

Effect of deflocculants on the rheological behavior of high alumina matrix suspensions for self-flowing bauxite castable

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Abstract. This study investigates the influence of commercially available polyphosphates and polycarboxylate ethers as deflocculants on aqueous suspensions containing high alumina cement (HAC), reactive alumina (RA), and various matrix compositions. Utilizing rheological methods, the research explores the interplay between dispersant concentration and the apparent viscosity and flow behavior of RA suspensions and matrix mixtures. The Casson model is employed to effectively describe the obtained flow data. Moreover, the study discerns specific impacts of polycarboxylate ethers on the thickening properties of matrix mixtures characterized by varying RA and HAC contents. Additionally, the research evaluates the physical and mechanical properties of low-cement bauxite castables treated with a range of polycarboxylate esters. The findings demonstrate that all deflocculated suspensions exhibit Non-Newtonian structured fluid behavior with a discernible yield stress (τ_0) at shear rates below 20 s^{-1} . At higher shear rates, there is a significant reduction in apparent viscosity, aligning well with the Casson model. Comparative assessments, based on flow curve values τ_0 , elucidate differing degrees of suspension flocculation depending on the type of dispersant used.

Keywords: Self-flowing castables, matrix suspensions, deflocculants, rheological behavior.

Classification numbers: 2.9.2.

1. INTRODUCTION

In recent years, a new generation of refractory castables, including low cement castables (LCC) and ultra-low cement castables (ULCC), with limited hydraulic binders such as high

aluminum oxide content calcium-aluminate cement (over 70%), have gained popularity [1]. These castables are heterogeneous binary composite materials with a matrix system that consolidates polyfractional aggregate grains [2]. The fillers have grain sizes ranging from 0.1 mm to 6 or 10 mm, while the matrix contains fine ingredients with particle sizes below 100 μm , mainly between 1 and 5 μm , and submicron particles below 0.1 μm . Typically, the matrix particles have a specific surface area exceeding 1 m^2/g [3, 4].

The development of special dispersant plasticizers has facilitated the incorporation of highly dispersive mineral particles into LCC and ULCC matrix systems, enhancing their flowability while reducing water requirements. Polycarboxylate esters (PCEs) have emerged as the most effective dispersing agents due to their comb-like structure, which includes a polycarboxylate main chain and polyester side chains [5, 6]. PCEs interact with mineral particles through electrostatic and steric effects, and their efficiency depends on various factors, including charge density and chemical structure [7-9].

Although previous research often recommended optimal dispersant concentrations as a proportion of the total mass of mineral components in LCC, this approach may not be suitable for understanding the effects of different deflocculants on casting systems. This is because dispersing effects primarily occur with highly dispersed components, which typically constitute only 25-35% of the total mass of LCC and ULCC. However, the matrix significantly influences the rheological properties of these materials, affecting workability, setting time, strength, and performance [10-13].

High alumina cement (HAC) plays a vital role in matrix mixtures due to its reactivity in hydration reactions with water. This results in the formation of highly dispersed hydrate phases and saturation with Ca^{2+} ions and $\text{Al}(\text{OH})_4^-$ [14]. The research demonstrated the impact of reactive alumina (RA) on setting processes, and certain additives showed retarding effects [15]. Furthermore, the flowability of vibration castables correlated well with matrix suspension viscosity at high shear rates. Another study investigated the dispersibility, setting, and hydration of high alumina refractory castables using matrix suspensions based on RA and HAC, yielding effective results [16].

This study extends previous research by evaluating different dispersant additives for both individual fine and reactive components, such as reactive α -alumina and HAC, as well as for functional matrix systems in general. These evaluations guided the preparation and testing of low-cement bauxite castable samples.

2. MATERIALS AND METHODS

2.1. Materials

Two polyphosphates, sodium tripolyphosphate (TPP) and sodium hexametaphosphate (HMP), as well as three commercial polycarboxylate ethers (PCE) - Peramin AL200, Peramin AL300 (Kerneos, France), and Sinteflow DM50 (Sintez Oka, Russia), served as dispersants. The matrix components comprised RA SMALOX-A and HAC. Both CA and RA were prepared by dry grinding the same alumina, following the method outlined in [17]. HAC was obtained through the grinding of high alumina clinker produced by heating a mixture of CaCO_3 and Al_2O_3 at 1450 $^\circ\text{C}$ [18]. Chinese bauxite was chosen for aggregate preparation. Detailed information regarding the physicochemical properties of these materials is provided in Tables 1 and 2 for reference.

Table 1. Chemical composition of the starting materials.

Materials	Weight (%)					
	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	CaO	TiO ₂	Na ₂ O
Calcined Bauxite	87.42	6.94	1.67	1.2	2.0	0.12
RA (SMALOX-A)	99.68	0.02	0.03	0.02	-	0.12
HAC	70.50	0.28	0.15	27.85	0.12	0.17

Table 2. Physical characteristics of the starting materials.

Materials	Granulometric composition					Primary phases
	D ₉₀ (μM)	D ₅₀ (μM)	D ₂₀ (μM)	< 1 μM (%)	BET (m ² /g)	
RA	8.3	2.4	0.9	22.5	1.4	α-Al ₂ O ₃
HAC	22.5	5.4	1.5	7.5	0.41	CA ^a - 65 % CA ₂ ^b - 35 %

CA^a - CaAl₂O₄(CaO·Al₂O₃); CA₂^b - CaAl₄O₇(CaO·2Al₂O₃).

2.2. Preparation of casting mixture

The high alumina model matrix mixture (MM) comprised RA SMALOX-A and HAC in two compositions: MM1 (90% RA, 10% HAC) and MM2 (80% RA, 20% HAC). Deflocculants were added in the following quantities: PCE at 0.17 and 0.34 wt.% and polyphosphate at 0.1 and 0.25 wt.% relative to MM. Polyphosphate deflocculants were combined in a 1:1 ratio, referred to as Peramin, and supplemented with 0.1 wt.% citric acid when used. HAC, RA, MM1, and MM2 suspensions were prepared by mixing powdered minerals in a plastic beaker to reach a minimum volume of 150 cm³. After 1 min of mixing, the necessary distilled water with dissolved deflocculant was added and mixed with a hand mixer for 3 min until achieving a homogeneous suspension.

Castables were made using a planetary mixer. Dry ingredients were mixed for 4 min, followed by the addition of water and another 4 min of mixing. The resulting mixture was cast into metal molds (50 × 50 × 50 mm³) without vibration. After curing at 20 °C and 90% relative humidity for 24 h, samples were demolded, dried at 110 °C for 24 h, and fired at 800 °C, 1300 °C, and 1500 °C for 3 h.

2.3. Methods

The rheological behavior of aqueous suspension was studied with a Visco Star plus rotary viscometer (Fungilab, Spain) in the range of shear rates from 5 to 100 s⁻¹. The flowability of the HAC, RA, MM1 and MM2 casting systems was evaluated using an Engler viscometer with a 5.4 mm outlet. Suspensions were mixed, rested for 30 s, and then flow time to reach 100 mL of suspension was recorded as F₀. After 30 min, the second flowability measure, F₃₀, was determined. The Thickening Index (Th) was calculated as Th = F₃₀/ F₀.

Self-flow tests were performed according to ISO 1927-5:2012 standards. The castable mixture was prepared using a planetary mixer and distilled water, mixed for 5 min at low speed,

then placed in a sealed container to maintain humidity. The castable was poured into a standard mold, and the percentage increase in spreading diameter after 60 s determined self-flow value (SF) using the formula $SF = (D2 - D1)/D1 \times 100\%$, where D2 is the final average diameter, and D1 is the initial diameter (100 mm). A castable is considered self-flowable when SF falls within the 80-110% range. SF evaluation occurred at a controlled ambient temperature of 20-25 °C.

Bulk density (BD), apparent porosity (AP), and cold crushing strength (CCS) were determined after drying and firing at various temperatures. BD and AP of dried and fired samples were determined hydrostatically, while CCS tests were conducted on an Instron 8801 servo-hydraulic testing machine. Presented data represent the average values for five samples of each composition and firing temperature.

3. RESULTS AND DISCUSSION

Initially, the rheological properties of aqueous RA suspensions, as well as those of model matrix mixtures with varying concentrations of dispersing agents based on polyphosphate and polycarboxylate deflocculants, were examined. In all experiments, the suspensions had a solid content of 80 wt.%. The rheological curves of $\mu = f(\dot{\gamma})$ and $\tau = f(\dot{\gamma})$, which illustrate the effects of the deflocculants HMP, TPP, Peramin, and DM 50, are depicted in Figs. 1 - 3, respectively.

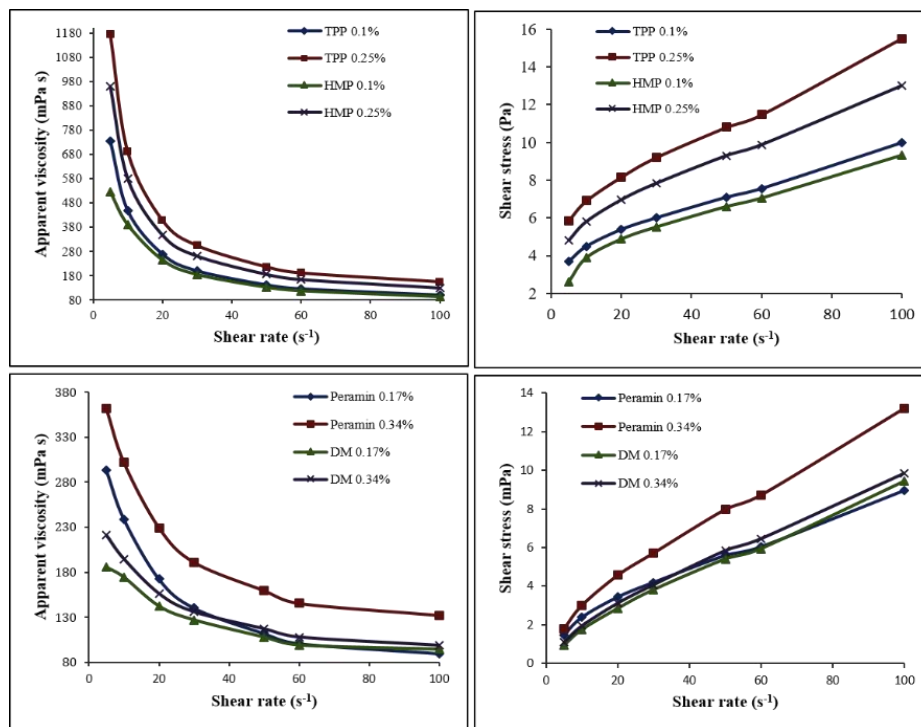


Figure 1. The effect of deflocculants content on the rheological behavior of reactive alumina suspensions.

As demonstrated in Fig. 1 to 3, all the deflocculated suspensions exhibit Non-Newtonian behavior and display pseudoplastic flow characteristics. The highest apparent viscosity (μ) values are observed at low shear rates, specifically between 5 and 10 s^{-1} . For RA suspensions with polyphosphate and PCE additives at a shear rate of 5 s^{-1} , the apparent viscosity (μ) ranges from 520 to 1170 mPa·s and 185 to 360 mPa·s, respectively.

