

Characterization and Design of Alkali Activated Binder for Coating Application

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Geopolymer has good bonding structure of amorphous inorganic polymer which has excellent bonding agent properties and shows high bond strength in an early stage. Therefore, inorganic material like geopolymer which have advantages in previous research and has potential in replacing the organic materials in coating application. The chemical composition of the geopolymer materials which were metakaolin, ground granulate blast slag (GGBS) and fly ash contain mostly Silica (Si) and Aluminium (Al) which were possible source material for the manufacture of geopolymer coating. Based on Si:Al ratio and water:cement ratio were the important factor in the process of geopolymer coating which Si:Al of 3.5 and water:cement ratio of 0.25 gave the best result in strength in geopolymer coating. The common techniques for coating method that have been used were brush, spraying and dipping. From previous research, coating method influenced the greater result in fire proofing materials and high temperature. For compositions with Si:Al = 2.5 the adhesive strength was >3.5 MPa while for Si:Al=1 and 2 only weak adhesion to the metal substrates was achieved in metakaolin based geopolymer coating. Fly ash geopolymer coating has improved the compressive strength of the coatings materials as high as 40 MPa with the high sintering temperature, 1500°C. Thermal expansion measurements in metakaolin geopolymer coating of the Si:Al = 2.5 composition revealed expansion of up to 6% at 800 °C, while for Si:Al = 1 and Si:Al = 2 compositions up to 4% shrinkage was observed.

Keywords: Alkali Activated Binder, coating, geopolymer, XRF, XRD, SEM

The geopolymer chemistry concept was invented in 1979 [1] and the idea of geopolymer concept is an aluminium silicate inorganic polymer formed by geochemistry in 1991 [2]. Geopolymer possess excellent properties such as good acid resistance, alkali resistance, fire resistance and high temperature resistance [3-6]. Geopolymer cements are developing into the focus of increasing research efforts as the need to reduce global CO₂ emissions increase [7]. Geopolymerization is the process of dissolution of aluminium and silicon species from the surface of waste materials followed by the polymerization of active surface group and soluble species to form a gel and subsequently a hardened geopolymer structure [8]. Geopolymer source materials displaying excellent mechanical strength and resistance to attack by aggressive environments, these materials represent an opportunity to simultaneously improve both environmental and engineering performance compared to traditional technology [3, 9].

Coating is application to allow the lifespan of existing infrastructure or product. However, the existing coating does not fulfill the required performance needed in coating application such as appearance, adhesion, wet ability, corrosion resistance, wear resistance, scratch and also its thermal resistance. According to the previous research, coating application has been studied to advance

the performance in aircraft [10], marine [11-13], concrete [14], and any other materials that used coating materials. Throughout the years, a large number of studies have been conducted on the performance of organic polymers used as protective coating [15-17] but most of conventional organic polymers were harmful to both humans and the environment. The use of inorganic polymer coatings to replace the organic coatings looks like an alternative way of improving the durability of marine concrete structures. Geopolymer, known also as inorganic polymer or alkali activated binder [3, 18-24], has expanded international interests and its high anticorrosion makes it a new coating material.

Characteristics of Various Geopolymer Raw Materials X-ray Fluorescence (XRF)

Previous research has studied about the characterization of the raw materials that use in geopolymer coating. Table 1 shows the chemical composition of the fly ash [25], metakaolin [26], and ground granulate blast slag (GGBS) [27], has been studied using X-ray Fluorescence (XRF). Based on the chemical composition, these previous raw materials that have been used in geopolymer coating contain high content in Silica (Si), Alumina (Al), Calcium (Ca) and Ferum (Fe). Material containing mostly Silica (Si) and Aluminium (Al) in amorphous form is a possible source

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Component	Source Materials		
	Fly Ash [23]	Metakaolin [24]	GGBS [25]
SiO ₂	26.4	43.36	34.39
Al ₂ O ₃	9.25	37.6	14.47
CaO	21.6	0.2	41.67
Fe ₂ O ₃	30.13	0.94	0.63
P ₂ O ₅	0.67	-	-
SO ₃	1.3	0.07	-
TiO ₂	3.07	0.8	0.53
K ₂ O	2.58	0.22	0.36
MgO	-	0.24	6.49
Loss on Ignition	4.34	13.5	-

Table 1
CHEMICAL COMPOSITION OF THE SOURCE MATERIALS OF GEOPOLYMER COATING.

material for the manufacture of geopolymer. All the characteristics and performance of geopolymer materials are good enough to be applied coating materials. Previous research proved for the fire resistance geopolymer panels and metal coatings were prepared by using metakaolin [26] and ground granulate blast slag (GGBS) [27] as the main raw materials.

