

Chapter-4

(Synthesis of high alumina cements by novel co-melt precursors and their implementation as castables with some micro fine additives)

Synthesis of high alumina cements by novel co-melt precursors and their implementation as castables with some micro fine additives

4.1 Introduction

High alumina cement is being produced commercially by fusing or sintering a mixture of argillaceous and calcareous materials above 1500°C and subsequent grinding to obtain a fine powder. Enormous quantities of air pollutants are emitted from cement production including SO₂, NO_x, CO, PM and result in significant regional and global environmental problems. To get rid of such environmental concerns, a novel process is required which may not only bring down the cost of production but can also fulfill the pollution norms.

The main use of HAC is in applications where casting with excellent performance at temperatures even above 1700°C is possible. HACs with 70-80 wt% Al₂O₃ can be used to produce castables with a temperature resistance ranging from 1800°C to 1900°C (Klaus et al., 2013). These materials are also used for many special applications in the construction works related to civil engineering because of their ability to gain strength rapidly and to withstand aggressive environments and high temperature (Midgley, 1990; Scrivener et al., 1999). The knowledge of their mechanical behavior at a young age becomes essential to forecast their performance in service. To describe hydration of cement paste at the early age (from a few minutes to a few hours after mixing), different approaches have been developed. Conductivity and pH measurements, usually carried out on dilute systems, have been applied to predict the dissolution and precipitation processes (Capmas et al., 1990; Jiang et al., 1995).

Other important factor to decide the properties of cements is its fineness. Nano ceramic materials (<100 nm grain size) are still in the early stages of development. They depict many processing and property advantages over their conventional coarse grained alternatives. In recent years, there has been increasing interest in the

synthesis of nano crystalline metal oxides (Myhre, 1994; Das et al., 1998). It is well-known fact that the morphology and particle size distribution are important factors that influence physico-chemical properties of materials. Presently consistent and low cost methods of nano material preparation have limited their industrial uses. Therefore this factor was also included in the present research work.

One approach used by researchers to improve the quality of refractory castable is through decreasing the cement content, i.e., 4%–8% in LCC or 1%–3% in ultra-low cement castables (ULCC) or <1% in zero cement castables (ZCC). Decreasing the CaO content in these castables eliminates the disadvantages of the conventional castables (10%–20%), which inherently included high porosity lower strength and slow curing and drying processes. These influences were due to their higher amount of water requirement, which was a major drawback resulting in their loss of strength during the dehydration process and subsequently reaching at 500°C–1000°C (Myhre, 1994). Moreover, the presence of a high content of CaO in conventional castables decreases the refractory properties due to the formation of low melting compounds at high temperatures. These disadvantages have been eliminated in the new generation of high technology castables, which enables their use in a wide variety of applications (Zawrah and Khalil, 2001). These castables develop very high performance during and after heating. They are used when high thermal shock resistance or resistance against abrasion and corrosion by slag or melted metals are required (Capmas et al., 1990, Myhre, 1994). These castables require good control during installation, i.e., rheology and hardening kinetics, which are influenced not only by the intrinsic reactivity of the calcium aluminate cement but also by the interactions between fillers and calcium aluminate cement (Serry et al., 2002). The attraction of low cement and ULCC for refractory applications is due to their high strength, thermal shock resistance, corrosion resistance and their economic effect derived from the use of natural bauxite resources. They have already been successfully employed in molten iron torpedo cars, blast-furnace troughs and incinerator linings and may yet acquire wider applications in high-temperature industries. Recently, a group of additives appeared in the refractory

industry that triggered new prospects for castable producers. The additives comprise ultra-fine particles, for example silica, alumina, mullite, spinel and carborundum. In previous investigations (Maczura et al., 1985), lower amount of cement in conjunction with fine grain-sized materials having high surface area and dispersing aids were used. Those materials were silica fume, Al_2O_3 , Cr_2O_3 , ZrO_2 , TiO_2 , SiC, clay mineral and carbon. It was concluded that the presence of such materials promote refractory properties, such as volume stability and well balanced physical and hot mechanical properties. The presence of zircon with fine alumina as a filler tends to form mullite/ ZrO_2 composite after firing. The formation of mullite and zirconia is of vital importance as these bonding phases exhibit high refractoriness, low creep rate, low thermal expansion, good chemical, thermal stability, toughness and strength (Piviniski, 1998).

Nowadays, low and ULCC containing SiC have gained wide acceptance as materials for hot metal and slag runners. Silicon carbide is being introduced to enhance the thermo mechanical properties of the composite under operating conditions. Some authors studied high temperature strength and thermal shock of ultra and LCC in relation to their microstructure feature. These castables have been composed of SiC-containing bauxite. They found that castable specimens had significantly improved in HMOR studied at 1400°C (Kronert and Schumacher, 1989). However, the problem of SiC oxidation limits its presence in refractory castables. The aim of the present part of research work was to synthesize a novel co-melt precursor HAC and their implementation in bauxite containing castable to investigate the fineness of cements and their physico-chemical behaviour along with sintering effect of SiC, silica fume, and reactive alumina in their castables.

4.2 Experimental procedure

4.2.1 Material characterization

The starting raw materials were aluminum nitrate, calcium nitrate, silica fumes, silicon carbide powder and reactive alumina of A.R. grade. These materials were supplied by Loba Chemie Pvt. Ltd., Mumbai India.

4.2.2 HAC powder preparation

Hydrated aluminum nitrate $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and hydrated calcium nitrate $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ were weighed and mixed on a laboratory scale to give the equivalent molar ratio of 7:3 and 8:2 $\text{Al}_2\text{O}_3/\text{CaO}$ denoted as HAC70 and HAC80, respectively. The mixtures were heated in a porcelain dish on a hot plate at about 250°C until it melted completely and then it was air quenched. The resulting solids were heated at 500°C for 3 h. Samples of the HAC powders obtained were finally calcined at 800°C , 900°C and 1000°C in a platinum dish using a SiC muffle furnace for soaking period of 10 min. After soaking, the powders were quenched and stored in a desiccator.

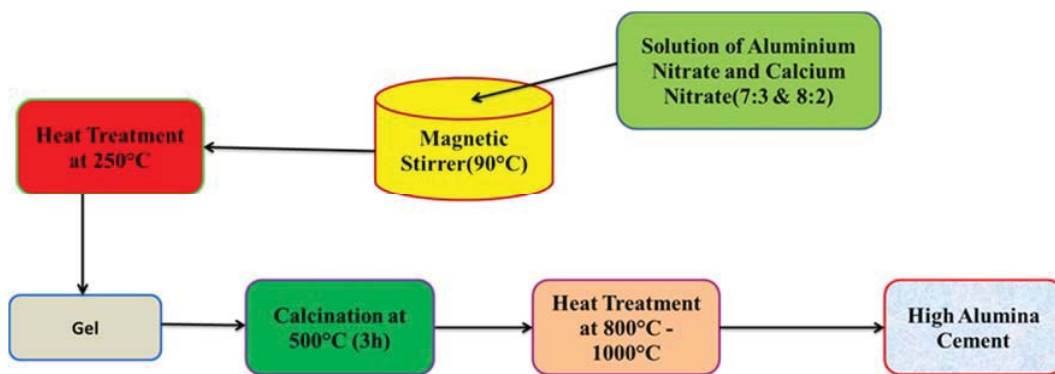


Figure 4.1: Flow chart of HAC powder preparation.

4.2.3 Consistency of cement

Consistency is a measure of plasticity of a cement paste. It refers to the degree of wetness exhibited by a freshly mixed concrete, mortar, or neat cement ground whose workability is considered acceptable for the purpose at hand. It is measured as the amount of water required as to a particular wt% of dry cement which permits the Vicat's plunger of 10 mm diameter to penetrate to a point 5-7 mm from the bottom of Vicat's mold with gauging time 3-5 min. Calcined HAC powder having 70% alumina and 80% alumina content samples were tested for their consistency by Vicat's apparatus.

