Silica Sol Bonded High Alumina Castable: Effect of Reduced Sol

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Effect of reduction in sol content in no cement high alumina castable using water as partial replacement was studied. Different castable compositions, with different particles size distributions, were prepared and processed as per conventional castable making method. Pure silica sol and fixed amount of sol and rest water, as required for mixing and flowability, were added as bond material in the castables. Comparison in characteristics amongst the castables was done after heat treatment at different temperatures.

1 Introduction

The iron and steel industry has undergone tremendous changes due to the adaptation of various advanced technologies to improve the quality and production of steel. As a consequence, refractory materials used in various areas had to face severe challenges to meet the critical operational parameters at high temperatures with less down time. Hence, the attention of refractory researchers, manufacturers and users has gradually shifted towards unshaped refractories substituting the conventional and shaped ones for improved performance and life.

Unshaped refractories have quickly grown at the expense of bricks because of their faster, easier and cheaper installation; ready availability and reduced corrosion due to the absence of lining joints, greater volume stability and better spalling resistance [1]. Again, among the different unshaped refractories, castables lead in all areas like, research and development, manufacturing and application [2].

Refractory castables are premixed combinations of refractory grain, matrix components, bonding agents, and additives. The proportions of each component used vary in each castable composition to attain the desired properties essentially required for the intended application. Alumina cement binder is the main bonding system for castables for a long time but presence of CaO in cement results in higher moisture demand, higher porosity, lower strength, formation of low melting compounds and deteriorated high temperature properties.

Incorporation of sol-gel based bonding system, by using silica sol replacing alumina cement, is a great improvement in unshaped refractories and resulted in much better high temperature properties. Colloidal silica sols are stable water-based suspensions, containing up to 50 mass-% of nanometric spherical amorphous silica particles (8–15 nm diameter). When combined with other solid particles, like refractories, sol particles link together in branched chains, in a process known as gelation [3–5]. This gelation can be induced by water removal or pH variation. During the drying step, the hydroxyl groups (Si–OH) on the surface of the particles generate siloxane bonds (Si–O–Si), which results in a three dimensional network [4]. So, in a refractory system formation of a three dimensional network gel structure from a sol that surrounds the refractory aggregates results in strength in the system during drying. Sol reduces/removes the cement from the castable composition and on subsequent heating develops strength through the formation of ceramic bonding at low temperature (faster sintering due to finer size) resulting in superior properties [6–8]. Also addition of silica sol helps in flowability and formation of mullite on firing in alumina-based system. This helps in improving the chemical resistance and the hot-strength properties [9, 10]. For such advantages, there are plenty of literature available on the use of silica sol as binder in alumina based castable system. [6–8, 11–16]

However, there are some associated disadvantages also for silica sol systems. Strength generation occurs only after gelation and in many a cases it is delayed due to weather conditions and chemicals may be required to be added for required strength development in a desired time schedule. [17, 18] Also sol required special on site attention for its stability and correct amount of addition. Moreover any variation in silica sol addition may affect the final properties as it may differ the amount of silica content in the final composition. Also special storage conditions has to be maintained on site to avoid any settling or coagulation of the particles [19–21].

In such a context, limited use of silica sol might be a better option for getting opti-

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Tab. 1 Physico-chemical properties of different starting materials

Constituent	WTA Grain [%]	WTA Fines [%]	Reactive Alumina Fine [%]	Silica Sol [%]
SiO_2	0,08	0,11	0,03	29,8
Al_2O_3	99,3	99,1	99,5	
Fe_2O_3	0,03	0,2	0,03	
Na_2O+K_2O	0,25	0,45	0,1	
LoI				70,2
Physical properties				
Avg. particle size [μm]		99%<45	3	0,02
Bulk density [g/cm³]	3,61			
Apparent porosity [%]	3,93			

