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Design of bauxite-based low-cement pumpable castables: a rheological approach

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Abstract

The growing demand for pumping of refractory castables, as a high efficient placing technique, has stimulated the development of compositions with specific rheological properties. In this work, a new type of rheometer has been used to measure the rheological behaviour and to predict the pumpability of bauxite-based low-cement castables. This overcomes the shortages of traditional techniques based on either flow measurements or working with the matrix portion of castable. The parameters which strongly influence the rheological properties and pumpability of bauxite-based low-cement castable such as particle size distribution, ratios of coarse to matrix and water to matrix, water addition and time have been studied. It has been found that the optimal flow resistance and torque viscosity are two important properties to be taken into consideration while designing a castable.

Keywords: A. Mixing; E. Refractories; Rheological properties

1. Introduction

In the late 1990s, a new installation method known as shotcreting (a term from concrete industry equivalent to ‘‘wet-spraying’’ or ‘‘wet-gunning’’ in castable technology) has been attracting more and more interests because of its simplicity in installation [1–4]. In this process, refractory ingredients (aggregates, fine powders, binder and dispersing agent) premixed with water are transported with the help of a pump to a nozzle where some accelerators are added and projected at high velocity onto a receiving surface.

In case of pumpable castable, only the wet mix is directly transported by a two-piston pump to the application field. It is possible to obtain similar properties of high strength and density as that of vibrated castables. The main advantages are:

- No nozzle-man needed.
- No rebound thus saving raw materials.
- Dust and noise free process and hence friendly to the installation environment.
- High efficiency process (less labor and installation time).
- Installation of complicated or random thickness lining.
- Homogeneous installation and no lamination.

Compared with normal self-flow castable, pumpable castable not only needs to flow well in pipe, but also to maintain self-flowability after pumping. So the following characteristics are required:

- Good self-flowability during the whole process.
- Stable flowability and no segregation leading to blockage.
- Optimum working time and no setting inside pipes.
- Easy cleaning-up.

For a good pumpable castable, the rheological properties are lethal factors governing the whole pumping process. Since castable refractories consist of aggregates, fine powders, dispersants and water, the study of flow behaviour of an aggregate-matrix-water system is quite important to improve the fluidity of castables.

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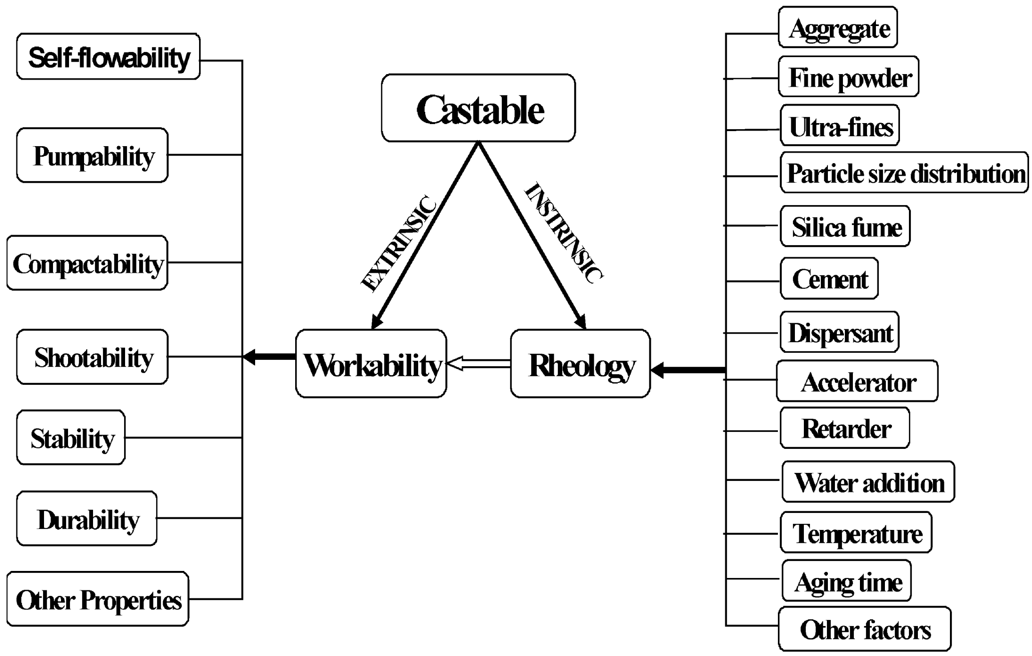


Fig. 1. Factors influencing rheology of castables.

The factors influencing the rheological behaviour of castables are shown in Fig. 1.

From the earlier discussions, it is understood that the study of rheology of castable are important to have better performance during installation. Unfortunately, most of the researchers worked only on rheology of fine matrix of castable and attempted to correlate the behaviour of castable mix with fine matrix rheology [5–8]. In practice, it is found that when coarse aggregate is introduced into matrix, there is a drastic change in rheological behaviour of castable. This necessitates one to consider the study of rheology of castable rather than considering the rheology of fine matrix. In this context, the present work is focused on the influence of particle size distribution (PSD), coarse to matrix (C/M) ratio and water to matrix (W/M) ratio on rheological behaviour of bauxite-based pumpable castables.