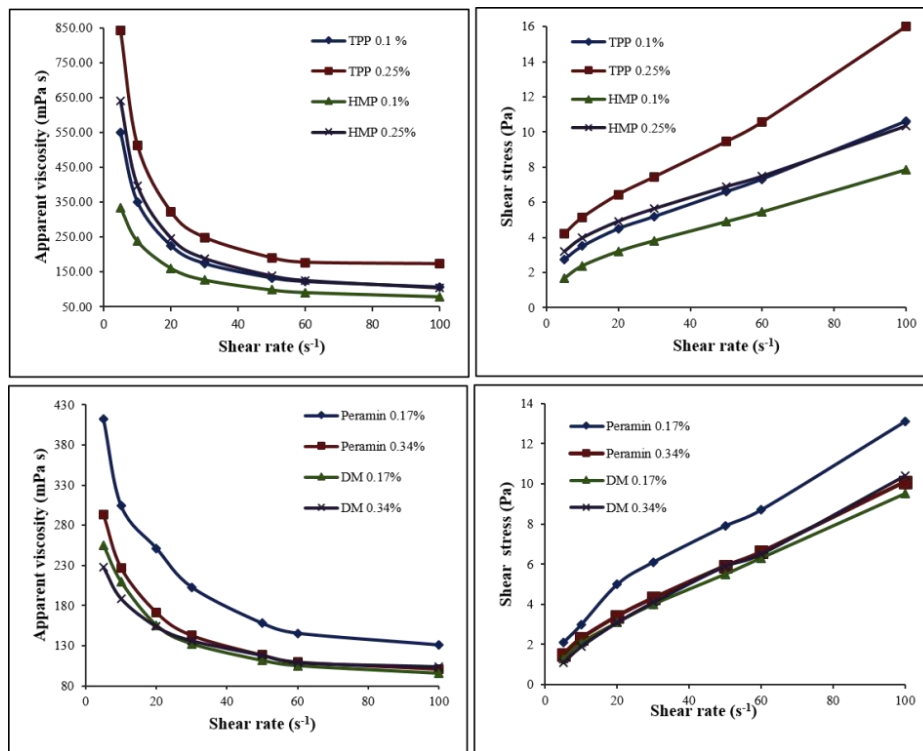


Figure 2. The effect of deflocclants content on the rheological behavior of MM1 matrix suspensions.

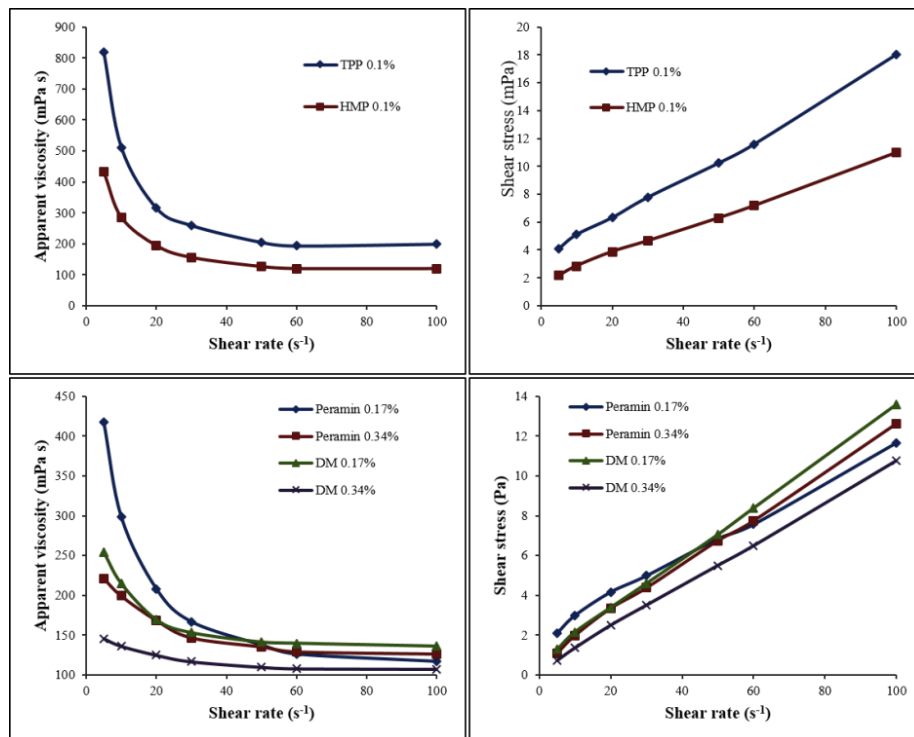


Figure 3. The effect of deflocclants content on the rheological behavior of MM2 matrix suspensions.

The obtained rheological curves $\mu = f(\dot{\gamma})$ and minimum viscosity values of suspensions RA at the optimum concentration of TPP and HMP 0.1 wt.% correlate well with the data of the study [20]. Increasing the concentration of all kinds of deflocculants generally leads to an increase in viscosity.

In the case of matrix suspensions with polyphosphate and PCE additives at $\dot{\gamma} = 5 \text{ s}^{-1}$, μ values for composition MM1 are in the range of 330 - 840 mPa·s and 230 - 410 mPa·s, respectively; and for composition MM2 - 430 - 820 mPa·s and 145 - 420 mPa·s, respectively. It should be noted that increasing polyphosphate concentration from 0.1 to 0.25 wt.% increases apparent viscosity of matrix suspensions, while increasing PCE concentration from 0.17 wt.% to 0.34 wt.%, on the contrary, decreases viscosity.