4.2.4 Setting time test

Calcined powders were tested for initial and final setting by Vicat's apparatus according to ASTM C403.

4.2.4.1 Initial setting of high alumina cement

This early period of the hydration and strengthening of cement is referred to as "initial setting" of cement. The initial setting time was measured by taking 50 gm of HAC mixed with the percentage of water required for normal consistency. In Vicat's apparatus a needle is allowed to penetrate through the cement block prepared. In the initial stage, a thicker needle is allowed to pierce through the test block. This procedure is repeated until the paste starts losing its plasticity and the penetration is limited only to 5-7 mm depth. This duration of setting is counted and termed as initial setting time.

4.2.4.2 Final setting of high alumina cement

The cement will be considered finally set when upon lowering the attachment, gently cover the surface of the test block and the thinner needle makes an impression. The duration of this process is considered as final setting time.

4.2.5 Castable formulation

Low cement refractory castables were prepared using approximately 5 wt% HAC cements and refractory grade high alumina containing bauxite with superfine additives. The calcined Chinese bauxite that was used in castable formulation in this study contained approximately 88.60%, 4.78%, 1.58%, 4.0%, 0.26%, 0.08% and 0.70% by weight Al_2O_3 , SiO_2 , Fe_2O_3 , TiO_2 , CaO and Na_2O , respectively. In the bauxite matrix, small additions of reactive alumina, micro-fine silica and SiC powder was made. The formulation of these castables are summarized in Tables 4.1 and 4.2, which show the detailed composition along with their specific names. The effect of these additions on thermo-mechanical and structural properties of castables was also observed. In the first step for cement castable formulation, calcined bauxite was oven dried, crushed and grounded for grading into different sizes in a planetary ball mill. The jar and grinding media were of titanium-coated stainless steel material. At one time 250g of calcined bauxite material was taken in a jar and grounded in a high energy planetary ball mill for 45 min at 600 rpm. Similarly, it was processed to complete the grinding of complete material. The ground material was then kept in various selected sieves and set up on the motorized vibro sieving equipment for grading. After separation of different graded bauxite, experiments were performed further. Figure 4.3 is the pictorial representation of particle size analysis done with the help of standard Tyler mesh. The particle size distribution of the fine fraction is generally a representation of the flow characteristics. The particle size distribution has an important role in the properties of refractory castable. Incorrect particle size distribution may cause militancy or the excess water requirement by the castables. The trials of aggregate proportions were taken in a 1000 cm^3 flask filled up to 250 cm^3 and vibrated for 30 sec. Packing density calculations were carried out for each trial. Aggregates having highest packing densities were chosen for further analysis. In the next step, batches were prepared by taking different grades of materials and additives in the proper proportion and are summarized in Tables 4.1 and 4.2. The materials were dry mixed in a plastic container for 10 min by spatula and were then used in sample preparation. Generally, ultra and LCCs require less than 5 wt% of

water to achieve the desired rheology. Therefore, water was added in two steps. The casting was done by initial adding the two-third proportion of water at a time. Then, one-third of water was added slowly to get a homogenous mixing. The wet mixing was performed for 5min to achieve proper flow. Immediately after doing wet mixing, the castable mix was filled into a rectangular bar shape mold (152 mm × 25 mm × 25 mm), made of hard steel. The mold was placed on the vibrating table, filled with the wet mixed castable and the mixes were vibrated for 10 min. For each composition, several samples were prepared for laboratory testing. The samples were cured in a moisture-saturated environment (95% RH) in a humidity chamber at room temperature for different time periods. For firing the samples, they were first oven dried at 110°C for 24 h. The test samples were fired at 1300°C-1550°C with a variation of ±5°C in an electric SiC heating element furnace with maximum soaking time of 3h. The cured samples as well as the fired samples were tested for bulk density, apparent porosity, HMOR, CMOR and cold crushing strength (CCS). These samples were also analyzed by XRD for phases present and by FE-SEM for their morphological behavior.

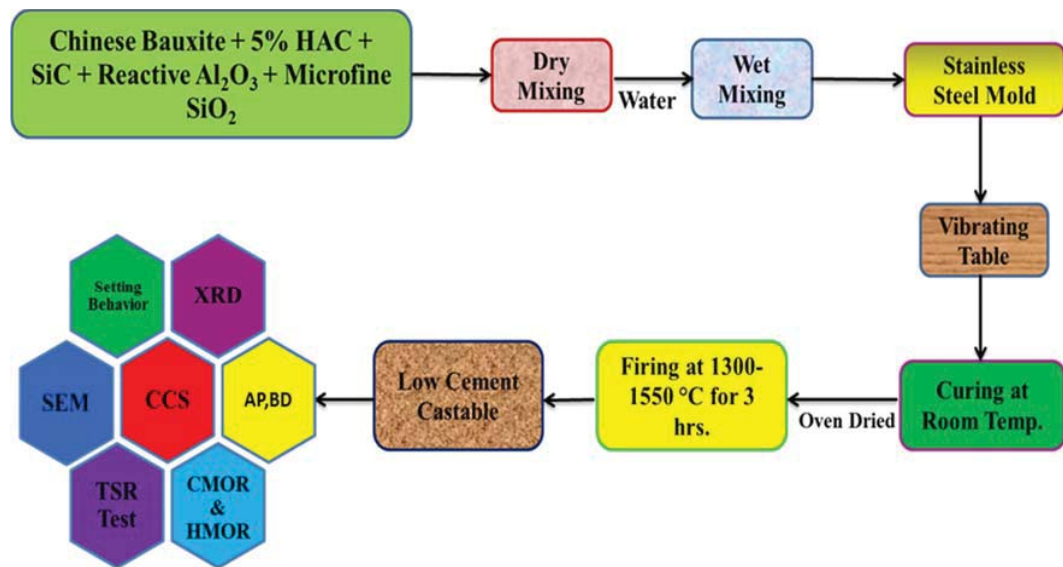


Figure 4.2: Low cement castable preparation.

Table 4.1: Batch composition with HAC70.

Sample	HAC70 (Wt%)	Chinese Bauxite (Wt%)	SiC (Wt%)	Reactive Al₂O₃ (Wt%)	Microfine SiO₂ (Wt%)
R1	5	80	5	5	5
R2	5	80	5	6	4
R3	5	80	5	7	3
R4	5	80	5	8	2
R5	5	80	5	9	1
R6	5	80	5	10	0

Table 4.2: Batch composition with HAC80.