2. Experimental work

2.1. Raw materials

Chinese high-grade bauxite (density: 3.4 g/cm^3, Al_2O_3: 85%) has been used as coarse aggregates. The size fractions used were: 8–5, 5–3, 3–1, and 1–0.21 mm. The fine matrix consisted of three fine bauxites of the same quality as aggregates (Bauxite-1, Bauxite-2 and Bauxite-3), micro-silica (971U, Elkem Materials) and calcium alumina cement (CA-14, Alcoa). Bauxite-1 is-200 mesh in size, Bauxite-2 is-325 mesh in size and Bauxite-3 is ultra-fine powder. Sodium hexametaphosphate (SHMP) was used as dispersant.

2.2. Particle size distribution

The PSD of fine matrix components were carried out using a particle size analyser (Coulter LS200, USA). The mean particle size values are 25.78, 8.10, 6.74, 12.13 and 0.15 microns for Bauxite-1, Bauxite-2, Bauxite-3, CA-14 cement and microsilica, respectively. The PSD of chosen bauxite fines are shown in Fig. 2 and all are bi-modal in distribution. Fig. 2(a) gives the differential volume% whereas Fig. 2(b) gives the cumulative vol.% of PSD. Bauxite-1 is coarser when compared with Bauxite-2 and Bauxite-3. The PSD of castable mixes have been designed using Andreasen's equation:

$$\mathrm{CPFT} = 100 \times (d/D_{\max})^q \quad (1)$$

where, CPFT is the cumulative percentage finer than, d is the particle size, $D_{\max}$ is the largest particle size and q is the distribution modulus. The details of the castable

Table 1
Details of Castable mixes

Composition	CM-1	CM-2	CM-3	CM-4
Andreasen modulus, q	0.23	0.26	0.29	0.26
$D_{\max}$, mm	5	5	5	8
C/M ratio	1.22	1.44	1.70	1.56
W/M ratio	0.16	0.17	0.19	0.18

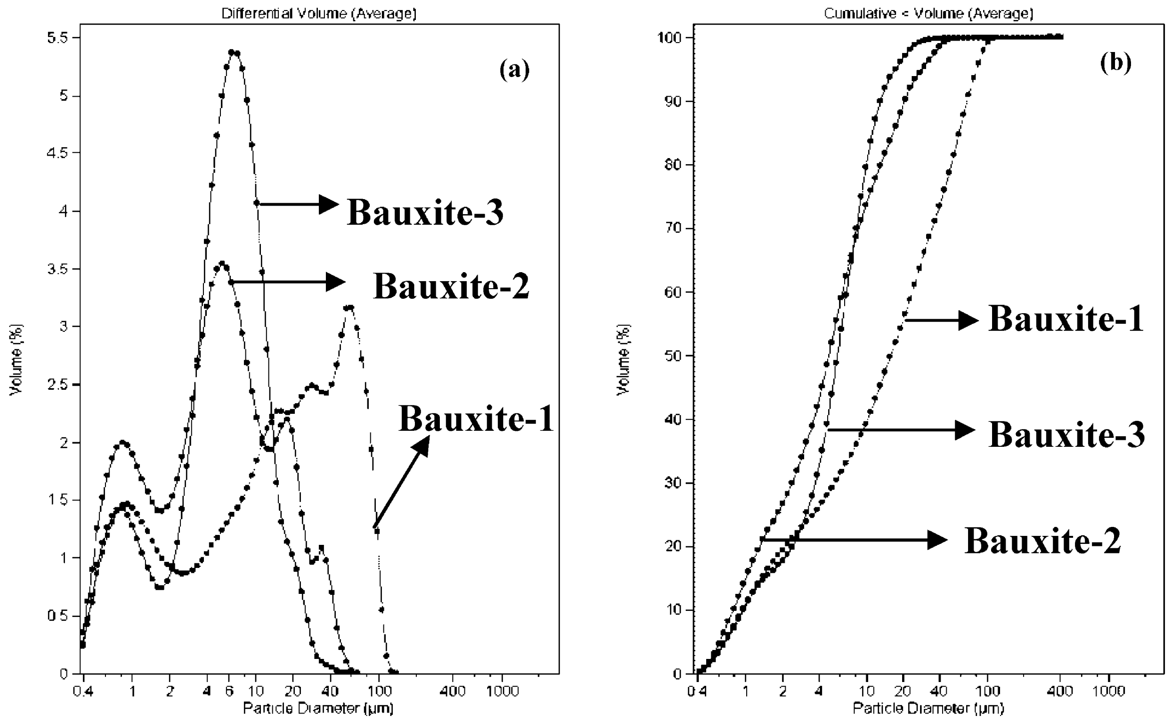


Fig. 2. PSD of three fine bauxite powders.

mixes are given in Table 1. The q values were 0.23, 0.26 and 0.29 and D_{max} was 8 and 5 mm for the present investigation. In this work, the cement and micro-silica contents were fixed at 4 and 5 wt.% respectively. The amount of water was fixed at 7 wt.% for all compositions. The reason for fixing the water amount will be discussed in Section 3.1.