Tab. 2 Particle size distribution of different batch compositions

Material	Average Size of Fraction [μm]	q = 0,21	q = 0,23	q = 0,27	q = 0,29
WTA	4500	17	17,7	19,45	20,35
WTA	2500	9	9,1	9,8	10,1
WTA	1500	13	13,8	14,45	14,8
WTA	800	9	8,8	9,05	9,15
WTA	450	10	10,4	10,45	10,4
WTA	172	22	21,3	20,35	19,75
WTA	27	10	8,9	6,45	5,45
Reactive alumina	2,5	10	10	10	10

mum benefits in different application environments. But literature survey hardly provides any information on any research or trial work in that direction. Nanometric sized sol particles impart various beneficial characters in the castable system, so great reduction in sol content for sol-gel bonded castable will hamper its performance. But partial reduction in sol content and replacement by water for flowability purpose can be an option. And in the present work such an attempt has been made to study the effect of reducing silica sol content in high alumina based castable system, replacing some part of silica sol by water. Variation in particle size distribution of different castables were done by varying the distribution coefficient, as per Dinger and Funk model [22–24] and all the castable compositions were processed as per conventional technique and characterized.

2 Experimental

Silica sol bonded high alumina castables with different particles size distributions were prepared using white tabular alumina (WTA) grains and fines (Almatis/IN), react-

ive alumina fines (Almatis /IN) and silica sol (Dr. Khan's Laboratory/IN). Details of the starting materials are provided in Tab. 1. Continuous particle size distribution model is used for variation in the particles size distribution, as per Dinger and Funk model with distribution coefficients, q = 0,21; 0,23; 0,27 and 0,29 using the coarsest particle size of 6 mm and the finest of 0,001 mm. Tab. 2 shows the percentages of the different particle size fractions of the starting materials, For all the compositions, organic fibre at an amount of 0,05 mass-% was used for better removal of physical water during drying. All the different castable compositions were studied using only silica sol as binder and also fixed 4 vol-% to mass-% (that means 4 cm³ of sol in 100 g batch) of silica sol and rest amount of water as required to attain desired consistency/ flow properties.

All the different batches were processed under similar conditions. First, all the raw materials were dry mixed in a planetary mixer (Hobart, GB make, model N50). After a dry mixing of about 2 min, silica sol or sol and water (as required) combination was

added till the mix attains the desired flowability/consistency. Mixed compositions were characterized for flowability measurement, using a flow cup, as per Fang et al. [25] on a vibration table (AIMIL, India make) at 3200–4000 Hz, 0,5 mm amplitude after a vibration time of 30 s. Measurement of flow values was carried out on the flowed/spread mass in four different directions and the average value is presented.

Next the mixed batches were poured into lubricated moulds of dimension 50 mm³. The excess mix was scrapped off and smoothened by a trowel. The mixes were allowed to age for 24 h in inside the mould under humid condition. Next they were demoulded and allowed for 24 h air drying followed by oven drying for 24 h. The dried samples were then fired at 900 °C and 1550 °C with 2 h soaking time at peak temperatures. Dried and fired cubes were then characterized for bulk density (BD), cold crushing strength (CCS) and cold modulus of rupture (CMOR) [all measured as per Bureau of Indian Standard (BIS) specifications, IS 1528–1974, Part XII, Part VIII and Part IV, reaffirmed on 2002]. Each data represented here are an average of five individual measurements. Also the matrix part of 1550 °C fired samples were tested for phase analysis by X-ray diffraction method. Details of the process of the work is provided as flow diagram in Fig. 1.

Chemical analyses of the starting raw materials were conducted using the acid dissolution method. Densification studies were conducted using the conventional liquid-displacement method according to the Archimedes principle. Phase identification was performed in a X-ray diffractometer (Rigaku, Japan make) using CuKα radiation with a scanning speed of 2°/min. Firing were conducted in a programmable electric furnace (Bysakh & Co/IN). Strength measurements were done in a compressive testing machine (AIMIL/IN).

3 Results and discussion

Physico-chemical properties of the starting raw materials indicate (Tab. 1) that the materials are pure and contain very minimum amount of impurities. Among the two different aluminas used, RA is marginally purer and much finer compared to that of WTA fines. Silica sol used is also pure and having about 30 % solid content. Tab. 2 shows the

particle size distribution of different castable compositions with different distribution coefficients. It can be seen that increasing q value resulted in decreasing amount of fines and increasing amount of coarse content in the granular compositions.