Sample	HAC80 (Wt%)	Chinese Bauxite (Wt%)	SiC (Wt%)	Reactive Al₂O₃ (Wt%)	Microfine SiO₂ (Wt%)
S1	5	80	5	5	5
S2	5	80	5	6	4
S3	5	80	5	7	3
S4	5	80	5	8	2
S5	5	80	5	9	1
S6	5	80	5	10	0

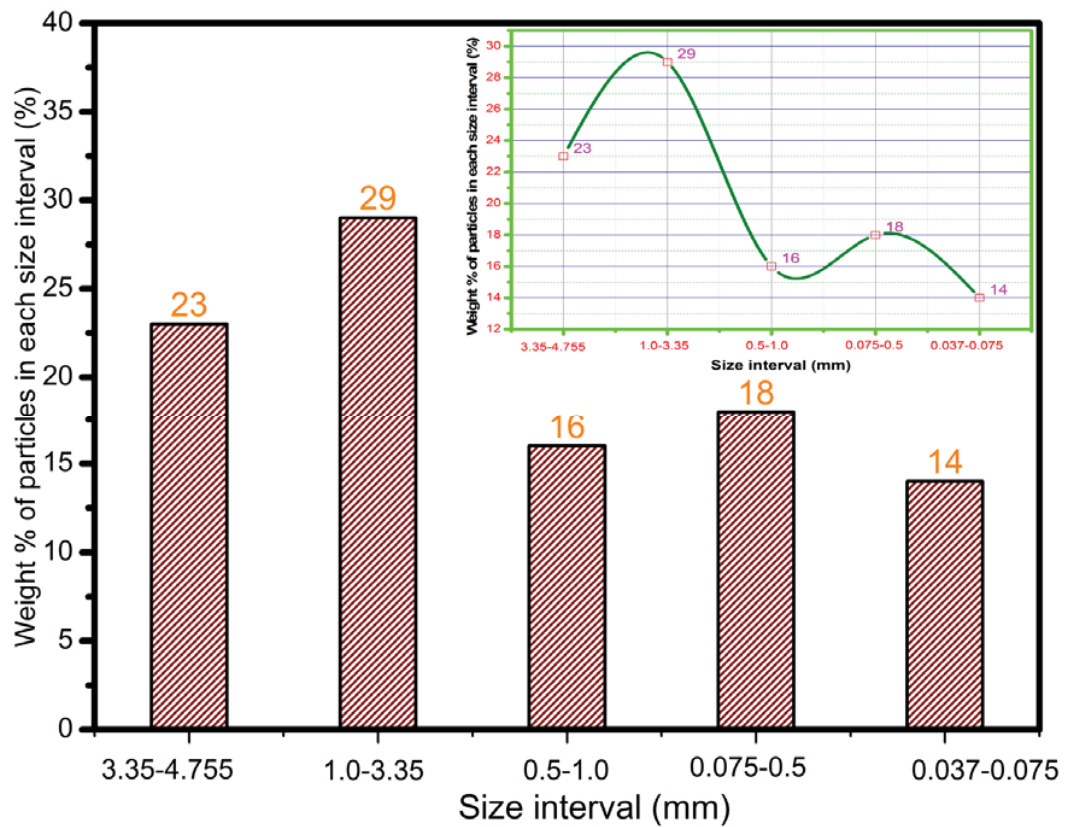


Figure 4.3: Bauxite particle size distribution.

4.2.6 Phase identification

X-ray diffraction patterns were observed using a Rigaku portable XRD machine (Rigaku, Tokyo, Japan). Calcined powdered samples were analyzed by XRD for phases present in the fired sample. Phase identification analysis was carried out by comparing the respective powder XRD patterns with the standard database stated by JCPDF. Detailed analysis of the phase formation and changes over the temperature range and variation in compositions were carried out.

4.2.7 Cold crushing strength of HAC and LCC formulated

The CCS is the capacity of a material to withstand axially directed pushing forces. By definition, the compressive strength of a material is that value of uniaxial

compressive strength reached when the material fails completely. CCS of cement was measured as the compressive strength of a 50 mm cement cube made of pure HAC (without any aggregates). These samples were tested for compressive strength after 6h, 24h and 48 h, respectively, according to (ASTM C1194-03).

4.2.8 Cold and hot modulus of rupture (CMOR and HMOR) of LCC formulated

Cold and hot modulus of rupture (CMOR and HMOR) measurements were carried out under three-point bending tests (ASTM C133-97 for CMOR and ASTM C583-10 for HMOR) using 152mm × 25mm × 25mm samples. CMOR and HMOR were calculated using the following formula:

$$\text{MOR} = \frac{3PL}{2db^2}$$

where,

MOR = modulus of rupture (MPa),

P = maximum applied at rupture (N),

L = span between supports (mm),

b = breadth or width of specimen (mm),

d = depth of specimen (mm).

CMOR tests were conducted at room temperature using Universal Materials Test Equipment (Model 810, MTS System, Eden Prairie, MN, USA) for samples pre-fired at 1300°C, 1350°C, 1400°C, 1450°C, 1500°C and 1550°C for 3h. HMOR measurements were made at 1400°C, 1500°C and 1600°C using Netzsch 414/3 HMOR Equipment (Netzsch, Selb, Germany) for samples pre-fired at 1550°C for 3h, cooled at room temperature and then reheated for testing.

4.3 Results and discussion

4.3.1 Evolution of phases by X-Ray diffraction

Figures 4.4 and 4.5 are the XRD patterns of samples, calcined in the range of 800°C-1000°C. Cements having 70% and 80% alumina is termed as HAC70 and HAC80, respectively. When calcined below 900°C, these powders proved to have X-ray pseudo-amorphous pattern, where initial formation of calcium-aluminate phases may be envisaged. Pure crystalline cementing phases only begin to form at the temperatures above 900°C in both compositions. A major improvement in crystalline behavior is evident when both cement samples were fired at 1000°C. Prime phases investigated in both compositions included CA, CA₂ and C₁₂A₇, which are readily formed and thermodynamically most stable compounds in the CaO-Al₂O₃ binary system (Richard et al., 1995). These peaks were identified by standard JCPDS cards numbered 41-0358, 34-0440, and 09-0413 for their corresponding peaks of hexagonal CA, monoclinic CA₂ and cubic C₁₂A₇, respectively. In the conventional preparation route by high-temperature solid-state synthesis, the batch usually contains CaO-rich phases and unreacted Al₂O₃ before the appearance of desired product phase. The formation sequence of phases in these mixtures is always from calcia-rich phases to the alumina rich phase which could be accounted for the formation of C₁₂A₇ as soaking period was very low as 10 min and thus saving energy.

Presence of broad peaks in XRD patterns of calcined cement powders show that particle size is small. Crystallite size 'd' of calcined powder was calculated from X-ray line broadening analysis using Scherrer's formula:

$$d = \frac{0.9\lambda}{\beta \cos\theta}$$

where, β is the full width at half maximum intensity of a Bragg reflection excluding instrumental broadening, λ is the wavelength of the X-ray radiation and θ is the Bragg angle. β is taken for the strongest Bragg's peak corresponding to 2θ . Both samples have crystallite size in the range 20-35 nm.

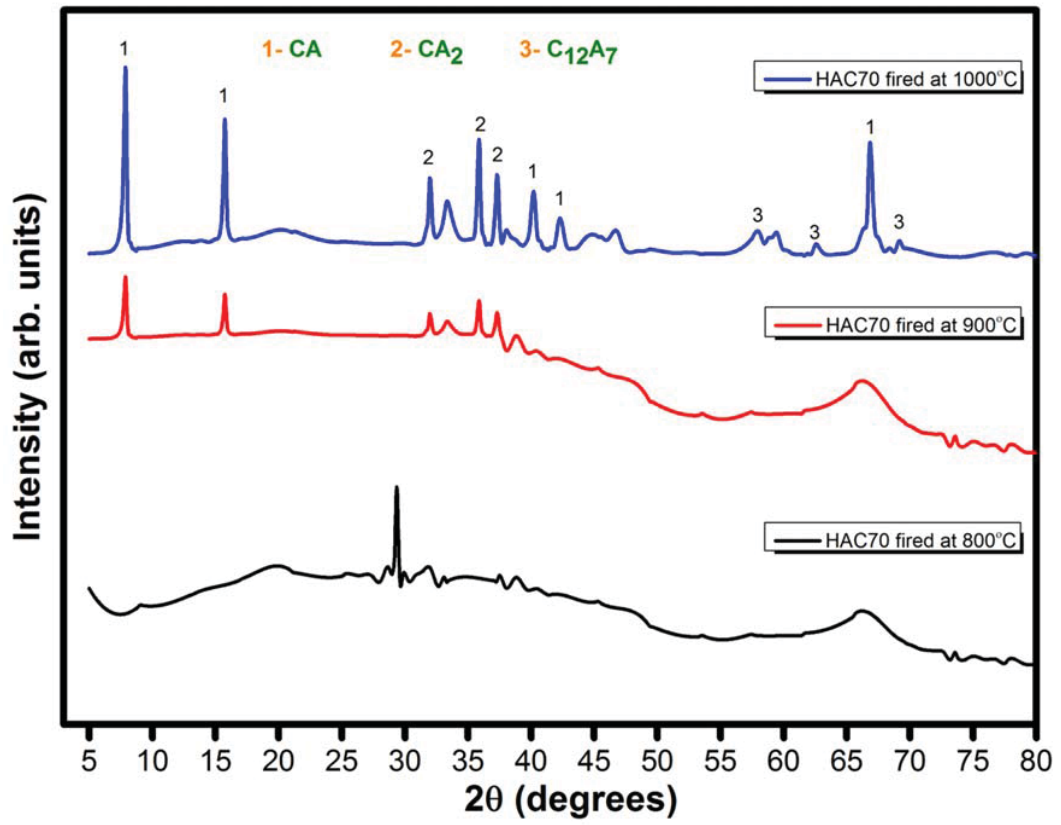


Figure 4.4: XRD plot of HAC70.

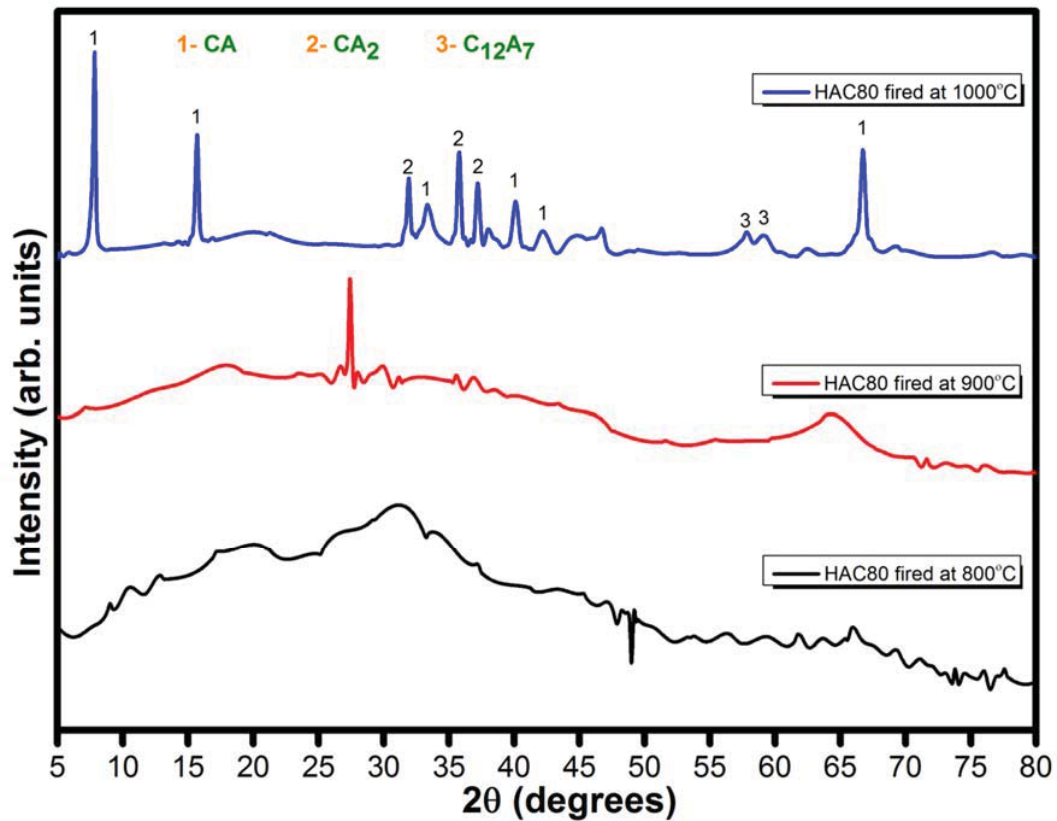


Figure 4.5: XRD plot of HAC80.

4.3.2 Setting behavior of HAC

Figure 4.6 shows the setting behavior of HAC. The prepared HAC powders were mixed with water (0.85 P). At room temperature, 70% alumina containing cement gave an initial setting time of 16 min and final setting occurred in 45 min. Another composition of HAC having 80% alumina had an initial setting time of 18 min and a final setting time of 60 min. With increasing alumina content in HAC the setting time showed an increasing trend.

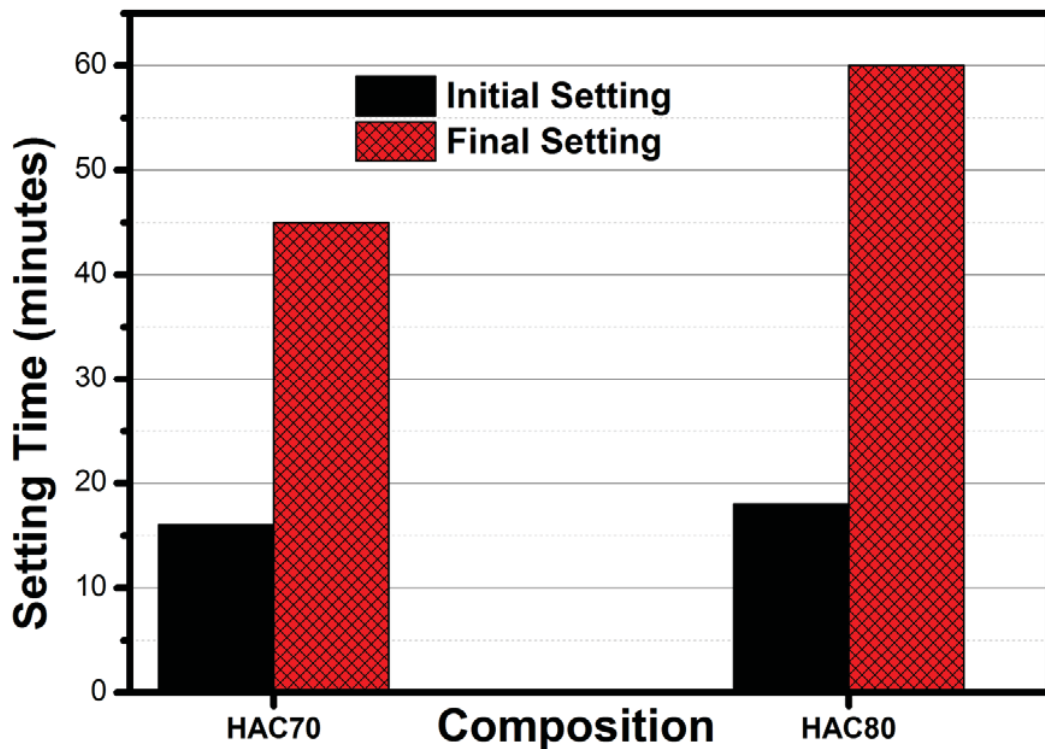


Figure 4.6: Setting time behavior of HAC70 and HAC80.