2.3. Samples preparation and testing

Castable mixing was carried out using the two-step water addition method. Castable mix (8 kg) was dry mixed for 5 min using Hobart mixer and 80% of total water was added and mixed for 3 min. The remaining water was added to wet mix and mixed further 3 min at the same speed. Self-flow measurements were performed using a flow cone as described in ASTM C860. Two measurements were made for each mix immediately after wet mixing and after 30 min. For rheological measurements, the wet mix was poured into the bowl of rheometer and the relationship between torque and speed was measured at the interval of 5 min for the total time of 45 min. The description of the rheometer is given in Section 2.4.

Samples of 160 mm×40 mm×40 mm prisms were cast in steel moulds by self-flow method, cured at room temperature for 24 h, demoulded and then dried at 110 °C for 24 h. Cold modulus of rupture (CMOR) and apparent porosity (AP) were performed as per ASTM C 133-97 and C 830-93 respectively.

2.4. Rheometer

IBB Rheometer V1.0 designed by Denis Beaupré [9] consists of an impeller and a sampling bowl. During testing, the impeller is driven by a motor at different speeds, through the bowl previously filled with castable, and the required torque and the impeller speed were measured. The major advantage of this kind of rheometer is that the rheological measurements can be made directly on castable unlike viscometer, which measures the rheology of fine matrix of castable only. The torque to drive an impeller (T), flow resistance (G), impeller angular speed (N) and torque viscosity (H) are correlated by the following equation:

$$T = G + (H \times N) \quad (2)$$

where, T and G are in Nm, H and N are in Nm.s and rev/s, respectively. The G and H are proportion to yield stress τ_0 (Pa) or plastic viscosity μ (Pa.s) when corrected with proper calibration factors. Both G and H values of castables are affected by the geometry of the apparatus in which they are measured. The value of G can be drastically changed by choosing proper type and amount of additive while keeping H at optimal value. By proper calibration, these constants (G and H) can be converted into the fundamental units of yield stress τ_0 (Pa) and apparent viscosity μ (Pa.s) so that the rheological parameters G and H can be used to evaluate the castable mobility or other related properties although they are not expressed in fundamental units [9].

3. Results and discussion

3.1. Self-flowability

The self-flowability of chosen compositions as a function of water is shown in Fig. 3. The compositions CM-3 and CM-4 have shown more self-flow for 6 wt.% water compared to CM-1 and CM-2. The same trend is observed with 6.5 wt.% water. With 7 wt.% water addition, the self-flow of all compositions are almost same and further addition of water does not improve flow (segregation observed) in all cases and hence not shown. Considering the minimum self-flowability as 100% for pumpable castable, the water amount for the present investigation has been fixed at 7 wt.%.

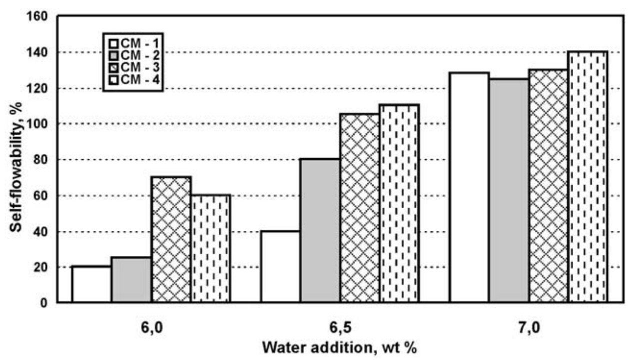


Fig. 3. Self-flowability vs. water addition.

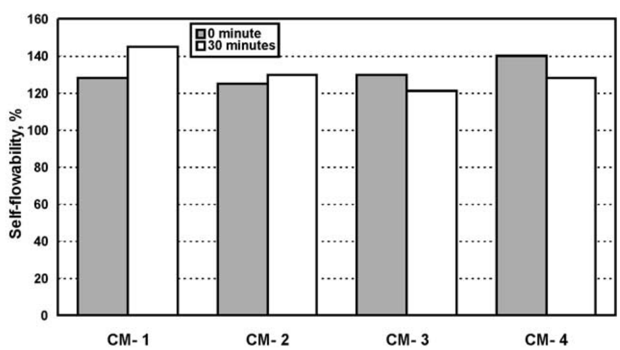


Fig. 4. Comparison of self-flowability of castables.

The self-flowability of different compositions measured immediately after mixing is compared with the self-flow values measured after 30 min (optimum working time) in Fig. 4. It is noted that at 0 min, self-flowability for all compositions looks similar at around 120%. After 30 min of rheological testing, CM-1 has shown considerable increase in self-flowability while observing little increase for composition CM-2. In case of composition CM-3 and CM-4, there has been a decrease in self-flowability which is the result of segregation. It should be noted that the compositions CM-4 and CM-2 differ only in D_{max} value. This indicates that it would be better to limit the maximum particle size below 8mm for pumpable castables, under such conditions, with 7 wt.% water.