Tab. 3 shows the amount of liquid (silica sol or silica sol and water) required to get the desired flow/consistency in the planetary mixer. In both the cases a decreasing trend of liquid requirement was observed with increasing q value, due to reducing amount of fines present in the systems resulting in reduced surface area to wet by the liquids. Also flow consistency in the planetary mixer was observed to be similar at a lower liquid content for only sol containing ones in all the compositions, this may be due to the presence of nano-metric silica sol particles which improves flowability. Fig. 2 shows the flow values in flow cup test of the different castable compositions. Compositions contain both silica sol and water showed marginally higher flow values compared to that of the pure silica sol containing ones. This may be due to the excess amount of free water available, which might have helped in increasing the flow values after vibration. Again Increase in q value was found to deteriorate the flow properties. This is due to increased coarse fractions present in the system resulting in greater friction within the system and producing lower flowability.

3.1 Densification study

Increase in q value was found to increase the bulk density values for all the temperatures (Fig. 3) due to the presence of greater extent of coarse content replacing fines. Also use of water (in sol + water compositions) partially replacing sol was found to result in relatively lower density values for all the different composition at all the heat treatment temperatures. Presence of excess water has produced voids/porosity in the compositions after drying and resulted in lower density values. Again increase in temperature from 110 to 900 °C was found to deteriorate the density values marginally for all the compositions, may be due to the breaking of hydroxyl bond (removal of chemically bonded water of sol/gel) and poor initiation of sintering. However, further increase in temperature to 1550 °C was found to enhance the densification strongly due to sintering. Very fine silica particles
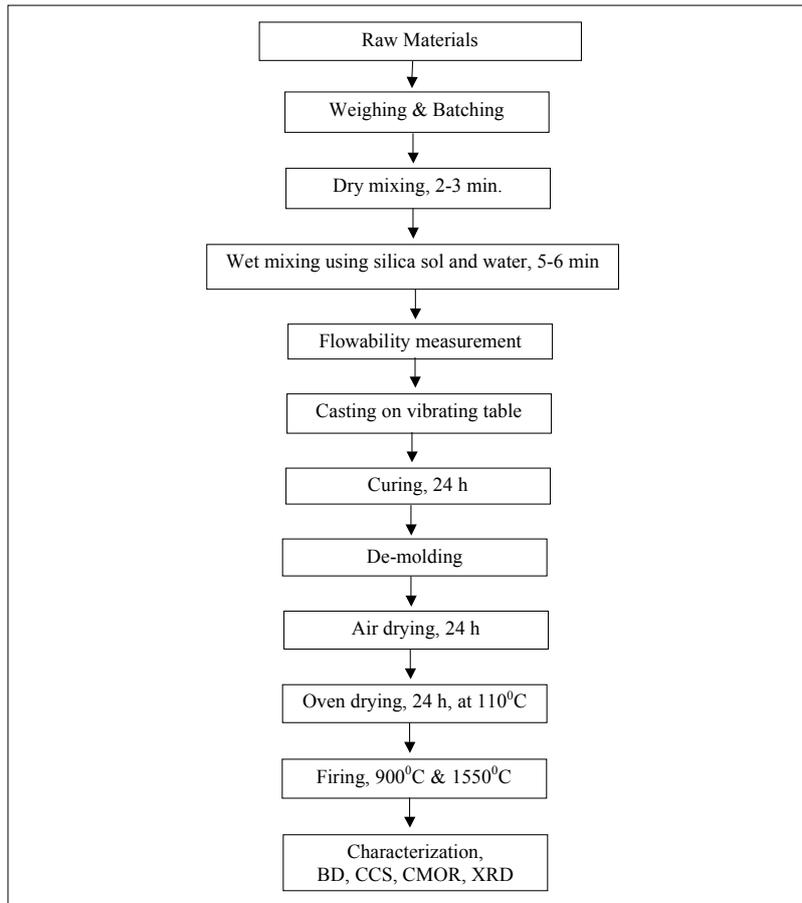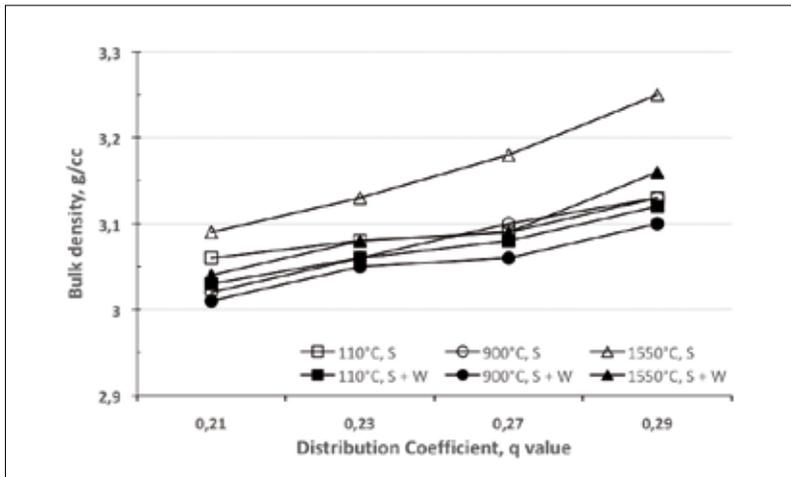