When the shear rate exceeds 20 s^{-1} , the apparent viscosity of all examined suspensions decreases significantly, and the $\tau = f(\dot{\gamma})$ relationships become linear. At a shear rate of 100 s^{-1} , the μ values for deflocculated RA suspensions, as well as matrix suspensions MM1 and MM2, fall within the range of 90 to 130 mPa·s. The measured flow curves $\tau = f(\dot{\gamma})$ were satisfactorily fit to the Casson model [21]. The Casson model equation is:

$$\tau^{1/2} = \tau_0^{1/2} + (\mu_p \dot{\gamma})^{1/2} \quad (1)$$

here, τ denotes the shear stress, $\dot{\gamma}$ represents the shear rate, τ_0 stands for the yield stress, and μ_p signifies the plastic viscosity at high shear rates.

The reliability of the R approximation is indicated by an R^2 value greater than 0.99. The Casson model parameter was employed to indicate the degree of suspension flocculation (Fig. 4-5). Fig. 4 shows that the maximum values of τ_0 are observed for RA suspensions dispersed with polyphosphate additives (2045 - 4000 mPa). By increasing the amount of polyphosphate from 0.1 wt.% to 0.25 wt.% τ_0 increases by a factor of about 1.6. In the case of PCE additives, τ_0 in the same suspensions is 200 - 700 mPa.

A similar character of different types of deflocculants influence on τ_0 is observed for matrix suspensions of compositions MM1 and MM2 (Fig. 5). Thus, with the introduction of polyphosphates in an amount of 0.1 wt.% and 0.25 wt.%, the values of τ_0 are in a range of 820 - 2245 mPa, and with the introduction of PCE in an amount of 0.17 wt.% and 0.34 wt.% - in a range 125 - 800 mPa. Thus, dispersants based on polycarboxylate esters are more effective in reducing the degree of flocculation of matrix suspensions compared to polyphosphates.

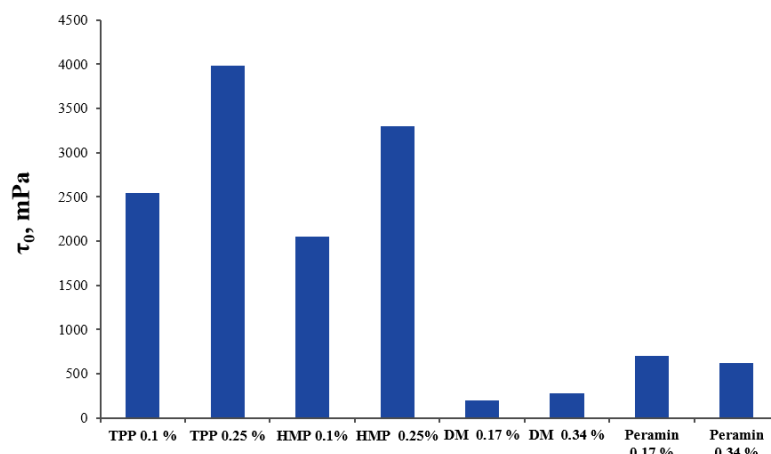


Figure 4. The effect of deflocculants content on τ_0 RA suspensions (80 wt.% solid content).

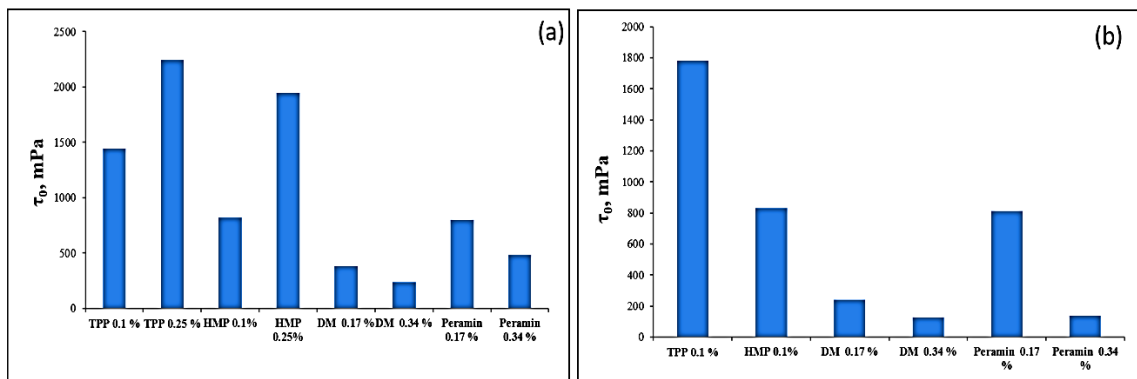


Figure 5. The effect of deflocculants content on τ_0 matrix suspensions (80 wt.% solid content): a – MM1 (90 wt.% RA and 10 wt.% HAC); b – MM2 (80 wt.% RA and 20 wt.% HAC).

In the second phase of the study, the effect of the type and amount of deflocculants on the flowability of HAC suspensions, RA suspensions and matrix suspensions were investigated. The flowability of all casting system samples was assessed using an Engler viscometer immediately after preparation of the suspensions and after 30 min of exposure. The test results are shown in Table 3 and Figs. 6 - 7.

Table 3. Flow of cement slurries (78 wt.% solid content).

Deflocculant	Wt.%	$F_0(c)$
DM	0.17	146.5
DM	0.34	110.8
Peramin	0.17	164.0
Peramin	0.34	116.5

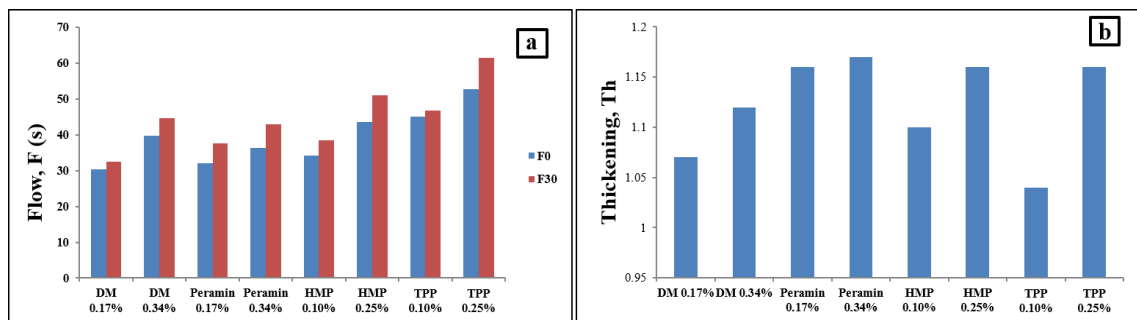


Figure 6. The effect of deflocculants content on flow (a) and thickening (b) RA suspensions.

The addition of polycarboxylate deflocculants enhances flowability in HAC suspensions, with comparable F_0 values observed for PCE DM and Peramin. Increasing deflocculant concentration leads to reduced F_0 values, as shown in Table 3. After 30 min, sedimentation resulted in dense sludge, making it challenging to measure flowability in HAC suspensions. Suspensions with polyphosphate and citric acid, as well as the reference suspension without

deflocculants, lacked fluidity. For RA suspensions (80 wt.% solid content), the addition of polyphosphate and polycarboxylate deflocculants significantly improved fluidity, with increases of 5 to 7 times compared to the reference suspension ($Th = 1.78$). Among polyphosphate deflocculants, HMP had the most significant impact, and further increases in the additive amount did not notably affect fluidity (Fig. 6a). Overall, polycarboxylate ester additives outperformed polyphosphates, with the most substantial and comparable improvement achieved using PCE DM and Peramin at a concentration of 0.17 wt.% with F_0 values of 30.3 s and 32.1 s, respectively. Notably, Th values were close to unity for all tested deflocculants, indicating rheological stabilization of aqueous RA suspensions within 30 min (Fig. 6b).