3.2. Rheometer results

Typical rheometer results are shown in Fig. 5 for composition CM-1. The hysteresis loops in these figures represent the relationship between torque and impeller speed for castable mix after 0, 5, 10, 15, 20, 25, 30, 35, 40 and 45 min of rheological testing. In Fig. 6, at 0 min, the forward cycle reveals Pseudoplastic nature of the mix while the reverse cycle displays almost Bingham behaviour (Newtonian with Yield). In the forward cycle, the rate of increase of torque is decreasing with testing

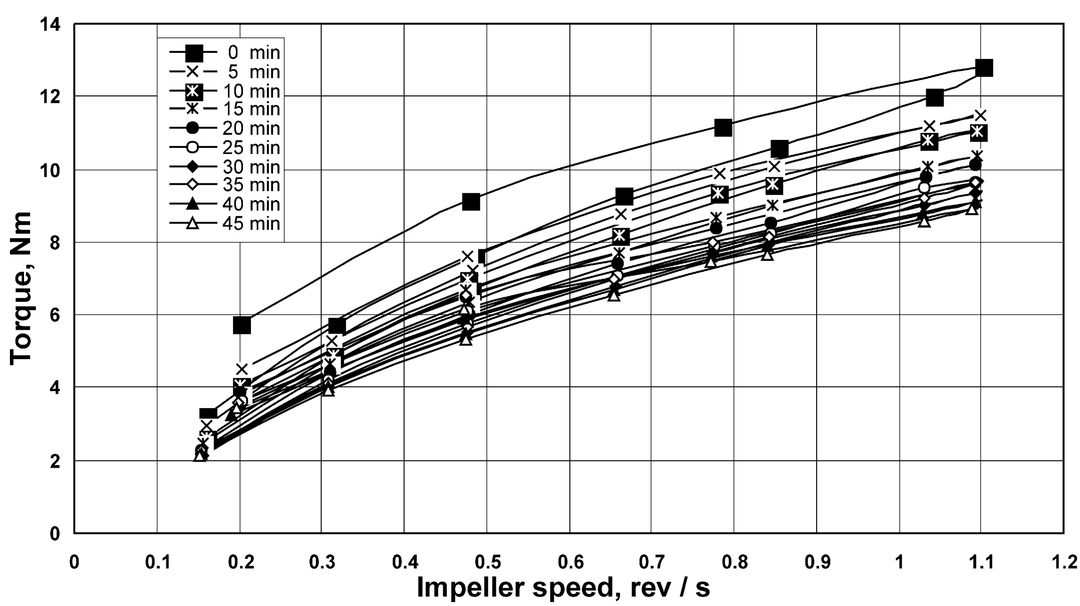


Fig. 5. Torque vs. impeller speed for CM-1.

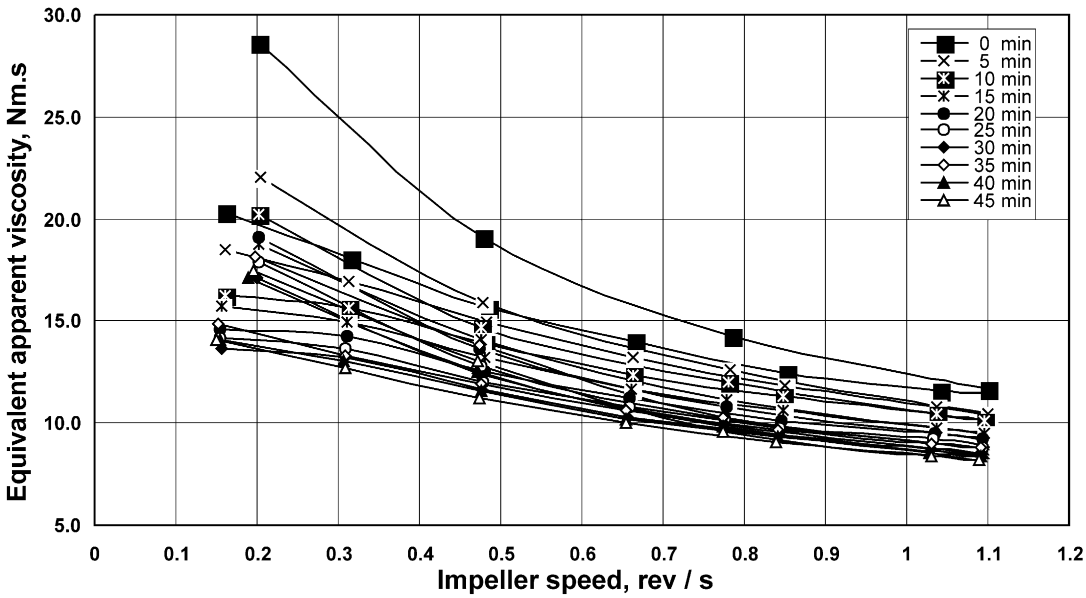


Fig. 6. Equivalent apparent viscosity vs. impeller speed for CM-1.

speed and the mix is getting homogenized. Once it reached maximum testing speed, the homogeneous mix behaves almost linearly in the reverse cycle. In the same test, after 5 min, there has been a drastic reduction of the hysteresis loop's area when compared with 0 min loop. The loops area remains almost the same then after 10, 15, 20, 25, 30, 35, 40 and 45 min but shift downward (i.e. rate of decrease of torque with speed decreases) till 20 min. and then follows almost similar path. The same kind of behaviour has been observed for the other compositions but with different rates of change of torque with speed, leading to different G and H values as shown later on.