Fig. 1 Flow diagram of the process

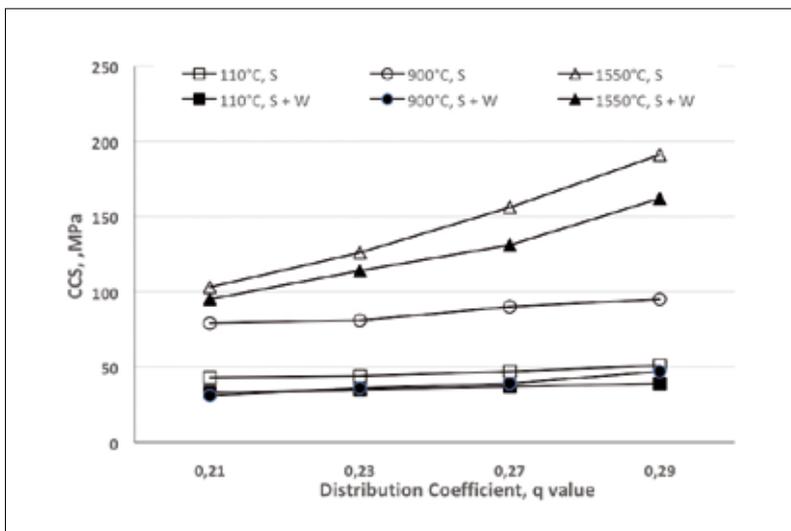**Tab. 3** Sol and water demand of different batches, vol.-% to mass-% (cm³ of liquid per 100 g batch)

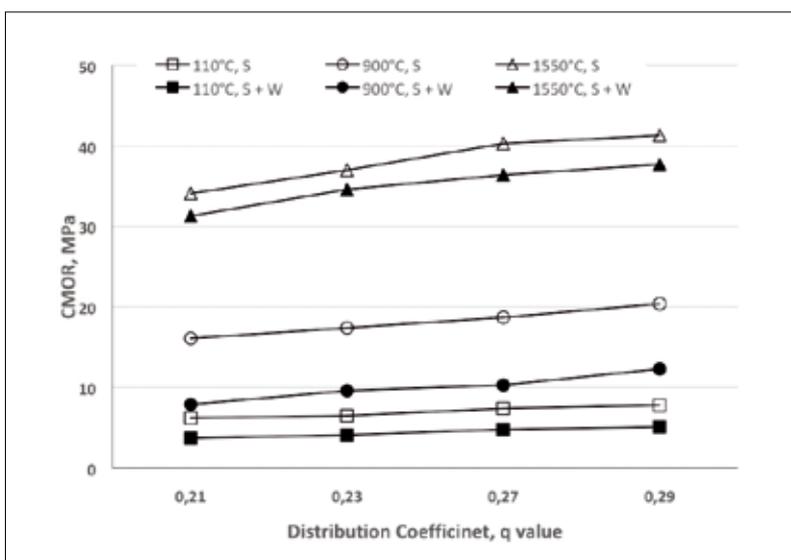| q Value	Silica Sol and Water Required	
	only silica sol	4 silica sol + water [%]
0,21	5,5	1,73
0,23	5,4	1,65
0,27	5,35	1,53
0,29	5,32	1,33
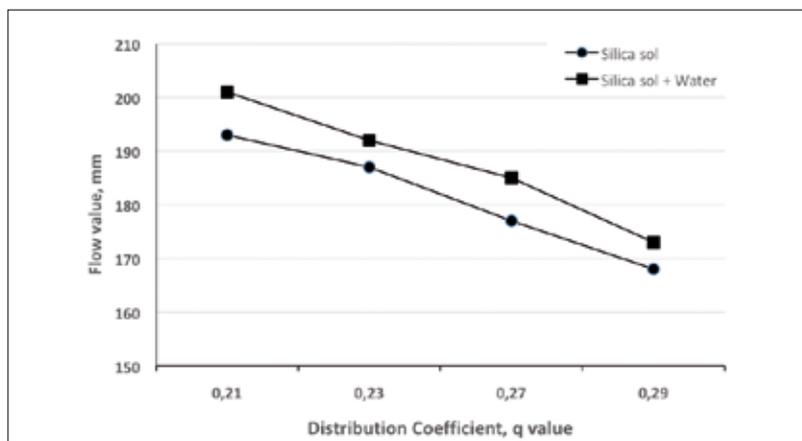
Fig. 2 Variation of flow value of the castables

