a flow within the best flowability range (190–210 mm) and to focus on the best flow values of about 200 mm. The water amounts were increased within the range of 4,5–5,2 %. All reactive aluminas were taken for the next tests. With regard to the deflocculants, Dolapix FF26 and Product "F" were considered for all tests and Product "A" only for "the best flow value" range with CT3000SG.

According to Fig. 3A, for the reactive alumina MR42 the water amount necessary for proper flow of DOLAPIX FF26 and Product "F" was achieved in the first part of the investigation in the range between 4,7–5 %. In view of the results from the "best flow" range (190–210 mm), further tests were not necessary.

For reactive alumina CT3000SG, the behaviour of the deflocculants differed (Fig. 3B). DOLAPIX FF26 requires a higher amount of

water for proper deflocculation (up to 5,2 % to achieve 200 mm); the products "F" and "A" start effective deflocculation at a water addition of 4,6 %. The recorded strength values were high in all cases:

- For DOLAPIX FF26 the best flow value was obtained after an addition of 5,1 % water. The strength value for LC castable with this additive was very stable through the entire range of water addition. In fact the difference in strength throughout the range was at a level of 5–10 % max. Such a performance may be a big advantage during installation at the job site, where it is very difficult to control the correct amount of water added.

- Product "F" already exhibited very good flow with 4,6–4,7 % water addition. A slightly higher amount caused an increase in flow beyond the expected range. The CCS values obtained were high but depended slightly more on the amount of water, which may in fact cause a 45-% reduction in the strength of the castable. The big advantage of this chemical is a strong reduction in the water demand of the LC castable.

- Product "A" was tested with 4,6–4,7 % water, achieving a very good flow value at this level. The recorded CCS was also very high. A small decrease in strength at 4,7 % was checked twice to confirm this phenomenon. Further tests concerning strength development and predicted sensitivity to water addition will be conducted in the future.

For reactive alumina CL370C, according to the test results (Fig. 3C), the water demand of the LC castable was much higher. To achieve proper flow values and appropriate deflocculation, it was necessary to increase the water

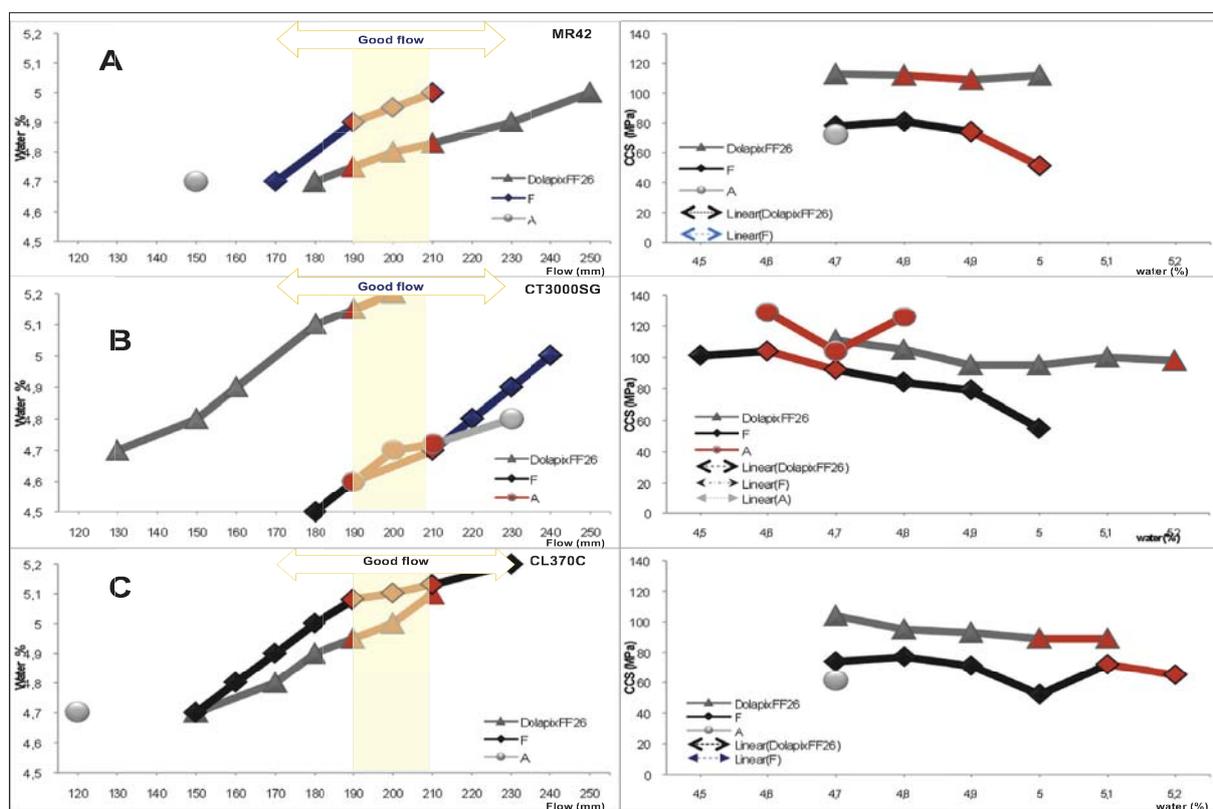


Fig. 3 Flow results of castables with water additions in the range from 4,5–5,2 %. Tests were conducted with each reactive alumina to show the performance of the deflocculants used (left); CCS results of the castables obtained during testing are presented on the right side. The values that are within “best flow” range are marked red (both on the flow curves and CCS)

addition up to 5,1 % in case of DOLAPIX FF26 and Product “F”:

