ABCSA Raw mix Design: Correlation between Chemical Constituents and Mineralogy

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ABSTRACT The cement industry has been identified as one of the main contributors to climate change due to greenhouse gas emissions, mainly CO₂. Therefore, to meet CO₂ reduction targets, cement producers are working on different strategies of minimizing its emission, one of which is alternative clinkers. This study assessed the impact of variations of the raw mix design, concerning the type and proportions of materials, on the formation of calcium sulphoaluminate belite-type clinkers. Various materials were used to produce raw mixes for different percentages of belite, yeeliminite, and other minerals in resultant clinkers. Computer-based theoretical mixes were designed with different percentages of SiO₂, CaO, Al₂O₃, Fe₂O₃, and SO₃ and then the designed mixes were fired in a laboratory furnace at 1250°C with 20 min retention time. The resultant clinker samples were characterized with X-ray diffraction. The quantification of minerals in each sample was carried out with Rietveld refinement. The obtained results confirmed the correlation between the mineralogy and chemical constituents in the raw mix. The C₄AF percentage of the resultant clinker samples increased with an increase in Fe₂O₃ percentage. C₄A₃\$ content varied with the amounts of Al₂O₃, SO₃, and CaO. The mineral percentage of C₂S in the designed mixes had a clear correlation with the constituents of SiO₂ and CaO. Anhydrite percentage in the resultant minerals changed with the SO₃ content in the raw mix. These results should aid in the determination of the optimum amount of chemical constituents and minerals required for the development of calcium sulphoaluminate clinker.

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1. INTRODUCTION

The cement industry consumes huge amounts of natural limestone, other raw materials, and fuels. The production of cement involves decarbonization of CaCO₃ and requires a high temperature at around 1450°C. Both these processes produce CO₂ into the environment. Hence, the cement industry has been identified as one of the main contributors to climate change due to greenhouse gas emissions, mainly CO₂. In 2018, global cement production was about 3.99 billion tonnes, accounting for about 8% of all anthropogenic carbon dioxide emissions (CEMBUREAU 2019). Ordinary Portland cement (OPC), a blend of clinker (about 95%) and gypsum, is the most used binder in the world. The OPC clinker is usually obtained by heating a mixture (raw meal) of limestone (Tregambi et al. 2018; Barcelo et al. 2014) and other additives such as laterite, bauxite, clay, etc., in a rotary kiln at about 1500°C. The entire process of clinker production involves consumption of thermal and electrical energy and releases about 0.83 kg of CO₂/1 kg of OPC.

Now the scenario of global cement changes into sustainability and minimization of CO₂ production and targets for CO₂ reduction came into the picture in all the countries through low carbon technology road map (LCTR) (World Business Council for Sustainable Development 2018). Because of LCTR guidelines, all the cement industries are working out on different levers to minimize CO₂ produc-

tion. According to the LCTR, alternative clinker is one of the potential levers for reducing CO₂. In this direction, researchers are working out to develop new alternative clinker systems to mitigate CO₂ emissions. Belite sulphoaluminate clinker is also one of the established alternative clinkers to address the CO₂ mitigation effectively (Telesca et al. 2017; Schneider 2019; Shi et al. 2019; Gartner and Sui 2018; Walling and Provis 2016). Some countries (China and Japan) already started the production of BCSA clinker and produced blended types of cement with different percentages along with the conventional clinker in bulk quantity for commercial applications.

For the conventional clinker, a quality expert uses Boague or Taylor (Bogue 1929; Taylor 2015) theoretical calculations to understand the mineralogy of the resultant clinker for its further applications. But, so far very few theoretical calculations are reported to assume its mineral percentage instead of quantitative estimation for its effective applications (Borštnar et al. 2020; Telesca et al. 2020; Winnefeld et al. 2017; Beltagui et al. 2017; Chen and Juenger 2011). In the present paper, the authors studied the impact of chemical constituents (Fe₂O₃, Al₂O₃, SiO₂, SO₃, and CaO) of the raw mix on the formation of the mineral such as ferrite (C₄AF), yeeliminite (C₄A₃\$), Belite (C₂S), and anhydrite (C\$) in the BCSA clinker and discussed the correlation between the chemical constituents of raw mix and its mineralogy of the clinker.