4.3.3 CCS of HAC fired at 1000°C

The pure (without any aggregates) HAC showed good structural strength as obtained from present experimental procedure. It can be seen in figure 4.7. These samples were tested after curing for 6h, 24h and 48 h, respectively. Sample blocks with inclusion of two commercialized cements of similar compositions were also made to have a better comparative analysis. The strength increased rapidly with curing time in all the compositions. Initially, in 6 h, the CCS value obtained for HAC70 is 10 MPa and for HAC80 is 14 MPa. After 48 h, it reached up to 65 and 70 MPa for HAC70 and HAC80, respectively. These values are better than the commercially available CA-14M-55 MPa and CA-25C-50 MPa (Almatis). Samples with prepared cements get the maximum strength at around 48h of curing and after this period, there is a very slight variation. It can also be seen that HAC80 samples have more compressive strength value than the HAC70 sample. The higher strength

of HAC80 is ascribed to the presence of larger amounts of CA and CA₂. It is well known that CA and C₁₂A₇ react significantly in the early ages of hydration and the hydration of C₁₂A₇ is very exothermic. So the formation of stable hydrates generally occurs sooner. Although CA₂ is known to react slowly with water in the early stages of hydration, its presence along with other phases results in an overall faster hydration rate as the heat of hydration resulting from the hydration of CA. This energy activates CA₂ and makes it react relatively faster with water than it would do alone, but not more than C₁₂A₇. CCS data of the sintered ceramic bodies exhibits high strength of both samples. This is due to the formation of the ceramic bond and absence of any impure phase (Singh and Ali, 1980; Singh and Mandal, 1982; Singh et al., 1990, Zawrah and Khalil, 2002).

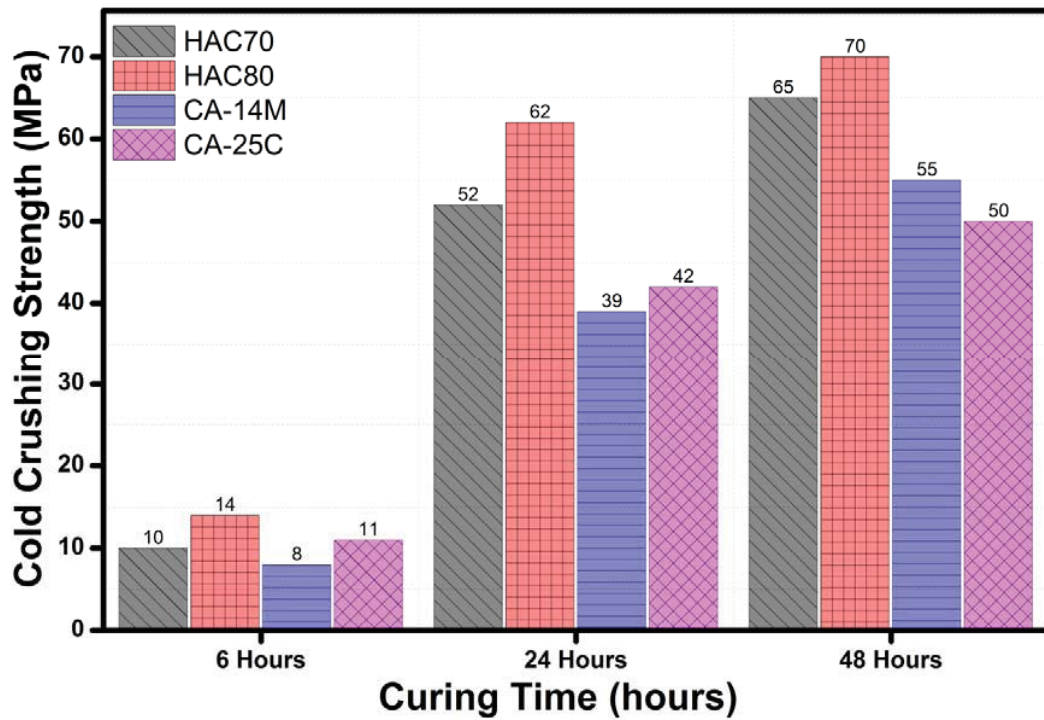


Figure 4.7: Cold crushing strength of HAC70 and HAC80.

4.3.4 FE-SEM of HAC

Figures 4.8 and 4.9 represent FE-SEM results of HAC70 and HAC80 cements, respectively. Networks of well-crystallized interlocking hexagonal plates of CA matrix of HAC70 and HAC80 fired at 1000°C can be seen. In both figures, a, b, c and d represent different magnifications of same sample. The monoclinic phases of CA₂ and cubic C₁₂A₇ can be observed in both figures as verified by XRD results also. High amount of CA and CA₂ is proposed to enhance the refractory properties. C₁₂A₇ phase is responsible for high bonding and quick hydration and small cubic crystals of this phase are evident in the most homogeneous distribution of CA phase matrix. With the EDS representation of both HAC70 and HAC80, which is a further confirmation of the exact elemental composition and it also a depiction of pure phases obtained.

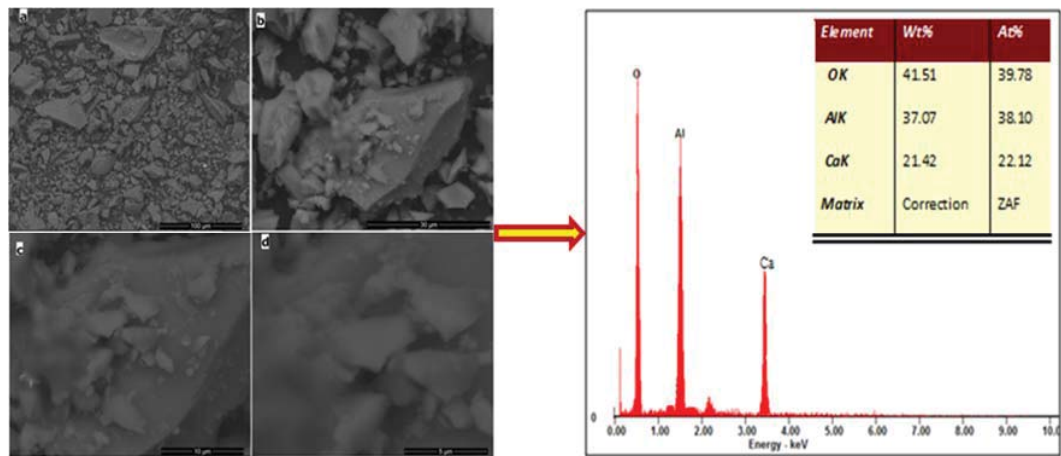


Figure 4.8: FE-SEM of HAC70 with EDS pattern.

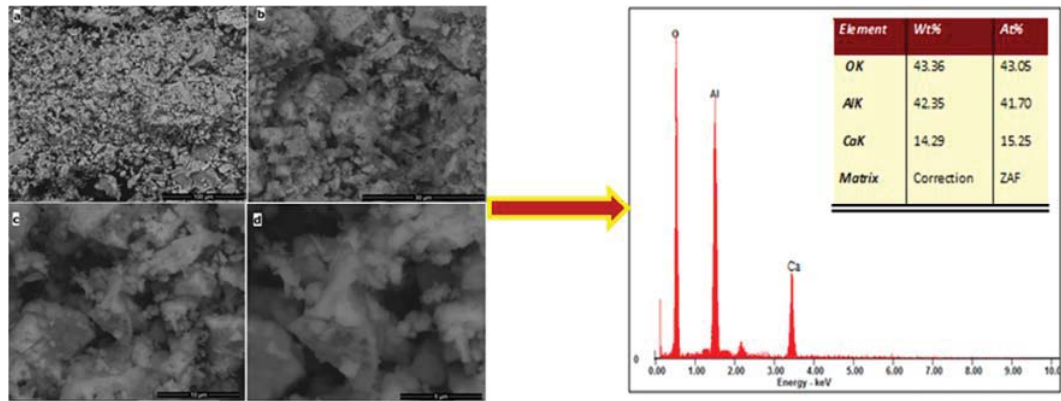


Figure 4.9: FE-SEM of HAC80 with EDS pattern.

4.3.5 XRD Patterns of castables fired at 1550°C for 3h

Figures 4.10 and 4.11 portray XRD patterns of castables fired at 1550°C for 3h. Here R1 to R6 and S1 to S6 indicate castables formulated with HAC70 and HAC80, respectively with varying composition. The corundum phase appeared as a major component due to the transformation of bauxite minerals to corundum. The second major phase was detected as mullite, which resulted due to the addition of microsilica and reactive alumina in all the castable samples. In both castable series, as we proceed from high to low batch number, the peak intensities of mullite increases. This is accounted by increase in micro fine silica content. Although SiC phase was added as a minor ingredients, but it fails to show its intensity peak in all the plots, so it could be extracted from the fact that at high temperatures its oxidation would have occurred forming silica and finally reacting with aluminum content to result in more mullite formation. These peaks were identified by standard JCPDS cards numbered 46-1212, 15-0776 for figure 4.10 and 46-1212, 06-0258 for figure 4.11. Minority peaks of silicon carbide in both figures were identified and matched with JCPDS card number 42-1091.

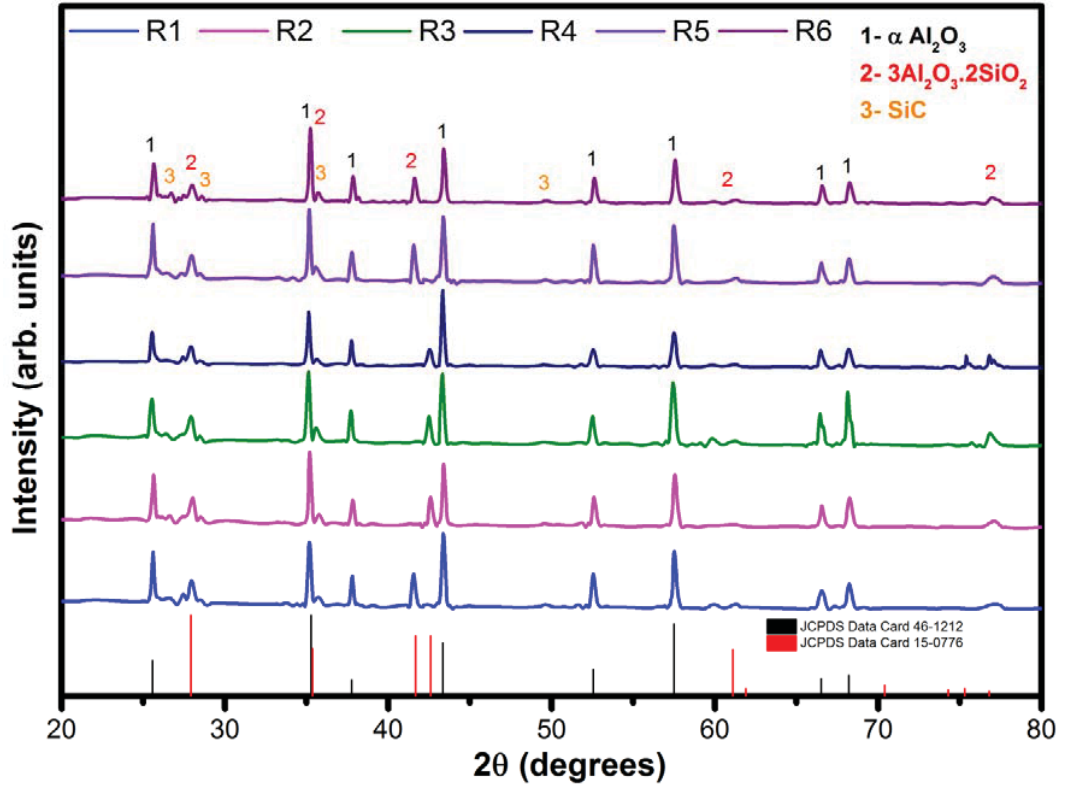


Figure 4.10: XRD plot of R series castables prepared with HAC70.

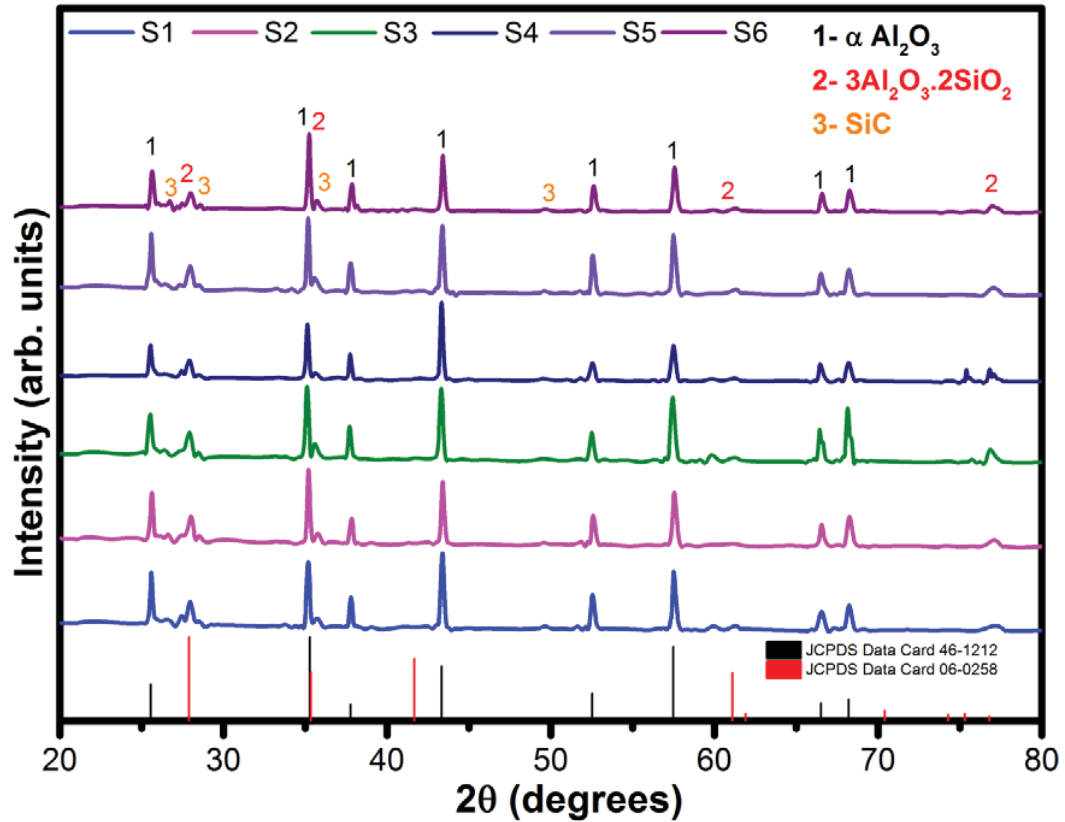


Figure 4.11: XRD plot of S series castables prepared with HAC80.

4.3.6 Bulk density and apparent porosity of prepared castables

Table 4.3 shows the values corresponding to bulk density, apparent porosity and thermal shock of R3 and S3 series castables sintered at varying temperature range. Only these series was selected as homologous to their other thermo mechanical superiority. In the castables prepared, highest bulk densities achieved are 2.91 and 2.93 g/cm³ for the compositions R3 and S3, respectively. This can be attributed to the use of higher percentages of alumina and micro-fine silica in the aggregate which resulted in most mullite solid solution formations. Apparent porosity was measured to be in the range of 8%-11% in all the castables prepared.

Table 4.3: Bulk density, apparent porosity and thermal shock cycles of prepared castables.