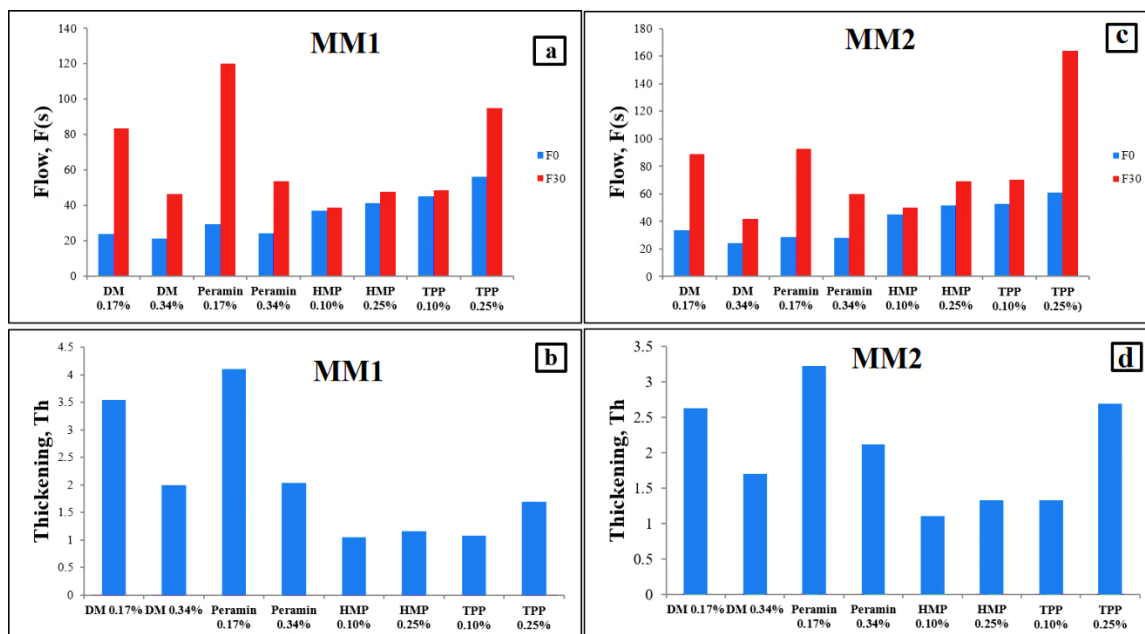


Figure 7. The effect of deflocculants content on flow (a, c) and thickening (b, d) matrix suspensions for MM1 and MM2

The influence of these deflocculants on the fluidity of MM1 and MM2 aqueous matrix suspensions (RA:HAC weight ratios of 90:10 and 80:20, respectively) was then examined, with a solid content of 80 wt.% (Fig. 7). Matrix mixtures without dispersant addition exhibited high viscosity pastes. As shown in Fig. 7a&c, HMP and TPP had limited impact on matrix suspensions, and an increase in polyphosphate concentration from 0.1 wt.% to 0.25 wt.% reduced flowability and increased thickening, especially with TPP. PCE-based dispersants demonstrated significant deflocculating effects in matrix suspensions. When 0.34 wt.% PCE was introduced, F_0 values for MM1 and MM2 compositions ranged from 24.4 to 28.2 s and 22.2 to 28.8 s, respectively.

The concentration of PCE additive significantly influenced Th values (Figs. 7b&d). At low deflocculant concentrations, Th values for MM1 and MM2 reached maximum values of 3.5-4.1 and 2.6-3.2, respectively. Increasing the additive amount to 0.34 wt.% slowed down suspension thickening, reducing Th values to 2.0 for MM1 and 1.7 for MM2. In contrast, HAC and RA suspensions with added PCE showed minimal thickening within 30 min due to the stabilization of casting systems within the studied range of PCE concentrations. Matrix suspension thickening primarily resulted from time-dependent hydration processes of HAC, leading to the coagulation

of hydrate dispersions. PCE molecules dissolved in the aqueous medium and were adsorbed onto fine mineral particles, blocking the interaction of cement with water, thus slowing down the formation of hydrate phases and coagulation.

For matrix mixtures with selected RA:HAC weight ratios of 90:10 (MM1) and 80:20 (MM2), PCE adsorption was more significant on RA particles due to their higher contribution to the total surface area (approximately 90 - 95 %). At high PCE concentrations (0.34 wt.%), sufficient deflocculant molecules blocked cement particles, slowing HAC hydration and reducing gelling in the first 30 min. At low PCE amounts (0.17 wt.%), insufficient deflocculant molecules led to intensified hydration and coagulation, resulting in increased Th values. Matrix suspensions deflocculated with polyphosphates, utilizing a linear structure and electrostatic dispersion mechanism, did not exhibit these patterns. Additionally, the presence of 0.1 wt.% citric acid significantly slowed down HAC hydration in matrix suspensions, with no significant change in Th values observed within 30 min of exposure.

In the final step, based on the rheological test results, self-flow bauxite castable samples were prepared using PCE as deflocculants (Table 4). Deflocculants and water were introduced in excess of 100%. Filler fraction ratios were chosen based on Dinger and Funk's packing model with a distribution modulus of $q = 0.23$ for self-flow castables. Rheological testing confirmed that adding 6 wt.% water resulted in well-flowing Castable 1 and Castable 2 with self-flow values of 93 % and 91%, respectively.

Table 4. Composition of self-flowing Bauxite Castables.

Components	Weight %	
	Castable 1	Castable 2
Calcined Bauxite, 0 – 6 mm	70	70
RA, SMALOX-A	24	24
HAC	6	6
Deflocculant Peramin AL200/AL300(1:1)	0.1	-
Deflocculant Sinteflow DM 50	-	0.1
Water	6	6

The results of determining the physical and mechanical properties of bauxite castables after drying and firing are shown in Table 5.

Table 5. Characteristics of Low-Cement Bauxite Castables.

Sample	110 °C			800 °C			1300 °C			1500 °C		
	BD, g/cm ³	AP, %	CCS, MPa	BD, g/cm ³	AP, %	CCS, MPa	BD, g/cm ³	AP, %	CCS, MPa	BD, g/cm ³	AP, %	CCS, MPa
Castable 1	2.95	11.8	79.1	2.90	15.3	67.0	2.94	14.1	78.4	2.98	10.8	129.9
Castable 2	2.98	11.0	83.4	2.92	14.7	70.5	2.96	14.0	87.1	3.00	10.2	134.5

The table of Bauxite Castables made with Peramin AL200/AL300 and DM 50 deflocculants shows comparable and sufficiently high physical and mechanical properties over

the entire processing temperature range. After drying at 110 °C and firing at 1500 °C, the BD and AP values are nearly equal. After baking at 800 °C, there is a slight increase in apparent porosity (up to 15.3 %), which is accompanied by a decrease in mechanical strength to 67 - 70 MPa. This is due to an increase in structure defects as a result of dehydration of the cement stone. After firing at 1300 °C, there is a reduction in apparent porosity and an increase in bulk density as a consequence of sintering processes. The maximum CCS values (up to 134 MPa) are reached after firing at 1500 °C.

4. CONCLUSIONS

Rheological analysis revealed the impact of polyphosphate and polycarboxylate additives on the deflocculation efficiency of HAC, RA, and matrix mixtures. Suspensions exhibited non-Newtonian behavior with a yield stress τ_0 at shear rates below 20 s^{-1} , transitioning to straightforward behavior at higher shear rates. The Casson model effectively described the flow curves $\tau = f(\dot{\gamma})$, allowing for comparative assessment of flocculation with different dispersants. Flowability correlated well with rheological relationships $\mu = f(\dot{\gamma})$ and $\tau = f(\dot{\gamma})$. For RA suspensions, higher polyphosphate (0.25 wt.%) and PCE (0.34 wt.%) concentrations decreased fluidity, while matrix mixtures benefited from increased deflocculant concentrations. Polycarboxylate esters were more effective than polyphosphates in reducing matrix suspension flocculation. The incorporation of polycarboxylates, specifically DM 50 and Peramin AL200/AL300, yielded good deflocculation of HAC suspensions (78 wt.% solid content), while TPP and HMP additives did not. Matrix suspensions thickened due to coagulation processes from HAC hydration, alleviated by introducing 0.34 wt.% PCE. Low-cement bauxite castables prepared with Peramin AL200/AL300 and DM 50 deflocculants exhibited comparable physical and mechanical properties, with maximum CCS values of up to 134 MPa achieved after firing at 1500 °C.

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Declaration of competing interest. The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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