2. MATERIALS AND METHODS

Computer-based raw mix designs were carried using different raw materials and control parameters of all the mix designs are given in Table 1. Alumina Modulus (AM) in the different raw mixes varied from 1.03 to 3.91, silica modulus (SM) varied from 0.39 to 0.76, and lime saturation factor (LSF) of the clinkers varied from 0.68 to 1.09. stoichiometric quantities of all the materials were weighed and mixed thoroughly for homogenized sample preparation. The nodules of about 10 mm size of raw mix samples were prepared by mixing manually and dried at ambient temperature. These nodules were subjected to thermal treatment at the temperature of 1250°C by retaining them for 20 min in a laboratory furnace. The ambient cooled fired nodules were then analyzed for mineralogical analysis by XRD. The resultant clinker samples were ground to powder with 100% passing through 45 μ IS sieve and then all the samples were characterized by XRD for mineralogical evaluation. XRD scans were acquired by using a Rigaku Smart Lab powder diffractometer equipped with a 3.0 kW sealed Cu X-ray source, a graphite monochromator to filter out the Cu K α radiation, and a 1D detector. The scans were performed between 10° and 70° 2 θ with a 0.02° step size and a counting time of 1 degree per minute.

The quantification of minerals in all of the samples was carried out through Rietveld refinement using the MDI JADE 2020 software. The crystal data for the Rietveld method contained monoclinic C₃S (ICDD 98-000-0237), monoclinic Belite (ICDD 98-000-0275), cubic C₃A (ICDD 98-000-0136), cubic yeelimite (ICDD 01-083-9042/98-000-0467), orthorhombic C₄AF (ICDD 04-0105796), anhydrite 98-000-0090, mayenite (98-000-0301), and cubic lime (ICDD 01-082-1690). The parameters refined included scale factors, lattice parameters, profile shape parameters, specimen displacement, and background coefficients. A pseudo-Voigt profile was used to model the background. Atomic coordinates, thermal parameters were not refined due to the complexity of crystal structures of the clinker phases. Correction for preferred orientation was not included in the refinement.

3. RESULTS AND DISCUSSION

Previous studies have shown that BCSA cement can be prepared effectively from natural materials and waste materials (Beretka et al. 1992; Roy et al. 1999; Sahu and Majling 1994), and these cements have been produced commercially in China for over 30 years. However, there are still many unanswered questions about this system. The effects of raw material chemical composition on phase formation in clinkers were not well defined in earlier published literature. The main phases in this clinker exhibit different hydration behavior. The hydration products of BCSA cement depend on the amount of calcium aluminosulfate compound which has a substantial impact on setting

TABLE 1. Lime saturation factor (LSF), alumina modulus (AM) and silica modulus (SM) of all the clinkers.

	CLR 1	CLR 2	CLR 3	CLR 4	CLR 5	CLR 6	CLR 7	CLR 8
AM	1.97	1.99	3.91	1.05	1.05	1.06	1.03	1.04
SM	0.37	0.39	0.71	0.76	0.70	0.68	0.72	0.74
LSF	0.91	0.94	1.09	0.72	0.68	0.76	0.73	0.73

time, strength development and volume stability (Glasser and Zhang 2001; Winnefeld and Barlag 2010; García-Maté et al. 2015). The hydration reaction of C₄A₃\$ with calcium sulfate is very fast, leading to the formation of ettringite (C₃A.3C\$.32H; C₆A\$₃H₃₂) and aluminum hydroxide (Al(OH)₃; AH₃) (Glasser and Zhang 2001; Winnefeld and Barlag 2010; García-Maté et al. 2015; Hanic et al. 1989). These product minerals were responsible for the quick setting and contributed to the early-age strength. In the absence of calcium sulfate, C₄A₃\$ reacted with water and formed monosulfate (C₃A.C\$.12H; C₄A\$H₁₂) (Winnefeld and Lothenbach 2010). The increase in ferrite content promoted the compressive strength after seven days of hydration, but it had an adverse effect on cement strength at early ages. The optimum content of ferrite was between 10 and 20% for BSA cement containing approximately 35% ye'elimite. The phases expected to synthesis were C₄AF, C₄A₃\$, C₂S, C\$ and free CaO (C), as shown in Equations 1 to 5.

$$\%C_4AF = 3.043 \times (\%Fe_2O_3) \quad (1)$$

$$\%C_4A_3\$ = 1.995 \times (\%Al_2O_3) - 1.273(\%Fe_2O_3) \quad (2)$$

$$\%C_2S = 2.867 \times (\%SiO_2) \quad (3)$$

$$\begin{aligned} \%C\$ &= 1.700 \times (\%SO_3) - 0.445 \\ &\times (\%Al_2O_3) + 0.284 \times (\%Fe_2O_3) \end{aligned} \quad (4)$$

$$\begin{aligned} \%C &= 1.000 \times (\%CaO) - 1.867 \times (\%SiO_2) - 1.054 \\ &\times (\%Fe_2O_3) - 0.550 \times (\%Al_2O_3) - 0.700 \times (\%SO_3) \end{aligned} \quad (5)$$

Theoretical potential phases of all the clinker samples were calculated based on the above equations and given in Table 2. The true mineralogical compositions of the resultant clinker samples were calculated through the Rietveld refinement and observed a difference of all the minerals between the actual percentage of C₄A₃\$, C₂S, C₄AF, C\$ and free lime with the theoretical calculations.

FIGURE 1. X-ray diffractogram of all the clinker samples.

TABLE 2. Potential mineral phases percentages of all the clinkers.

	CL 1	CL 2	CL 3	CL 4	CL 5	CL 6	CL 7	CL 8	CL 9
C ₄ A ₃ \$	32.06	32.29	22.51	28.53	26.51	25.09	26.38	27.87	28.48
C ₂ S	30.27	28.66	34.29	46.33	47.14	45.22	47.16	47.93	48.66
C ₄ AF	22.57	17.75	10.49	12.80	19.01	20.04	17.81	16.15	12.01
C\$	1.23	9.22	9.99	5.59	3.07	2.58	2.08	1.71	5.13
C	10.30	8.57	17.46	1.14	-1.42	3.95	2.48	2.68	1.29

TABLE 3. Chemical constituents of all the clinkers.

	CL 1	CL 2	CL 3	CL 4	CL 5	CL 6	CL 7	CL 8	CL 9
SiO ₂	10.56	10.00	11.96	16.16	16.44	15.77	16.45	16.72	16.97
Al ₂ O ₃	20.80	19.90	13.48	16.98	17.27	16.78	16.96	17.35	16.79
Fe ₂ O ₃	7.42	5.84	3.45	4.21	6.25	6.59	5.86	5.31	3.95
CaO	52.72	51.09	57.01	50.01	49.06	52.93	51.97	52.30	51.10
MgO	1.20	1.17	2.61	1.90	1.88	0.88	1.22	1.19	1.42
SO ₃	4.93	9.66	8.83	7.03	5.28	4.81	4.68	4.66	6.76
Na ₂ O	0.61	0.54	0.20	0.36	0.48	0.56	0.97	0.63	0.95
K ₂ O	0.20	0.18	0.34	0.87	0.92	0.37	0.19	0.57	0.25

In order to understand the effect of chemical constituents on the resultant minerals, raw mix were designed with varying the LSF, AM and SM from 0.68–1.09, 1.05–3.97 and 0.37–0.76. Because, these three were the critical parameters for control the product quality at an industrial scale. Chemical analysis of all the clinker samples was carried out by the conventional method and the obtained results in the hypothetical oxide form are summarized in Table 3. From these results, it was observed that all hypothetical form of chemical constituents in the designed mixes varied significantly. The SiO₂ constituent varied from 10.00 to 16.97, Al₂O₃ varied from 13.48 to 20.80, Fe₂O₃ varied from 3.95 to 7.42, CaO varied from 49.06 to 57.01 and MgO varies from 0.88 to 2.61.

Mineralogical analysis of all the clinker samples was carried out by QXRD and the obtained results are reported in Table 4. The results confirmed the formation of belite (C₂S), yeelimite (C₄A₃\$), brownmillerite (C₄AF), alite (C₃S), anhydrite (C\$), etc. at 1250°C. Quantification of all the minerals was investigated through whole profile pattern fitting (Rietveld refinement) and observed a correlation with the chemical constituents. An X-ray diffractogram of all the samples is displayed in Figure 1. The correlation of mineral percentage with chemical constituents was studied and results are presented in Figures 2 to 5. The typical clinker

mineral composition of the industrial clinker was in the range of 45–65% of C₃S, 15–35% of C₂S, 3–10% of C₃A and 8–16% of C₄AF.

It was observed that when the percentage of C₄AF in the CLK 3 and CLK 9 samples were 12.10% and 14.50% when the Fe₂O₃ content in the clinker was 3.45% and 3.95% respectively. Similarly, in the other clinker samples as the content of Fe₂O₃ was increased, C₄AF content increased significantly.

The hypothetical chemical constituent of SO₃ formed anhydrite and yeelimite in the resultant clinkers. The SO₃ content played a vital role in the formation of BCSA clinker. A correlation with the hypothetical chemical constituent of SO₃ was observed in the formation of anhydrite mineral in the resultant clinker samples. All the clinker samples significantly showed a correlation of anhydrite mineral percentage with a hypothetical form of SO₃ chemical constituent. CLK 1, 6, 7, and 8 clinkers contained an SO₃ amount in the range of 4.66–4.93, having less than 1% of anhydrite mineral. The SO₃ content in CLK 2 and 3 clinkers are 8.83 and 9.66 produces 5.10 and 5.40% of anhydrite in the resultant clinkers respectively. Hence, the increase in the amount of SO₃ enhances the formation of anhydrite in the resultant clinker. It is observed that amount of anhydrite in the clinker samples mainly depends on the amount of SO₃. The

FIGURE 2. Correlation between C\$ minerals and hypothetical form of SO₃ content. FIGURE 3. Correlation between C₄AF minerals and hypothetical form of Fe₂O₃ content.

FIGURE 4. Correlation between $C_4A_3\$$ minerals, hypothetical form of Al_2O_3 , Fe_2O_3 , SO_3 , and CaO .

maximum amount of SO_3 required for the formation of less than 1% of anhydrite is 4.93%. The present study confirmed that the formation of yeelimite in the resultant clinker does not depend only on any single chemical constituent, but it had a correlation with hypothetical form of Al_2O_3 , SO_3 , CaO , and Fe_2O_3 . Even though CLK 2 and CLK 9 samples had almost the same percentage of CaO , the amount of $C_4A_3\$$ (27.60%) present in the sample CLK 2 was greater than that of $C_4A_3\$$ (25%) in the CLK 9 sample. This may be due to the presence of a greater quantity of Al_2O_3 in CLK 2 than that in CLK 9. There was a notable difference in the mineral percentage of $C_4A_3\$$ in CLK 2 (27.60%) and CLK 3 (17.50%) and also in the amount of Al_2O_3 in CLK 2 (19.90%) and CLK 3 (13.48%). CLK 5 and CLK 8, CLK 4 and CLK 7, and CLK 6 and CLK 9 pair clinker samples had almost equal percentage of Al_2O_3 content and the amounts were 17.27% and 17.35%, 16.98% and 16.96%, and 16.78% and 16.79%, respectively. CLK 8 sample had a high content of SiO_2 , CaO and lower content of Fe_2O_3 when compared with CLK 5 and it was observed that $C_4A_3\$$ of CLK 8 was more (21.20%) than that of CLK 5 (17.80%). The results indicated that the increase in the amount of CaO and decrease in the amount of Fe_2O_3 influenced the percentage of $C_4A_3\$$ content in the resultant clinker. However, sample CLK 7 had a high content of SiO_2 and CaO compared with CLK 4; the amount of $C_4A_3\$$ in CLK 7 (13.80%) was lower than that of CLK 4 (18.30%). This may be due to the greater amount of Fe_2O_3 in CLK 7. Hence, the content of Fe_2O_3 significantly influenced the amount of $C_4A_3\$$. CLK 5 and CLK 7 samples had a similar percentage of SiO_2 (16.44% and 16.45%, respectively). CLK 5 had a higher content of Fe_2O_3 and SO_3 than that of CLK 7 and $C_4A_3\$$ of CLK 5 was more (17.80%) than that of CLK 7 (13.80%). Increasing the Fe_2O_3 content or decreasing the amount of SO_3 enhanced the amount of $C_4A_3\$$. This may be due to insignificant variation of Al_2O_3 and high

FIGURE 5. Correlation between C_2S minerals, hypothetical form of SiO_2 , and CaO content.

content of SO_3 in CLK 5 than that of the CLK 7 clinker sample. These results confirmed that the formation of $C_4A_3\$$ synergistically depends on the optimum amount of chemical constituents such as Al_2O_3 , CaO , SO_3 , SiO_2 , and Fe_2O_3 .