- DOLAPIX FF26 resulted in a very good flow value within the 4,9–5,1 % range of water content. Tests confirmed steady strength development of the LC castable within this range. The product does not seem so sensitive to changes in the amount of water amount added.
- Product “F” exhibited a very good flow value at a water addition of about 5,1 %. It was proven that with just a slight addition of 0,1 % the flow values were already over the “best flow” range. CCS results tended to be constant (except at 5 %) and were 20 % lower than those obtained for the solution with DOLAPIX FF26.

2.3 Temperature dependence of the results

During installation or demoulding, the green strength of LC castable is crucial. Setting time and easy handling of prefabricated pieces or parts of a just installed lining may be the deciding factor with regard to selecting the solution and its performance. The aim of this study was to confirm the results of the “best

flow” value (200 mm) in various temperature conditions too.

The next samples were therefore tested at the following temperatures: 20 °C (green strength value), 110 °C (after drying), 900 °C, and 1500 °C (Fig. 4).

For reactive alumina MR42, the deflocculants DOLAPIX FF26 and product “F” were used (Fig. 4A):

- With DOLAPIX FF26 at water content 4,8 % (flow 200 mm) the green strength achieved was 37 MPa after 24 h; after drying in 110 °C the product had 112 MPa; at 900 °C the CCS was at 115 MPa accordingly at 1500 °C at 143 MPa. During demoulding no sticking of the product to the mould was observed.
- Product “F” achieved with a water content of 5 % (flow 200 mm) a green strength of 6 MPa after 24 h; after drying at 110 °C the product exhibited 51 MPa; at 900 °C the CCS was 63 MPa and at 1500 °C it was 125 MPa. During demoulding, some sticking of the product to the mould was observed.

Strength development was similar in both products but DOLAPIX FF26 showed a high

green strength, which may be an advantage with regard to handling after demoulding. It has been confirmed that application of both types of chemical additives will have no negative impact on properties changes within all temperatures range.

For the reactive alumina CT3000SG all three deflocculants were tested (Fig. 4B):

- With DOLAPIX FF26 at a water content of 5,2 % (flow 200 mm) the green strength achieved was 24 MPa after 24 h; after drying at 110 °C the product had 98 MPa; at 900 °C the CCS was 75 MPa and 170 MPa at 1500 °C. During demoulding, no sticking to the mould was observed.
- Product “F” with water content of 4,7 % (flow 200 mm) achieved green strength of 9 MPa after 24 h; after drying at 110 °C the product exhibited 92 MPa; at 900 °C the CCS was 85 MPa and respectively 168 MPa at 1500 °C. During demoulding, slight sticking to the mould was detected.
- For product “A” at a water content of 4,7 % (flow 200 mm) the green strength achieved was 23 MPa after 24 h; after drying at 110 °C the product reached