In the beginning of the test, the mix cannot be considered as completely mixed and gains in homogeneity with mixing speed. The shearing stress helps to the breaking of agglomerates, and to fill the voids and improve dispersibility, due to the post mixing action of the impeller. Once the mix reaches the maximum speed, the mix is in a more homogeneous state and then shows Bingham behavior in the reverse cycle. Further on, in the testing cycle, the mix is more and more homogeneous and reaches after 20 min a state where further homogenization with mixing action is not possible. The relationship between equivalent apparent viscosity and impeller speed is shown in Fig. 6, always closely typical

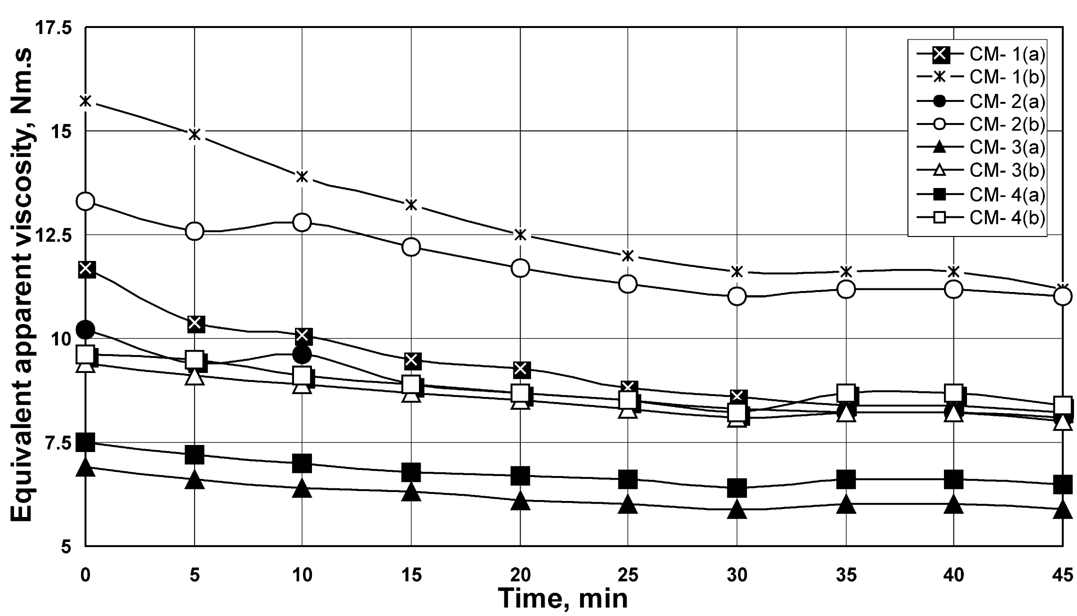


Fig. 7. Equivalent apparent viscosity vs. time at high (a) and low (b) impeller speed.

to a Bingham fluid. Though the values of equivalent apparent viscosity are not in fundamental unit of apparent viscosity (Pa·s), in real sense it represents the values of apparent viscosity of the chosen system with proper conversion factors. The trends are the same for the other compositions and hence not shown.

Though the mixes are acting as Bingham fluids, when analysed as a function of time at constant impeller speed, the mixes displayed thixotropy (Fig. 7). Thixotropy is defined as the decrease of viscosity (softening) with time at a constant shear rate [10].

In Fig. 7, CM-1 (a) and CM-1 (b) represents the equivalent apparent viscosity calculated for the higher (1.10 rev/s) and lower speed (0.48 rev/s), respectively (in the reverse cycle) in each 5-min cycle for composition CM-1. The same nomenclature is valid for CM-2, CM-3 and CM-4. The equivalent apparent viscosity is decreasing with time at constant shear rate in all cases but with different rates.

The method of evaluation of *G* and *H* values using the test results for mix CM-1 at 0 min. is shown in Fig. 8. The intercept *G* and the slope *H* are being obtained by a linear regression analysis using six points in the reverse cycle (i.e. from high speed to low speed) as they represent the behaviour of the homogenized castable mix. The calculated values of *G* and *H* are shown in Table 2.

