Relationship between morphology analysis and durability of geopolymer paste

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Abstract. Geopolymer may improve corrosion protection by slowing the corrosion of steel in concrete or paste. When the alkalinity of the paste is reduced, the passivating layer is destroyed. If the paste is more porous, it can be easily absorbed by NaCl solution or moisture. The improved properties of the geopolymer paste increase its durability by preventing corrosive elements from harming the steel contained within the geopolymer paste. Two types of paste were used in this research which geopolymer paste and Ordinary Portland Cement (OPC) paste. A Scanning Electron Microscope (SEM) was used to determine the surface morphology of geopolymer paste and OPC paste. Based on the result of this investigation, the morphology characterisation is related to the concrete’s endurance. The formation of an aluminosilicate gel during the geopolymersisation process is critical for stability, increasing the geopolymer matrix’s density. As a result, this can increase the durability of geopolymer paste compared to OPC paste which geopolymer also can improve corrosion protection of steel in paste.

1 Introduction

Corrosion is an undesirable effect which could lead to the loss of billions of dollars in construction industry all over the world. A few research have been conducted to prevent the corrosion of steel bar by improving the quality of the concrete. The uses of geopolymer material to improve the quality of concrete have attracted and obtained great attention nowadays. Geopolymer material especially fly ash is used in construction industries because its ability to withstand corrosion for long-term performance. Fly ash has the advantage of reducing concrete permeability and mitigates against corrosion of reinforcement. Class F fly ash is highly pozzolanic material and contains almost 70% pozzolanic compounds such as silica oxide (SiO₂), alumina oxide (Al₂O₃) and iron (III) oxide (Fe₂O₃).

The Scanning Electron Microscope (SEM) is used to characterise the morphology of concrete, which is then connected to the compressive strength of the concrete. The durability of cement may appear to avoid the corrosive nature of steel in cement. Several approaches for characterising SEM imaging of porous materials have been documented, each with advantages and disadvantages. Both the pores’ structure and morphological characteristics are visible in the scanning electron microscope image. Zhang Qi et al. developed a set of related functions to characterise the SEM image of cement paste [1]. According to the literature, geopolymer materials made more compacted more tightly than sand-based material possesses superior mechanical qualities [2-5].

2 Methodology

2.1 Geopolymer Paste

During the sample preparation of the geopolymer paste the following steps were followed. An alkaline activator was prepared 24 hrs prior to use with the ratio of the mixture of Na₂SiO₃/NaOH is 2.5. The ratio is very important in order to obtain a homogeneous solution. The alkaline activator was later mixed with the fly ash for about 30 minutes. Fly ash was used as a source material in this study [6]. Then, the mixtures were placed in a 50 mm x 50 mm x 50 mm moulds. The samples were kept in the moulds until it becomes hardened. After 24 hrs, all the samples were taken out from the moulds. The samples requiring curing were cured at 60°C in the oven for 24 hrs, then the samples remained at ambient temperature until morphology testing at days 7, 14 and 28 [6].

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2.2 Ordinary Portland Cement (OPC) Paste

OPC containing water at an optimum ratio of 2:1 is prepared. It is placed in a mold compacted and stored in a mold at ambient temperature. The OPC sample is then removed from the mold after 24 hours and cured in water for 24 hours as well. The sample is left at ambient temperature for 7, 14 and 28 days before the morphology test is performed.

2.3 Morphology Analysis

The morphology of the fly ash and geopolymer images were obtained using a scanning electron microscope SEM (JSM-6460LA, JEOL, Japan) utilising secondary electron detectors. All specimens were coated with palladium using Auto Fine Coater (JFC-1600, JEOL, Japan) before carried out the morphology analysis by SEM. At the same time, the surface elemental composition was carried out by Energy Dispersive Spectroscopy (EDS) using the same procedure.

3 Result & Discussion

In general, geopolymer morphology is more compacted, and the majority of the fly ash combines with an alkaline activator to produce high compressive strength, which is a desirable property. The differences between fly ash and OPC are illustrated in Fig 1. In addition to having spherical shapes, the fly ash particles come in various sizes, including micrometres (Fig 1a). It retains the characteristic shape of the original fly ash, a fine solid spherical particle close in size to microspheres. Fly ash in its natural state is composed of a series of spherical vitreous particles of varying diameters. Naturally, the precipitator-type fly ash particles are spherical. Although most of these spheres are hollow, some of them may hold additional particles of a smaller size in their interiors, which is unusual. Because of these characteristics, when the mixing procedure is done properly, the flying ash particles’ fill in the space between them and are organised closely and densely. Thus, porosity can be minimised to the greatest extent possible.

Conversely, OPC particles are distinguished by their rough and irregular surface characteristics as well as their non-uniform and sharp corners (Fig 1(b)). Particles created by this form of OPC cannot be arranged closely and densely. The pores become more open, and moisture can more easily penetrate the concrete, as seen in Fig 2(a). Fig 2(b) shows SEM images of geopolymer and OPC paste that have been cured for 7 days. It is highlighted that there is a clear distinction. The size of fly ash particles appears to be larger, but certain OPC particles are unmistakably smaller and finer. When comparing with OPC paste, has a few pores, unreacted OPC grain for day 7. Due to the corrosive chemicals present, these pores will readily permeate the steel reinforcement.

![Fig. 1. SEM image of (a) raw fly ash and (b) OPC](image-url)
Fig. 2. SEM image of (a) Geopolymer paste and (b) OPC paste at 7 days.

Fig 3(a) from day 14 shows that the reaction at the geopolymer paste was almost complete. A small amount of fly ash has reacted with an alkaline activator (NaOH solution and Na$_2$SiO$_3$ solution), resulting in a strong alkaline reaction. A lower amount of aluminosilicate gel is produced when fly ash was activated with a mixture of NaOH and Na$_2$SiO$_3$ solution, with presence of some pores. When fly ash activated with NaOH, a hydroxy-sodalite is formed with quartz and mullite phases from unreacted fly ash. