



Synthesis and characterization of chloride resistant cement from industrial waste through geopolymerization



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ABSTRACT

Chloride dissemination in cementitious materials is one of the key elements influencing the strength execution of mortar and concrete. In this paper the impact of silica and alumina ratio (Si/Al) on strength and chloride resistance in sodium and magnesium chloride solutions on bagasse bottom ash and china clay based geopolymer composites is studied and reported. Nine geopolymer composites with Si/Al ratio of 2.5–3.5 containing thermally activated clay after curing in 5% sodium and magnesium chloride solutions for seven, fifteen and thirty days were studied. It was found that geopolymer with silica to alumina ratio of 2.7 show maximum compressive strength 18.85 N/mm² and highest resistance to chloride environment. The X-ray Diffraction pattern showed that chloride does not affect the basic frame of geopolymer when having optimum silica to alumina ratio. Geopolymer was found to be more resistant to magnesium chloride as compared to sodium chloride.

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1. Introduction

Ordinary Portland cement, not only causes production of environmental pollution, but also suffers from the attack of chloride in aggressive environment when exposed for a longer time. Concrete used in comparatively harsh environment especially in chemical industries etc, chloride resistance become the most important requirement. Attack of chloride on concrete results to a strength loss and damage to its chemical composition (Amin et al., 2015a, 2015b; Chindaprasirt et al., 2017; Delair et al., 2012). In order to address these problems, a number of alternatives for cement are being developed in which the most promising ones are the geopolymer binders (Ren et al., 2017; Innocent et al., 2010), which are being synthesised by the activation of aluminosilicate material like metakaolin and industrial ashes, using an alkali or alkaline solution. A three dimensional structure, having AlO₄ and SiO₄ units with alkali ions balancing the charges, is obtained as a result (Renata et al., 2015; Babae and Castel, 2016). Palomo et al. (1999) studied a metakaoline based geopolymer in sulphuric acid (pH = 3) for 270 days, and a very minute change in compressive strength after the treatment was found. A fly ash-based geopolymer was exposed to 1 M sulphuric acid for 56 days and reported that it was structurally

undamaged and sustained an extensive load capacity after the treatment of acid (Habert et al., 2011; McLellan et al., 2011). The same geopolymer has also been studied in HCl solution with a pH of 1 for 90 days and concluded that binder revealed an adequate performance in chloride atmosphere (Amin, 2014; Karakoç et al., 2016). The behaviour of Geopolymer has also been studied in sulphuric and acetic acid with a pH of 0.8 and 2.4 respectively for 150 days and found that the activator solution had a substantial impact on the geopolymer stability in hostile environment (Chindaprasirt et al., 2013a, 2013b). The chloride permeability and initiation of chloride induced corrosion of geopolymer concrete in accelerated chloride environment using longer test period has been studied. Corrosion initiation was monitored in embedded rebar in 2% chloride contaminated concrete. Corrosion state of the rebar was monitored using non-destructive test method using Cu/CuSO₄ reference electrode. The results showed that the apparent chloride diffusion coefficient of blended fly ash and slag geopolymer concrete is lower than that of OPC concrete (Temuujin et al., 2010).

In spite of the fact that advantages of geopolymer is broadly accepted, still some degradation in structure has also been reported. Allahverdi and Šivára 2001; Allahverdi and Šivára 2005 proposed a potential mechanism of degradation of geopolymeric binder in a solutions of HNO₃ and H₂SO₄ with a pH of 1 each, the mechanism for which is summarised as: a). substitution of cations like K⁺ and Na⁺ by H⁺ (b). Development of siliceous structure because of vacancies in the framework.

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The present work aims to study the behaviour of bagasse bottom ash and thermally activated china clay based geopolymer in chloride environment. Two types of solutions were used as chloride environment; sodium chloride and magnesium chloride. Different geopolymer with different $\text{SiO}_2/\text{Al}_2\text{O}_3$ in the range of 2.5–3.5 were studied. The mass of geopolymer bars was measured before and after the acid treatment.

2. Material and methods

In this work bottom ash of sugar industry, sodium silicate, china clay, de-ionized water and sodium hydroxide were used for the synthesis of geopolymeric binder. A mixture of 3.0 M solution of NaOH and Na_2SiO_3 each in the ratio of 1:1 was used as alkaline solution. A number of geopolymer composites were synthesised with different Si/Al like 2.5, 2.6, 2.7, 2.8, 2.9, 3.0, 3.16, 3.28 and 3.5 and polymer obtained were named as GP₁, GP₂, GP₃, GP₄, GP₅, GP₆, GP₇, GP₈ and GP₉ respectively as shown in Table 1. The paste of geopolymer after full mixing was molded into metallic molds having inner diameter of 50 mm as per American Society for Testing and Materials (ASTM) and treated in oven for 24 h keeping the temperature as 60 °C and then de-molded. The samples were further treated at the same temperature for 7 days. Compressive strength of geopolymer composites at the curing age of 7 days was studied using Universal Testing Machine (UTM). To study the stability of the geopolymer in acidic solutions, all the samples were molded into bars and dehydrated at room temperature for 2 h, then dipped in 1% aqueous solution of sodium and magnesium chloride each for 7 d, 15 d and 30 d. The effect of chloride attack on the geopolymer was studied in terms of the mass loss.

During the first 5 h of the contrast of specimens with acid solutions, the pH acid solution was monitored after every 2 min with a PH meter at the room temperature (25 °C). Then, the pH was recorded after 7, 15 and 30 days of immersion period.

3. Results and discussion

XRD peaks of ash used as a raw material is presented in Fig. 1. The figure shows the major diffraction peaks at 20° and 40° showing the amorphous behaviour of silica while some small peaks represent other components like SiC and TiO. Major peaks are shown for quartz and kaolinite representing the maximum clay content in the ash. The XRD pattern of thermally treated clay is given in Fig. 2. It is clear from the figure that the major phases of calcined clay are metakaolinite and quartz represented as MK and Q respectively. It may be noted that kaolinite phase is converted into metakolinite at 900 °C. Some small fractions of kaolinite are still present, which are shown in XRD represented as K. Fig. 3 shows the

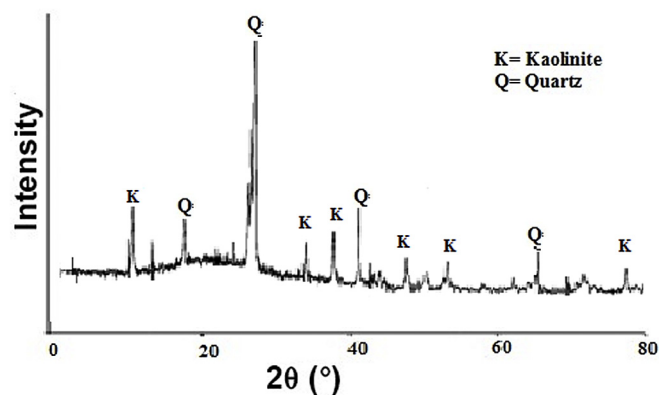


Fig. 1. XRD Pattern of the bagasse ash.

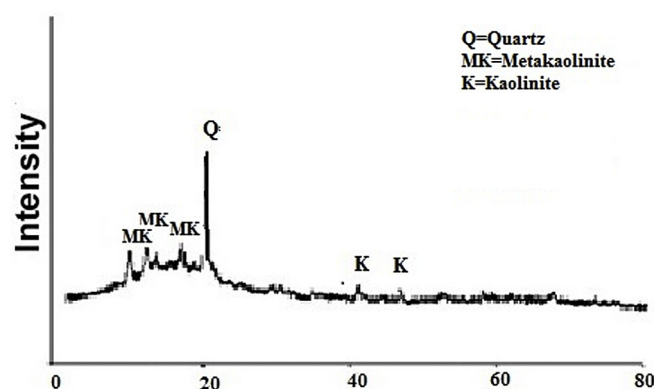


Fig. 2. XRD of clay calcined at 900 °C.

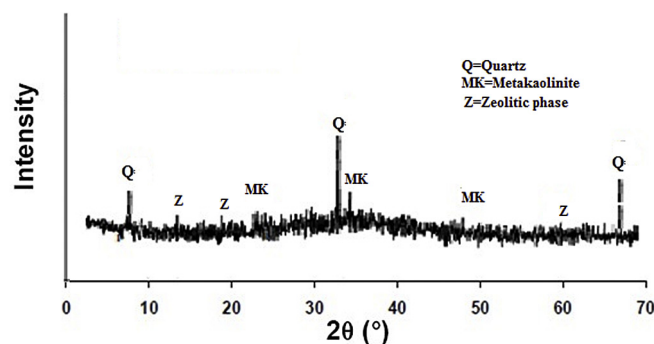


Fig. 3. XRD pattern of geopolymer.

Table 1
Ratio of Geopolymer composite.

Geopolymer ID	Material selected (Wt %)			$\text{SiO}_2/\text{Al}_2\text{O}_3$
	Bagasse ash	China clay	Sodium silicate	
GP ₁	36.00	64.00	0.00	2.50
GP ₂	34.00	61.00	5.00	2.60
GP ₃	36.00	59.00	5.00	2.70
GP ₄	38.00	57.00	5.00	2.80
GP ₅	40.00	55.00	5.00	2.90
GP ₆	40.00	53.00	7.00	3.00
GP ₇	41.00	51.00	8.00	3.16
GP ₈	42.00	49.00	9.00	3.28
GP ₉	44.00	46.00	10.00	3.50

XRD peaks of geopolymer composite. Geopolymer synthesised in this work, though seems amorphous in the early stage, but zeolitic phase may also be seen in the peaks represented as Z.

The compressive strength of geopolymer composite is greatly dependent upon the mixing ratio of silica and alumina. Strength of geopolymer having different ratio of Silica/Alumina is given in Fig. 4. The figure shows that the highest compressive strength is observed as 18.85 N/mm² for a composite (GP₃) having silica to alumina ratio of 2.7, which may be regarded as due to the stoichiometric ratio of silica and alumina in the geopolymer while the lower compressive strength for other composites may be attributed to the fact that surplus silica may disrupts the bond formation in geopolymer.

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