The *G* value is higher for CM-1 followed by CM-2, CM-3 and CM-4 respectively (Table 2). G decreases drastically for the initial 20 minutes and remains constant during further testing period. In case of CM-1, around 40% reductions are observed by 20 minutes while only 28% for other three compositions. Although the value of G can be drastically changed by choosing the proper type and amount of dispersant, only the ratios C/M and W/M have been considered here, as defined in Table 1. The relationships between the ratios C/M, W/M and *G* can be understood by comparing Tables 1 and 2. It is observed that *G* is decreasing as the ratios increase, except for C/M ratio of 1.56 (CM-4). In CM-4, the maximum particle size is 8 mm rather than 5 mm and for this mix segregation has been observed. The reason for segregation is that C/M ratio for CM-4 is less than for CM-2, with same *q* of 0.26. The reason for decrease of *G* with ratios is that more addition of water with decreasing fine matrix with same *q* reduces the force required for yielding the castable system. It is also observed that the *G* decreases with testing time, which is due to the additional mixing. Though the test was conducted to study the rheology, it gave additional mixing energy to the castable system as discussed earlier. The castable system with additional energy requires only less force to yield. Each *G* value reaches a constant value after 20 min, indicating that system has attained saturation stage in mixing and *G* cannot be further reduced. The 40% reduction of *G* in case of CM-1,

Table 2
G and *H* values for all compositions

Time, min	Flow resistance *G*, Nm				Torque viscosity *H*, Nm.s			
	CM-1	CM-2	CM-3	CM-4	CM-1	CM-2	CM-3	CM-4
0	2.28	1.86	1.5	1.18	9.9	8.65	5.8	6.58
5	2.06	1.65	1.37	1.1	9.44	8.47	5.67	6.52
10	1.69	1.62	1.34	0.93	9.33	8.47	5.56	6.54
15	1.65	1.61	1.28	0.9	8.68	8.02	5.53	6.36
20	1.49	1.52	1.19	0.92	8.47	7.78	5.45	6.13
25	1.46	1.46	1.17	0.85	8.07	7.58	5.27	6.11
30	1.4	1.4	1.07	0.88	7.8	7.43	5.26	5.84
35	1.42	1.41	1.16	0.89	7.7	7.41	5.24	6.05
40	1.45	1.43	1.2	0.89	7.61	7.4	5.22	6.14
45	1.44	1.33	1.16	0.92	7.38	7.34	5.11	5.95

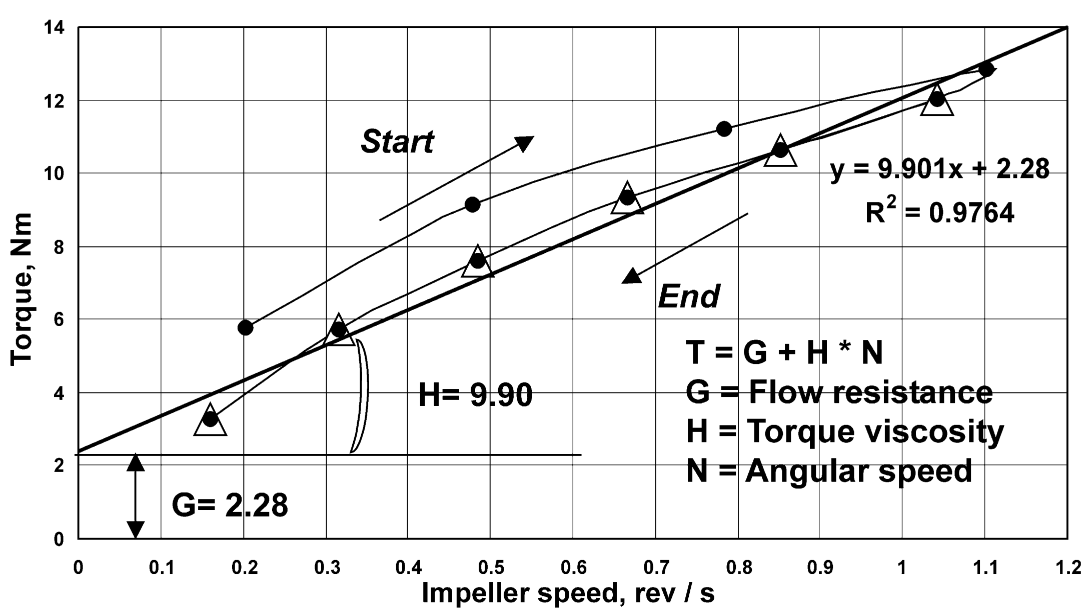


Fig. 8. Calculation method of *G* and *H* for CM-1 at 0 min.

which is due to the lowest C/M ratio, means that the castable has got more fines in the system and the additional mixing is drastically favoured when compare to other compositions.

H is the highest for CM-1 followed by CM-2, CM-4 and CM-3, respectively (Table 2). The trends of *H* and *G* values are different for composition CM-1 and CM-2. H decreases by 25 and 15% from the initial value in the first 20 min, and remains constant further on. In case of CM-3 and CM-4, the *H* values are almost constant with time and are around 50% lesser than for CM-1 and CM-2. This has a direct impact on the design of castables in practice. *H* has to be at optimum value to maintain the coherence of the castable system at given C/M and W/M ratios. It is observed that above the C/M ratio of 1.44 there has been a drastic reduction in torque viscosity, *H*. This means that an optimum amount of fine particles and sufficient viscosity are required to avoid segregation by maintaining coherence, integrity and stability of the castable mix.