X-ray Diffraction (XRD)

Previous research has studied the characterization of raw material calcined kaolin that has been used in coating application by using X-ray Diffraction (XRD) or the so called peak ratio method [28]. Figure 1 displays the XRD pattern of calcined kaolin. Calcined kaolin commonly showed amorphous to semi-crystalline pattern with halo at 2θ between 15 and 30° due to presence of amorphous silica. Kaolinite peaks at 2θ values of 19.8, 24.2, 35.0, 39.2 and 45.4° were observed in the XRD diffractogram. This inferred that the thermal treatment of calcined kaolin was not complete. Quartz phases could be detected at 2θ values of 21.2°, 26.9° and 50.3° whereas the alunite peak could be found at 2θ value of 17.8° [28]. Previous research has found that calcined kaolin is not only a simple mixture of silica and alumina but retains some long-range order due to the stacking of the hexagonal layers [29]. After calcination, kaolin was transformed into calcined kaolin, which shows semi-crystalline to amorphous pattern. Most of the diffraction peaks of kaolinite disappeared [30].

Scanning Electron Microscope (SEM)

In the previous research, A.M. Mustafa has studied the microstructure of the fly ash before being activated

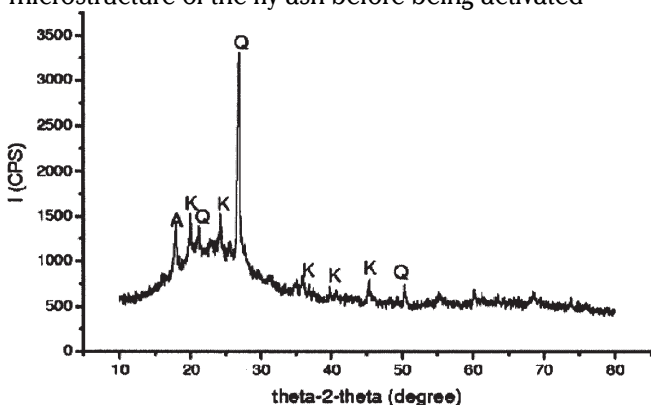


Fig. 1. XRD patterns of calcined kaolin (K=Kaolinite; Q=Quartz and A=Alunite) [28]

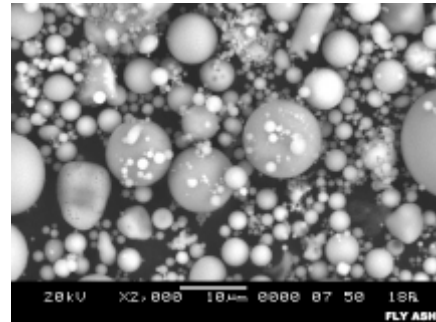


Fig. 2. SEM micrographs of the raw material fly ash [31]

with the alkaline activator to produce geopolymer fly ash [31]. Scanning Electron Microscope (SEM) micrographs is conducted to observe the microstructure of the raw material fly ash. Figure 2 shows the microstructures of the fly ash that consists of spherical particles of different sizes and mostly of hollow micro spherical particles with the appearance of bright crystals. Based on the previous research, fly ash suitable for use in geopolymer consists mostly of glassy, hollow, spherical particles, which are thin walled hollow spheres [32].

Processing of Geopolymer Coating

Mix design

In coating application, geopolymer source material has used as aluminosilicate source to produce geopolymer coating material by using sodium hydroxide and sodium silicate as alkaline activator [26, 33-35]. Table 2 showed the summary composition of metakaolin geopolymer coating mixtures [26]. Temuujin J. mentioned in producing of geopolymer coating, industrially available sodium silicate shows better result in thermal and water resistant than prepared from synthetically prepared sodium silicate [36]. Besides that, another research used sodium hydroxide and distilled water as alkaline activator and adding with polypropylene (PP) fibre and MgO as additive agent have proved that this combination is good in permeability, excellent anticorrosion and efficient bonding [11]. Previous research has studied the epoxy resin as hardener in coating production [37-39] and Astruc et. al. have proved that the synthesised of epoxy resin has improved the result in microhardness [40]. Another research that used the combination of 90% of metakaolin, 10% ground granulate blast slag (GGBS), polypropylene (PP) fibre and alkaline activator have showed excellent results in anticorrosion property of geopolymer in sea water [12]. In addition, by

Table 2
COMPOSITION OF METAKAOLIN GEOPOLYMER COATING MIXTURES [26]

Samples	Sodium silicate, w%	Metakaolin, w%	Sodium hydroxide pallet, w%	Water, w%
MK 1	70.24	28.43	0.89	0.42
MK 2	67.64	27.38	0.86	4.09
MK 3	64.56	26.13	0.82	8.47

Mix Code	Si:Al	Na:Al	Starting Material	Water:cement
MK1	1	1	Metakaolin, sodium aluminate, NaOH, H ₂ O	0.61
MK2	2	1	Metakaolin, sodium silicate, NaOH, H ₂ O	0.45
MK2.5	2.5	1	Metakaolin, cabosil, NaOH, H ₂ O	0.74

Table 3
COMPOSITION PRECURSOR
MATERIALS AND WATER:
CEMENT RATIO OF GEOPOLYMER
COATINGS [26]

adding polypropylene (PP) fiber and MgO expansion agent in the process of geopolymer coating, the large shrinkage can be reduced and ensure the curing at an early age [33].

Furthermore, in previous research Si:Al ratio and water:cement ratio were the important factor in the process of geopolymer coating which Si:Al of 3.5 and water:cement ratio of 0.25 gave the best results in strength [34]. Previous research reported that by using geopolymer composites, it has strengthened geopolymer coating in transportation infrastructure [14, 41]. Celik *et al.* have studied on corrosion rate which strongly depend on the porosity and thickness of the coating [42]. Besides that, there were two significant factors affecting the performance of geopolymer coating in marine concrete that have been studied by Zhang *et al.* which had humidity at tidal area and thickness of the coating layer greater than 5mm. These studies have proved that by considering these two factors, the performance of geopolymer coating has suitable in setting time, high bonding strength and excellent anti-corrosion [13].

Mixing process

Based on the previous research, fly ash geopolymer coating paste was prepared by mixing with alkaline activator solution. The combination of 12 M concentration of sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃) is prepared before it was to be mixed with the fly ash [25]. The ratio of fly ash/alkaline activator to NaOH/ Na₂SiO₃ was used at 2.5 for all mixtures. After the alkaline solution was added to fly ash powder, the mixture forming a slurry paste was applied to ceramic substrates by dipping method. The coating thickness varied between 0.3 – 0.5 mm and ceramic plates were placed in a plastic bag and cured at 70°C for 24 h before sintered [25].

According to the preparation of metakaolin geopolymer coating, previous research used the calcined kaolin at 750 °C for 24 h and mixing with alkaline solution [26]. The mix proportion of three sample were contributed as MK1, MK2 and MK2.5 varying to the Si:Al ratio. The alkaline activator was prepared from sodium hydroxide solution and adding sodium aluminate in MK1 and sodium silicate in MK2, followed by addition of the respective amounts of the metakaolin for the geopolymer preparation. For MK2.5 cabosil was dissolved in sodium hydroxide and left to age in a closed plastic container for 1 day before adding metakaolin. All three metakaolin based compositions were homogenised by using a high speed Thinky mixer for 5 min followed by de-foaming for 30 s. Typical coating thickness achieved using the dipping method was 0.3–0.8 mm to coat metal substrates. Composition of the geopolymer coatings and precursor materials are summarized in table 3.