The formation of belite content varied with the hypothetical form of chemical constituent of CaO and SiO_2 . The difference in SiO_2 content in CLK 1 and CLK 2 samples was negligible. Hence, the percentage of belite content in both the samples was approximately equal. The CLK 2 and CLK 9 samples contained almost the same percentage of CaO and there was a significant difference in the percentage of SiO_2 (10.00% for CLK 2 and 16.97% for CLK 9). CLK 2 and CLK 9 contained 32.10% and 52.80% of belite with substantial variation. Similarly, although the difference of SiO_2 content in CLK 1 and CLK 3 samples was marginal, a significant difference in the belite content was observed (CLK 1 contained 32.20 and CLK 3 contained 51.90%), it may be due to the variation of CaO (51.09% of CLK 2 and 57.01% of CLK 3) and Fe_2O_3 content of CLK 2 (5.84%) and CLK 3 (3.45%). The amount of Fe_2O_3 significantly reduced the $C_4A_3\$$ content in the resultant clinker. Hence, Fe_2O_3 indirectly increased the C_2S percentage in the resultant clinker samples. The amount of C_2S in CLK 4 and CLK 5 samples clearly indicated that the SiO_2 content influenced the amount of C_2S in the final product.

4. CONCLUSIONS

Resultant mineralogy of BCSA clinkers was significantly affected by its modulus values such as AM, SM, and LSF of mix designs. Theoretical calculation showed the correlation between mineralogy with their respective one particular chemical constituent. In order to understand this thoroughly, the actual percentage of minerals was estimated using the whole profile pattern refinement method (Rietveld

TABLE 4. Mineralogical QXRD results of all the clinkers.

	CL 1	CL 2	CL 3	CL 4	CL 5	CL 6	CL 7	CL 8	CL 9
$C_4A_3\$$	21.80	27.60	17.50	18.30	17.80	24.30	13.80	21.20	25.00
βC_2S	32.20	32.10	25.50	36.50	51.60	48.20	39.80	50.10	44.50
αC_2S	0.40	0.00	26.40	19.60	3.00	0.00	9.80	4.30	8.30
C_4AF	28.90	21.50	12.10	14.00	21.90	22.10	23.80	18.40	14.50
$C\$$	1.00	5.40	5.10	3.80	2.60	0.30	0.70	0.90	1.60
$C_{12}A_7$	1.50	1.20	0.00	0.70	0.00	0.00	3.70	1.40	0.70
C_3S	5.1	3.7	3.8	2.10	0.00	1.40	6.40	1.4	0.7
C_3A	0.6	2.6	2	1.70	1.40	0.10	3.00	0.4	1.3
FCaO	2.1	2.8	4.5	0.9	0.2	0.1	0.8	0	0

method) and a difference was observed between the theoretical and calculated percentage of minerals. In view of this difference, the correlation between the resultant minerals and their hypothetical form of chemical constituents was studied. A noteworthy correlation was observed in the case of C_4AF with the hypothetical form of Fe_2O_3 , anhydrite with the hypothetical form of SO_3 , C_4A_3S with the hypothetical form of Al_2O_3 , Fe_2O_3 , SO_3 , CaO , and C_2S with SiO_2 . The amount of anhydrite mineral was less than 1% when the content of SO_3 was in the range of 4.66–4.93%. As the percentage of SO_3 increased beyond 4.93%, it significantly improved the anhydrite mineral in the resultant clinker. Fe_2O_3 proportionately increased the C_4AF percentage in the respective clinker. However, the effect of Fe_2O_3 on the amount of C_4A_3S depended on other hypothetical forms of chemical constituents. The C_4A_3S mineral percentage varied with the hypothetical form of Al_2O_3 , $Al_2O_3+Fe_2O_3$, SO_3 , and CaO . In the presence of the optimum amount of SO_3 in clinkers, CaO and Al_2O_3 linearly increased the C_4A_3S percentage. The percentage of C_2S increased while improving the CaO content and Fe_2O_3 due to decreasing C_4A_3S content. The chemical constituents of SiO_2 , CaO , Al_2O_3 , Fe_2O_3 , and SO_3 were key parameters in the design of BCSA clinkers. Hence, these constituents should be optimized according to the desired percentage of belite, yeelimite, C_4AF , and anhydrite in the mix designs. This correlation would help to determine the optimum level of constituent and mineral percentage for achieving desirable properties and designing BCSA clinkers.

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AUTHORS' CONTRIBUTIONS

SP, SV conceived and designed the analysis, collected the data, performed the analysis, and wrote the paper. SR performed revisions. SKC and BNM provided authorization for submission to the journal. All authors read and approved the final version of the manuscript.

COMPETING INTERESTS

The authors have no conflicts of interest to declare.

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