Both *H* and *G* are decreasing with increasing *q* value but the rate of decrease is more for H (Table 2). This indicates the variation of bauxite fines in the matrix has influenced more the torque or apparent viscosity than the flow resistance or yield stress. In the present study, the type and amount of dispersant have been fixed and hence no drastic rate of change of *G* has been observed.

3.3. Optimization of rheology

Normally, a castable with good flowability is considered as pumpable and this is not necessarily so. Though castables are considered as a simple mixture of aggregates, fines and binders they are complex in terms of rheological behaviour. A test of flowability alone does not describe complete by such a behaviour. In this paper, CM-1, CM-2, CM-3 and CM-4 show the similar self-flowability with 7 wt.% water (Figs. 3 and 4), but all of them are not good pumpable castables. For good pumpability, a castable must possess optimum combination of torque viscosity and flow resistance in order to flow well and to avoid blockage and segregation. The relationship between G and H is shown in Fig. 9. The composition CM-1 is having good flow resistance (no segregation) but very high torque viscosity. CM-2 provides almost similar flow resistance but lesser torque viscosity than CM-1. Between CM-3 and CM-4, values of *G* are higher and *H* lower, but both CM-3 and CM-4 have been found to segregate (Fig. 10). This indicates, that the values of *G* and *H* should above a critical value to avoid segregation, hence composition CM-2 appears to be the optimum combination

3.4. Properties and appearance

The CMOR and AP of all compositions are almost similar, which are around 7–9 MPa and 17.5–18.5% with 7 wt.% water addition. The appearance of castables at different time for different composition is shown in Fig. 10 (a)–(h). The appearance of the fresh (0 min) castable for compositions CM-1 and CM-2 are very good, whereas dewatering and bleeding has been observed for compositions CM-3 and CM-4. After 45 min of mixing, some bubbles appear on the surface of castable compositions CM-1 and CM-2. Total segregation is observed with composition CM-3 and CM-4.

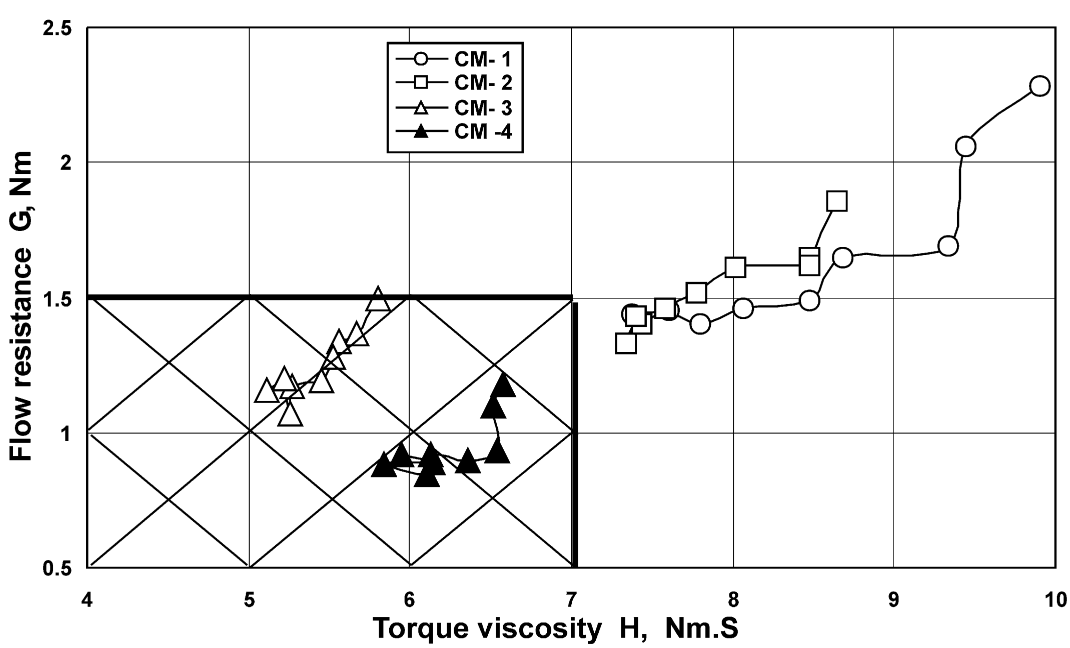
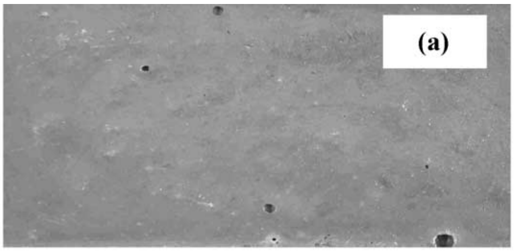
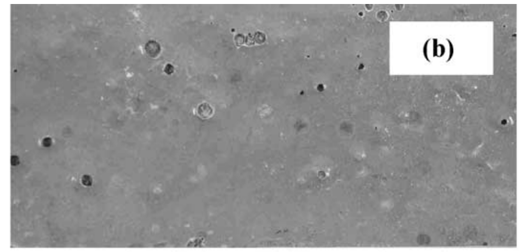