Coating method

In coating application, the common techniques that have been used were brush, spraying and dipping. Based on Mustafa *et al.* [25], fly ash geopolymer coating was

prepared by mixing aluminosilicate with the alkaline activator solution of 12 M sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃). Then, by using dipping method, the slurry paste was applied to the ceramic substrates. The thickness of the coating is between 0.3-0.5 mm. The dipping process had influenced the thickness of the coating and directly influenced the greater result in fire proofing materials that suitable for high temperature application [6, 16]. Temuujin J. *et al.* (2012) used spray gun with 0.5 mm diameter nozzle, which resulted in thin coating of 0.5-0.7 mm in spray-coated [43]. Coating method such as spray coating is preferable to use and has a strong uniform coating which can be applied to a steel substrate. The thermal properties of spray-coated fly ash geopolymer coating strongly depend on the internal structure of geopolymers. The water: cement mass ratio is very important when applying spray-coated because increasing water contents of the spray-coated composition decreased its geopolymerization reaction rate [17].

Mechanical properties

Adhesion Strength

Previous research has studied on the metakaolin based geopolymer coatings on both stainless and mild steel substrates with different proportion on Si:Al ratio. Samples were labelled as MK1, MK2 and MK2.5. Adhesion of the geopolymer coating strongly depended on composition. MK2.5 showed strong adhesion behaviour with values recorded as >3.5 MPa for both stainless and mild steel substrates as the adhesion was beyond the capability of the Elcometer 106. Even though, Latella *et al.* (2006) showed that metakaolin based geopolymer with Si:Al = 2 and Na:Al = 1 is suitable for strong adhesion on steel the present research indicates that for geopolymer with the same composition the adhesion to steel is weak [44]. This changeability may be due to differences in preparation of the geopolymer. Differences of the adhesion strength to the metal for different geopolymer compositions are clearly not related to surface roughness as all metal substrates were prepared in the same way. High shrinkage of MK2.5 is believed to be caused by the presence of a large amount of weakly bound water and may be one of the reasons for the strong adhesion of this sample to metal substrates. Table 4 shows compressive strength of the geopolymer composition and their adhesion strength to the metal substrates.

Flexural Strength

According to the research of fly ash geopolymer coating by Mustafa *et al.* [25], the mechanical properties were measured using flexural testing. The compressive strength of geopolymer significantly improved with increasing of sintering temperature [6]. Besides that, fly ash which is coated onto mild steel stated that the result of adhesive strength is 2.7 MPa while for stainless steel result is 0.25 MPa [18]. The presence of calcium in high amount may interfere with the polymerisation process and alter the

Mix Code	Adhesion strength (MPa)	Compressive Strength (MPa)
MK1	–	6.8 +/- 1.4
MK2	–	3.6 +/- 1.8
MK2.5	>3.5 for both mild and stainless steel	–

Table 4
COMPRESSIVE STRENGTH OF THE
GEOPOLYMERS AND THEIR ADHESION
STRENGTH TO THE METAL SUBSTRATES [26]

Temperature (°C)	Flexural strength at 7 days (MPa)
600	14.18
900	19.50
1200	28.99
1500	40.00

Table 5
RESULT OF FLEXURAL STRENGTH FLY ASH
GEOPOLYMER COATING [25]

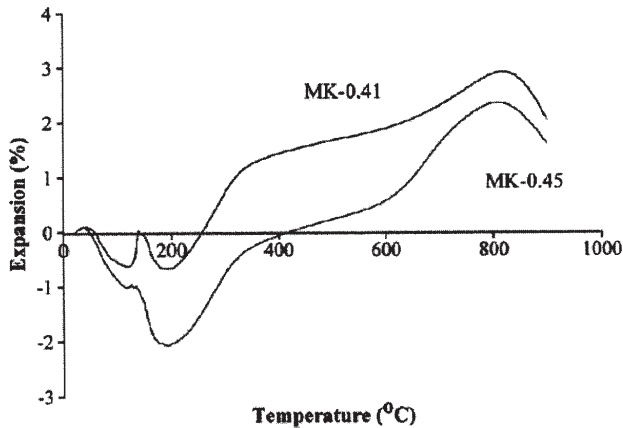


Fig. 3. Thermal expansion characteristics of geopolymer samples [36]

