XPS Investigation of Geopolymer Binder Structure

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Four different activating solutions (Table 1) were used to activate the metakaolin particles and the samples were cured at 40ºC for 28 days at 100 percent humidity. The reaction of magnesium hydroxide and titanium oxide in the geopolymeric system was studied by adding 10 weight percent of the respective oxide/hydroxide into the mixed. The reaction between cement (cured for 1 month at 40C) and different types of geopolymer was also studied by obtaining spectra across the cement-geopolymer interface. The reason for this study was to investigate the type of reactions and the role of Ca cations in the geopolymeric system in comparison to the model system.

Table 1: Activating Solutions used for different systems.

	NaOH conc. (M)	Silicate conc. (M)
System A	8	0
System B	8	1
System C	5	0
System D	5	1

For the system with added magnesium hydroxide, the results showed that the magnesium hydroxide reaction in the system is dependent on the activating solutions, and for System A, the change in concentrations of different Si2p(nAl) (n=0,1,2,3 and 4) species possibly indicates that certain amount of Mg cations act as charge balancers in the geopolymeric gel whilst in System B, the magnesium hydroxide have formed M-S-H phases, and also reduces the degree of metakaolin dissolution. Whereas, for the titanium oxide system, the Si 2p and Al 2p peak binding energy increases, indicating a change in concentrations of different Si2p(nAl) (n=0,1,2,3 and 4) species. This possibly indicates that certain amount of titanium oxide particles have reacted for there is an increase in binding energy of the Si 2p and Al 2p peaks. The increase in binding energy was greater for the Si 2p peak compared to the Al 2p peak, indicating that the titanium oxide preferentially react with the leached silicate in the system compared to the aluminium. The degree of reaction of the metakaolin was not affected in system B as compared to the system with magnesium hydroxide. System C and D for both oxide/hydroxide show similar trends to system A and B.

For the geopolymer systems reacted with cement surfaces (cured at 40°C for 1 month), the interface of cement-geopolymer was studied by obtaining XPS spectra from the bulk of the geopolymer into the bulk of the cement by moving in increments of 100 um. For system A, from the bulk of the geopolymer to the cement interface, it shows an increase in the binding energy of the Si 2p and Al 2p peaks as well as an increase in the concentration of Ca cation with a decrease in Na concentration. The increase in Si 2p binding energy is an indication of the increase in Si2p (1Al) and Si2p (2Al) species. As it approaches the interface, there was a slight decrease in the Si 2p binding energy which could be attributed to numerous reasons and there were also two new Si 2p peaks. The peak at the lower binding energy is due to newly formed C-S-H phases from the dissolution of Ca cations into the geopolymeric system and the other, due to C-S-H depletion of Ca phases (cement) (Figure 1). This Ca depleted C-S-H phase decrease in Si 2p binding energy as the bulk of the cement was approach, indicating less Ca have been leached. Whereas, in the Al 2p peaks, there were clear peaks showing the formation of C-A-H phases at the interface and this phases decreases at the bulk of the cement. Similar trends were observed for the other systems but with slight differences in concentrations of different phases due to the difference in the starting activating solution.

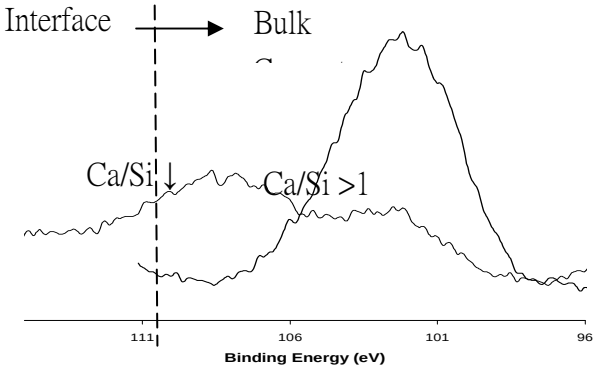

Figure 1. Si 2p XPS spectra for interface of the reaction between Geopolymer (System A) -Cement