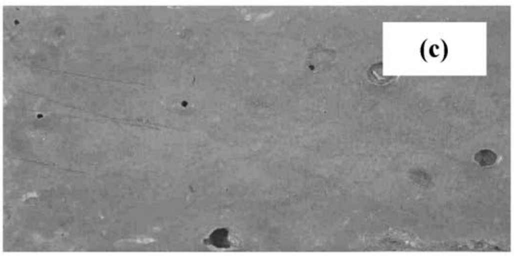
Fig. 9. Flow resistance vs. torque viscosity.



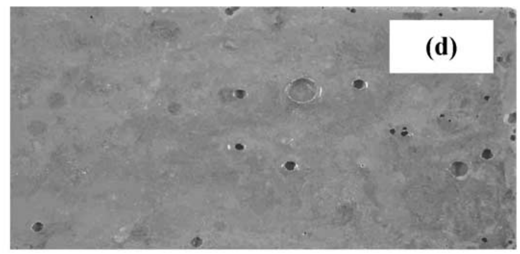
CM-1 surface cast immediately after mixing (0 minute).



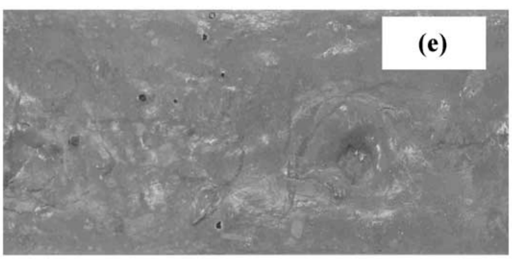
CM-1 surface with degassed bubbles cast after 45 minutes.



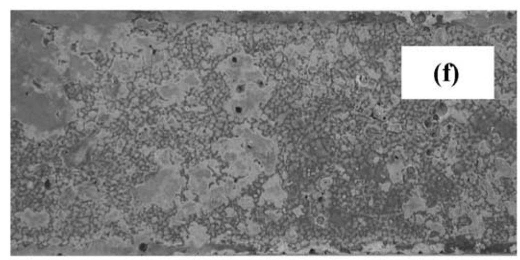
CM-2 surface cast immediately after mixing (0 minute).



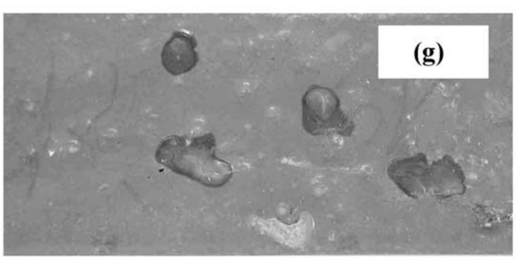
CM-2 surface with degassed bubbles cast after 45 minutes.



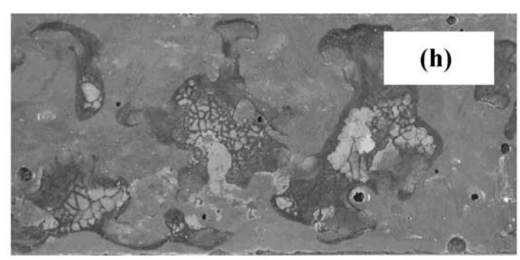
CM-3 surface with slight segregation cast immediately after mixing (0 minute).



CM-3 surface with serious segregation cast after 45 minutes.



CM-4 surface with slight dewatering and bleeding cast immediately after mixing (0 minute).



Sample CM-4 surface with serious dewatering and bleeding cast after 45 minutes.

Fig. 10. (a)–(h) Appearance of cast samples CM-1, CM-2, CM-3 and CM-4 cast after 0 and 45 min of testing.

4. Conclusions

The rheological behaviour of bauxite-based low-cement castable has been studied using IBB Rheometer V1.0. The conclusions based on this investigation are:

1. The rheometer has been found to be an effective tool to measure the rheology of castable mix directly. Two important properties torque viscosity (*H*) and flow resistance (*G*) have been evaluated using this rheometer. The mixes have shown Bingham behaviour for single cycle of rheometer test and thixotropy has been observed when analyzed with testing time at constant impeller speed.
2. Though self-flowability is found to be same for all compositions at water amount of 7 wt.%, they have shown different flow resistance and torque viscosity. This confirms that only flow measurement is not sufficient to define the castable behaviour.
3. *G* and *H* have got direct correlation with Andreasen modulus, D_{max}, C/M ratio and W/M ratio under the present set of conditions, the maximum particle size should not exceed 8 mm (with *q* of 0.26 and W/M ratio of 0.17) for achieving good *G* and *H* values.
4. The composition CM-2 has been found to have optimum values of *H* and *G* under the given conditions.

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