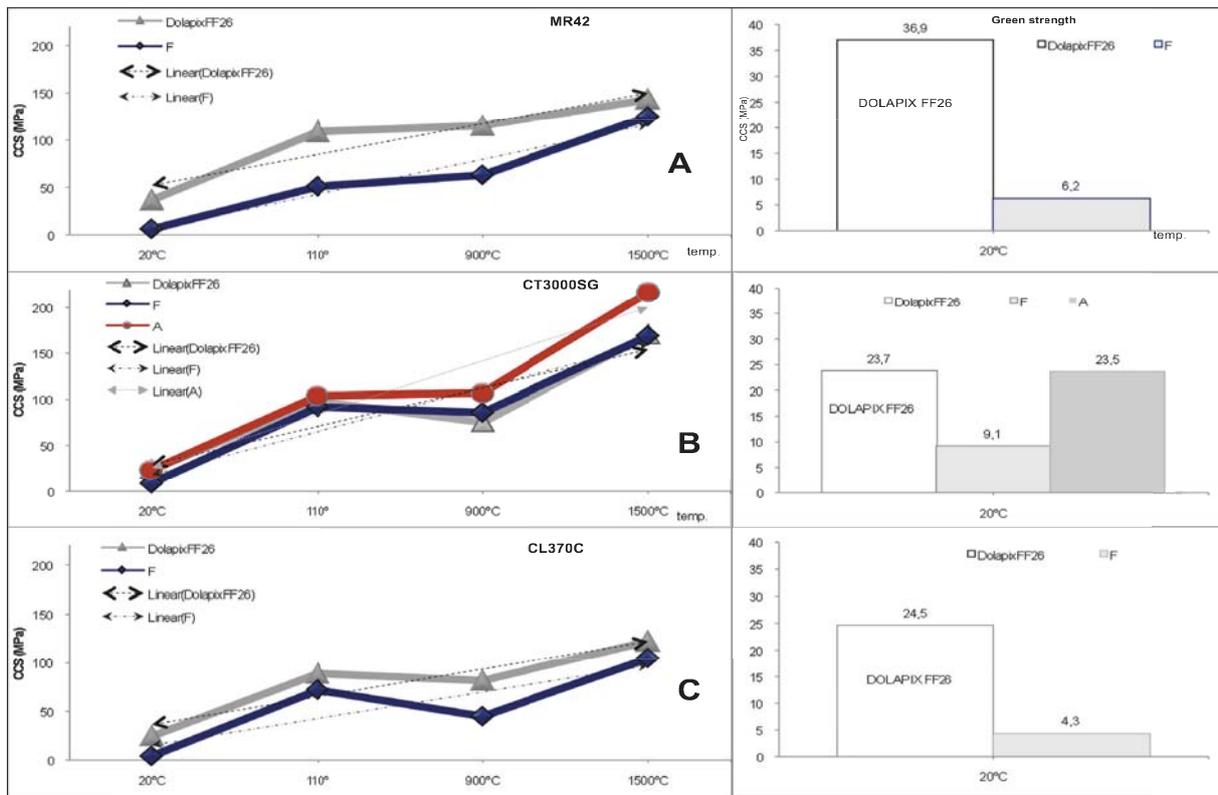


Fig 4 Compressive cold strength at ambient temperature (20 °C) and at 110 °C (after drying), 900 °C, and 1500 °C

104 MPa; at 900 °C the CCS was 107 MPa and 216 MPa at 1500 °C. During demoulding, no sticking to the mould was detected.

Strength development was similar in all cases. Product "A" showed significantly high CCS results already after demoulding. DOLAPIX FF26 and Product "F" exhibited very similar strength development at high temperatures. DOLAPIX FF26 and Product

"A" have achieved far better green strength results compared to Product "F", which may be more suitable for the application of pre-fabricated shapes.

For reactive alumina CL370C the deflocculants DOLAPIX FF26 and product "F" were used (Fig. 4C):

- DOLAPIX FF26 with a water content of 5,0 % (flow 200 mm) reached a green strength of 24 MPa after 24 h; after drying

at 110 °C the product had 89 MPa; at 900 °C the CCS was 82 MPa and 122 MPa at 1500 °C. During demoulding, no sticking to the mould was detected.

- For Product "F" at a water content of 5,1 % (flow 200 mm) the green strength achieved was 4 MPa after 24 h; after drying at 110 °C product had 72 MPa; at 900 °C the CCS was 44 MPa and

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104 MPa at 1500 °C. During demoulding, slight sticking to the mould was observed. Strength developed in a similar way in both products. DOLAPIX FF26 is characterized by the steady increase of the mechanical parameters (with just a small decrease of 7 MPa in 900 °C), what Product "F" showed in 900 °C circa was 50 % of CCS value decrease. It may have important consequences if this solution is applied in articles when working at low temperatures (for example in the aluminium industry).

Further strength development in both options is very similar, achieving about the same results at 1500 °C.

3 Summary

In this investigation, DOLAPIX FF26 demonstrated a good mechanical performance compared to the other deflocculants in all the tests. Strength development starts after casting and increases together with the rise in temperature. This deflocculant is not so sensitive within a wide range of water add-

itions, which makes it suitable also for standard installation on the job site. Green strength of the castables with different reactive aluminas was high, so handling and demoulding should be simplified. DOLAPIX FF26 may be applied in vibrated materials (subject of this study) as well as in self-flowing castables.

The authors would like to underline that this investigation is just the first step in a full study. We are aware that further changes in the formula, the origin and type of the raw materials may yield different results. Nevertheless, we can confirm that various changes in the matrix will influence the mechanical and rheological parameters of LC castables. On the basis of this study, we should like to direct your attention to some points, which may lead to better cost effectiveness and improve final product performance:

- For reactive alumina – as presented in this study – the right selection of deflocculant may allow the application not only of sophisticated and expensive types but also

standard options with similar effects in the castable.

- The deflocculation system has to be selected for the particular type of LC castable, and not just as one solution for all. In practice, it also means that the deflocculant should perform consistently and not react so sensitively to differences in the reactive alumina and/or water content changes. Experience from the job site confirms that the water addition and final product sensitivity may be very crucial there. Lower sensitivity of the deflocculant may be one of the options to consider.
- Another point is the handling of "fresh" castables. If the LC castable is too weak after demoulding, it may cause severe damage to the final product or the lining.
- The secondary flow is an effect that is very difficult to control on the job site.

4 References:

- [1] Krebs, R.: Ungeformte feuerfeste Erzeugnisse - Einführung" Sankt Augustin, Jan. 2009

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