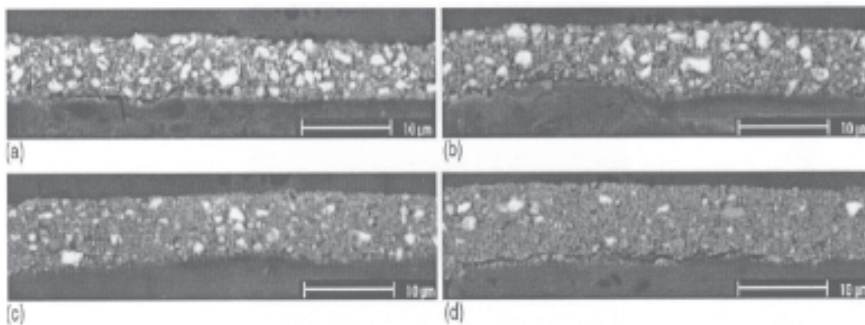


Fig. 4. SEM images of cross-sections of coatings with: (a) 20% kaolin, (b) 40% kaolin, (c) 60% kaolin and (d) 80% kaolin (15 g/m²) [45]

microstructure [27]. Due to these performances, fly ash is believed to be one of coating materials which has potential to form a solid surface which has high bond strength, long-term service life as coating materials of infrastructure and maintenance cost [45]. As an inorganic material, source material of geopolymer has a potential in fire resistant and protective coating due to its superior mechanical, chemical and thermal resistance properties [46]. Table 5 shows the result of flexural strength fly ash geopolymer coating.

Thermal Evaluation

Based on the past research, the thermal evaluation characteristics were obtained by using a DI-24 Adamel Lhomargy dilatometer to determine the thermal expansion behaviour of the of metakaolin geopolymer coating [36]. This research was carried out based on the different composition of water:cement ratio of the metakaolin geopolymer coating. Compositions were denoted as MK-n, where n is the water:cement ratio of the mixture. The measurements were conducted over the range of 20–900 °C with a heating rate of 5 °C/min. A pyros 56 standard was used to calibrate the measurements. The average of 3 measurements is presented as an expansion versus temperature curve. Thermal expansion curves of the MK-0.41 and MK-0.45 compositions are shown in figure 3. Both samples initially shrink then expand. Shrinkage occurring for both samples up to 200 °C is most probably related to dehydration of free or weakly bound water from the geopolymer. The higher shrinkage of the MK-0.45 sample is possibly caused by the presence of a larger amount of weakly bound water compared to MK-0.41. It is also apparent that both samples have almost identical expansion curves which differ only by the temperature at which expansion starts. The consistent and large expansion up to 800°C suggests an intumescent like

expansion. Finally above 800 °C the sample starts to shrink and this is ascribed to a densification of the structure possibly from sintering and viscous flow. Maintaining structural integrity is an important and essential requirement of the thermal coatings. Although the dilatometry curves show an initial shrinkage of the samples with increase in temperature, when coated on steel the geopolymer is obviously sufficiently flexible to cater for the mismatch between itself and the steel.

Morphology Study

The morphology of cross-sectional characterizations of coating with different kaolin content was carried out by using Scanning Electron Microscope (SEM) and is shown in the figure 4 (a) – (d) [47]. SEM images described the cross-sections of coating from low kaolin content to the high kaolin content and visualize the effect of calcium carbonate particles on the orientation of kaolin particles. The white particle in the gray-scale images shows the calcium carbonate particle while kaolin particles appear gray and the pores and the binder area black. According to the figure 4, the lower kaolin content presented higher white particle which was calcium carbonate because the calcium has higher atomic number than the other elements present in the coating. This research proved that when the kaolin content was increased, the porosity of the coatings has decreased [47].

Conclusions

As conclusion, geopolymer source materials like metakaolin, GGBS and fly ash are good potential to use as geopolymer coating materials. However, there are other source materials of geopolymer such as white clay, kaolin, and silica sand that can be used as geopolymer coating since the chemical composition and the performance among geopolymer source materials the quite is same.

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