Test Method	Temperature (°C)						
	Sample Name	1300	1350	1400	1450	1500	1550
Bulk Density (gm/cc)	R3	2.60	2.67	2.72	2.80	2.81	2.91
	S3	2.69	2.71	2.76	2.81	2.83	2.93
Apparent Porosity (%)	R3	13.00	12.50	11.69	11.41	11.20	11.00
	S3	10.00	9.50	8.90	8.40	8.21	8.00
Thermal Shock Cycle	R3	16	15	15	14	15	13
	S3	15	16	15	14	14	14

4.3.7 Thermal-shock resistance tests of prepared castables

Table 4.3 displays the thermal shock cycles values evaluated for R3 and S3 series castables. This series was chosen in accordance with their superior thermo-mechanical properties. Thermal-shock resistance tests were run to analyze the influence of the mullite content and the characteristics on the castables thermal-mechanical properties. The tests consisted of 10 heating/cooling cycles (DT = 1200°C). Samples pre-fired at 1550°C completed one thermal cycle after a holding time of 20 min inside the furnace followed by another 20 min of cooling in air at room temperature. All the S and R series samples exhibited high thermal shock and spalling resistance completing 10 cycles while S3 and R3 attained 16 cycles.

4.3.8 CCS of different castable compositions sintered at 1300°C–1550°C for 3h

Prepared HAC was used for formulating LCC according to the Tables 4.1 and 4.2. Reactive Al₂O₃ and micro fine SiO₂ content have been varied, which according to the above mentioned plots resulted in more or less mullite solid solution. This reaction

also has a varying effect in the CCS of the castables formulated. Figure 4.12 depicts the castable series S and R. High values of CCS up to 295-375 MPa were achieved at the maximum sintering temperature of 1550°C for S series castables. Similarly, 280-350 MPa was achieved for R series castables. This can be predicted that rise in sintering temperature has a positive effect on the mechanical properties of both series of castables. Formation of mullite strengthens the structure at high temperature and solid solution helps in increasing structural strength. The CCS of the castables prepared with HAC70 and HAC80 were higher in comparison with the CCS of conventional bauxite containing LCC containing Secar-71 (160 MPa) and Secar-80 (200 MPa).

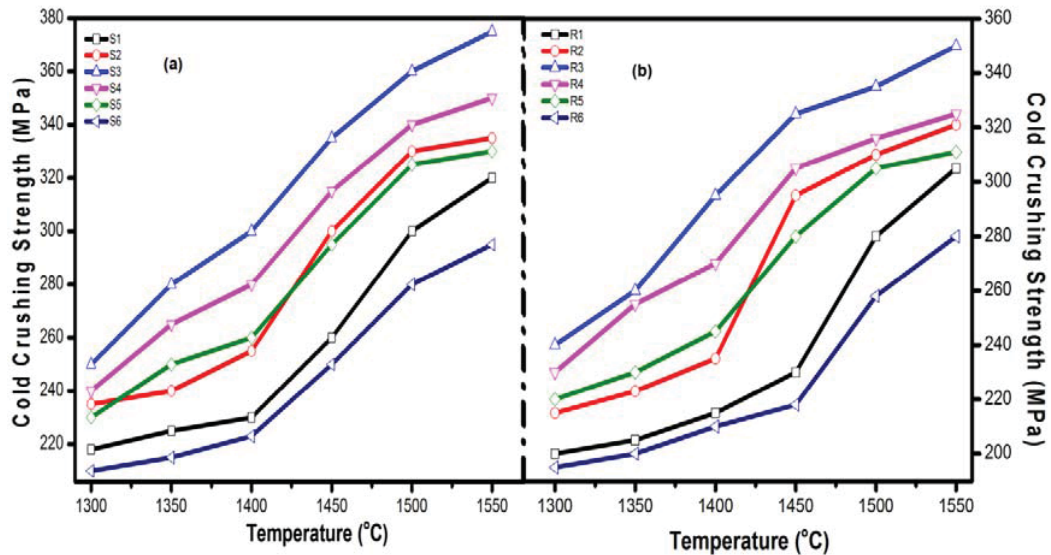


Figure 4.12: Cold crushing strength of castables.

4.3.9 CMOR and HMOR of different castables

Cold modulus of rupture strength (CMOR) of castables S and R series is shown in figure 4.13. It can be seen that CMOR of both series castables has increased with the increase in sintering temperature and was maximum for S3 in which maximum amount of mullite grains was formed. Presence of SiC could also be accounted for high mechanical properties. This was reflected in the strength of the whole castable

sample. Hot modulus of rupture (HMOR) of castables S and R series is shown in figures 4.14 and 4.15, respectively. Here, again S3 achieved the maximum strength at high temperature/load. The increase in HMOR values of few samples from 1400°C to 1500°C is due to the formation of mullite solid solutions. To conclude, we may say that the safe range for these types of castables at higher loads is lower than 1600°C. The impurity in the bauxite aggregate increases the liquid phase sintering and mechanical properties, but decreases the porosity.

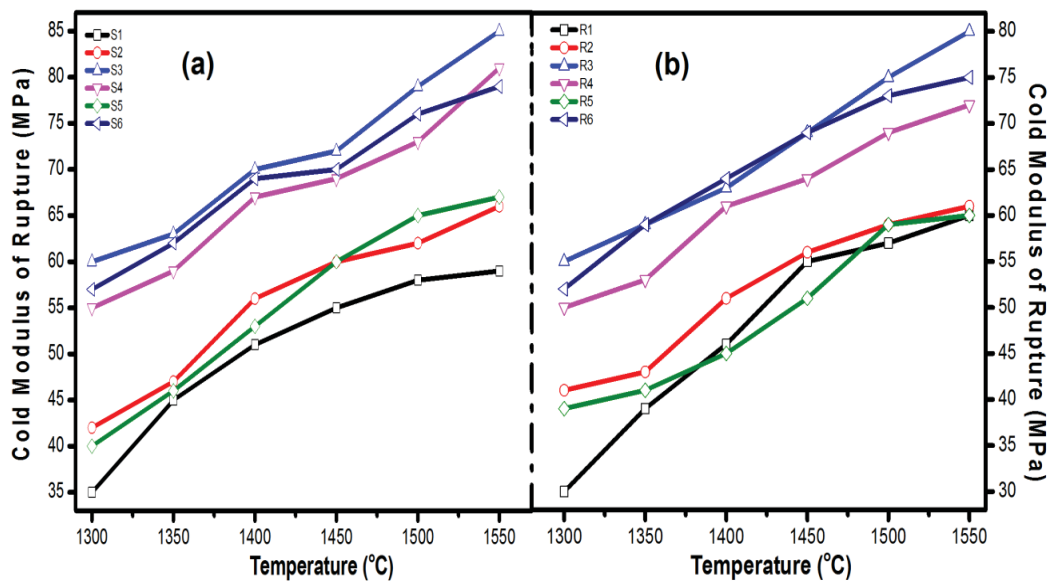


Figure 4.13: Cold modulus of rupture of castables.

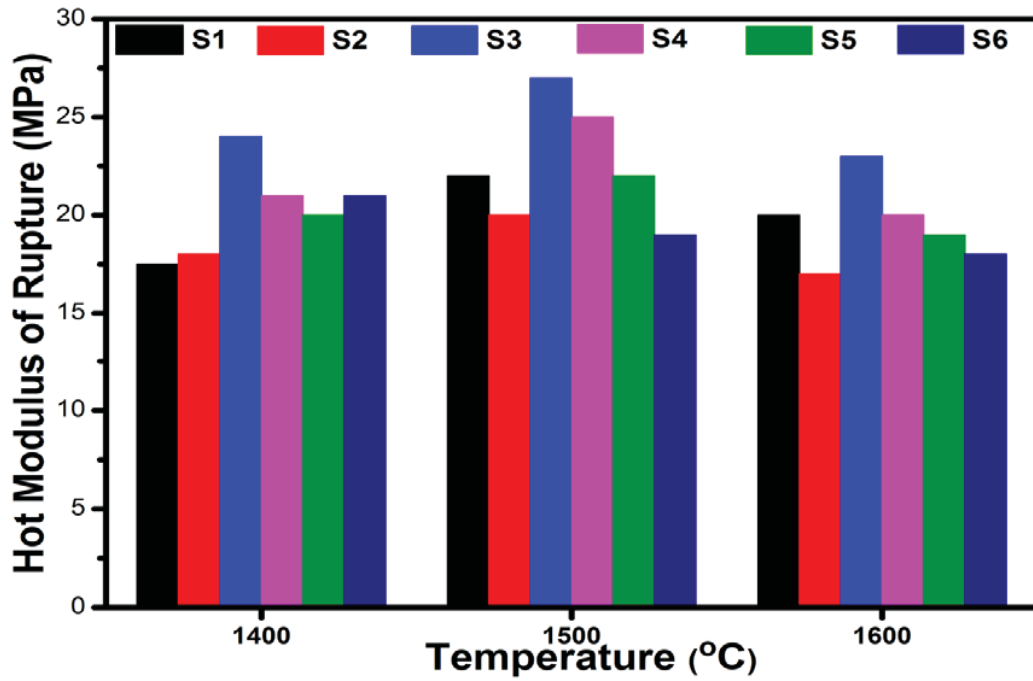


Figure 4.14: Hot modulus of rupture of castables prepared with HAC80.

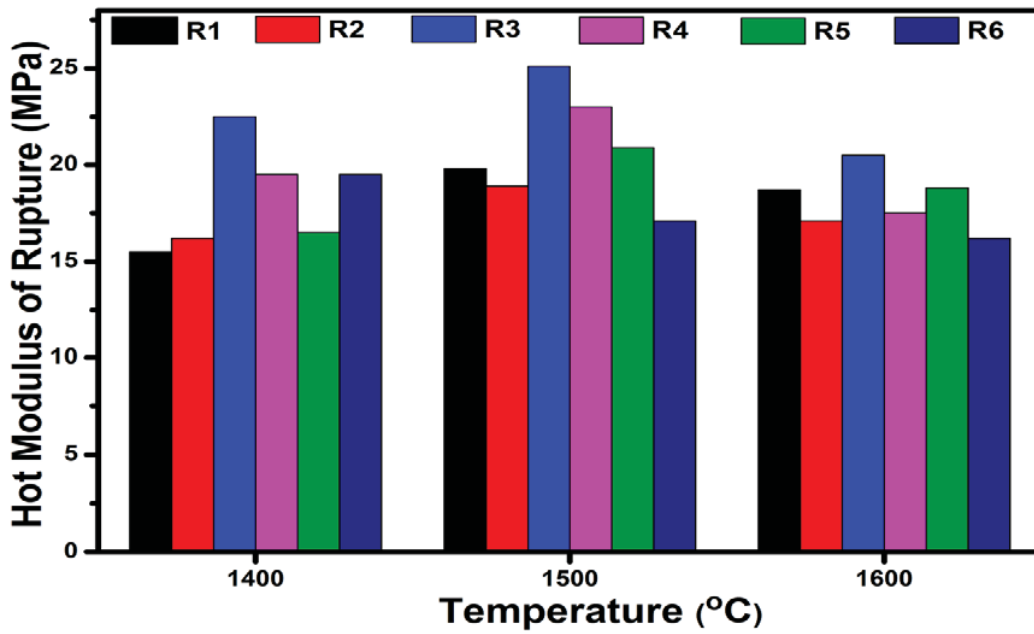


Figure 4.15: Hot modulus of rupture of castables prepared with HAC70.

4.3.10 FE-SEM of S3 castable

The microstructural evolution of castables fired at 1550°C was examined using field-emission scanning electron microscopy of representative regions of fractured specimens. Figure 4.16 depicts FE-SEM photo-micrographs of S3 castable after firing at 1550°C for 3 h, where a, b, c and d represent different magnifications. Only S3 sample was chosen for analysis through FE-SEM as it represented superior thermo-mechanical and physical properties than all other S and R series castables. The matrix includes CA and some of liquid phases. Photo-micrographs exhibit densely packed microstructure with an abundant orthorhombic mullite grain of comparable sizes, embedded in the trigonal corundum matrix. Some needle-shaped mullite beside alumina grains appeared in the microstructure. The presence of such in situ formed phase (mullite) had given interlocking nature of castable and reinforced the matrix confirming the high strength and refractory properties. On the other hand, the impurities that were present in Chinese bauxite may have formed a low melting glassy phases, which tend to decrease the refractory properties and their microstructure may appear as partially diffused structure in some regions. This property is more evident when we zoom into the microstructures while moving from a to d in each plot. The EDS pattern of S3 castable is portrayed in figure 4.16, which is in correspondence with exact stoichiometric composition of the formulated block. The influence of trace impurities presented during the processing of ceramics is well-known and exploited in the control of densification, grain growth and morphology during sintering (Mukhopadhyaya and Das Poddar, 2004). The ability to control microstructure is important for achieving desired properties.

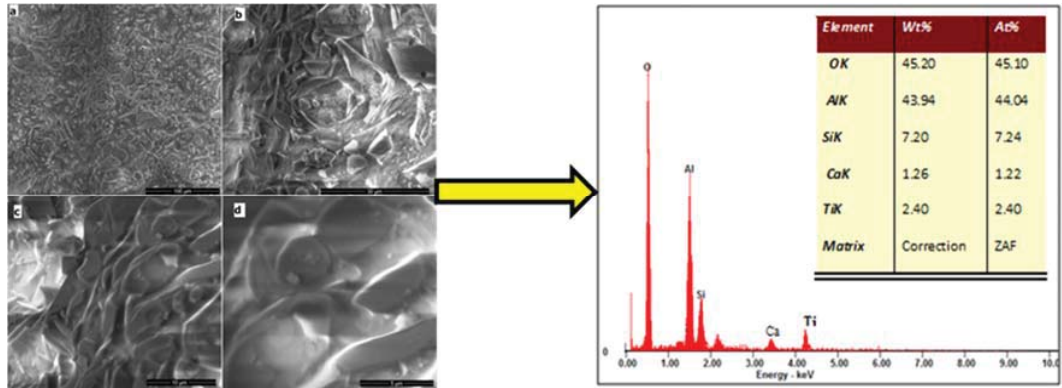


Figure 4.16: FE-SEM with EDS pattern of S3 castable prepared with HAC80.

4.4 Conclusion

By the present novel process, HACs were successfully prepared with very small crystallite size ranging 20-35nm. Cementing behavior promises a new era of industrial evolution through commercial implementation. Desired cementing phases, having high refractoriness, similar to CA, CA₂ and C₁₂A₇ were formed at relatively low temperatures. One advantage of this process is to exclude silicate phases having low eutectic temperatures in such cements. These silicate phases decrease the refractoriness and the cement cannot be used at higher temperatures. The XRD patterns of castables containing Bauxite and SiC show new phases formed, such as corundum and mullite, which were responsible for superior thermo-mechanical and physical properties. Castable samples prepared by HAC having 80% alumina have better physical and cementing properties than the HAC having 70% alumina. Needle like mullite formed at high temperature acts as a bonding phase which was responsible for increase in the CMOR. FE-SEM represents dense microstructure of all the samples and glassy phase appearance is due to the impurities present in bauxite.