Fig. 3 Variation of bulk density of the castables against processing temperature

Fig. 4 Variation of cold crushing strength of the castables against processing temperature

Fig. 5 Variation of CMOR of the castables against processing temperature

from sol has resulted in good densification and produced high density at 1550 °C.

3.2 Strength measurements

Strength values were found to be dependent on q values of the composition, temperature of heat treatment and extent of sol content (Fig. 4). In general increasing temperature resulted in an increase in cold crushing strength (CCS) but the rise in strength was very prominent when the compositions were fired at 1550 °C. Increase in q values were also found to increase the strength values, which may be associated with the increased density values and corresponding packing. But use of water replacing partial extent of sol was found to adversely affect the strength for all the heat treatment conditions, associated with lesser extent of gel bonding, compared to that of only sol containing compositions. Also higher amount of free water present in sol + water containing compositions produced greater extent of void space/porosity and resulted in poor strength. Cold modulus of rupture values (CMOR, Fig. 5) were also found to follow exactly the similar trend as observed for the CCS values.

3.3 Phase analysis

Phase analysis study of the castables fired at 1550 °C was done by powder x-ray diffraction technique. All the different compositions showed very similar XRD pattern, corundum was observed as the main phase with mullite as the minor one. Silica particles from sol have reacted with the fine alumina particles during sintering and resulted in formation of mullite phase. This mullite formation has resulted in further bonding within the castables and resulted in much increased strength at 1550 °C. Fig. 6 shows the phase analysis study of the only sol containing composition with q value 0,21, as a representative one.

4 Conclusion

High alumina refractory castable with different particle size distributions (by varying the distribution coefficients) were studied using bond material as pure silica sol and reduced amount of sol and water in combination. Marginally better flow values were obtained for the sol and water containing system but density and strength values were found to be poorer compared to that of the pure sol
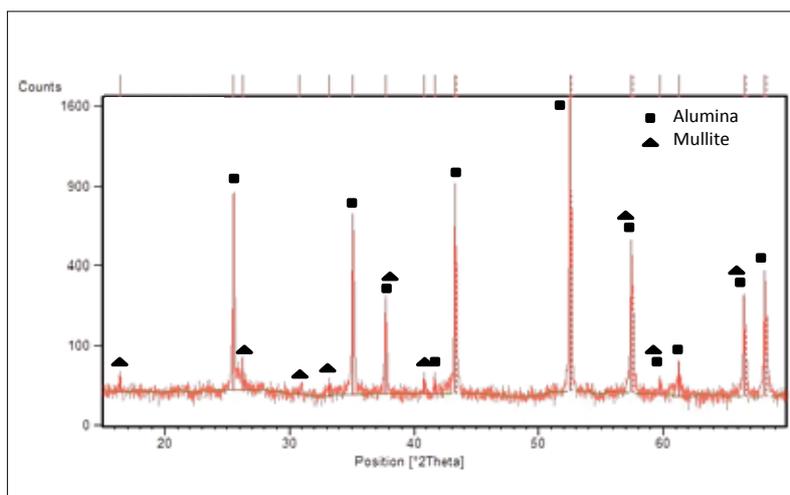
containing system. Mullite was found to be formed in the composition on firing at 1550 °C which is responsible for better bonding and increased strength at high temperatures.

Acknowledgements

Fig. 6 Phase analysis of 1550 °C sintered only sol containing castable with q = 0,21

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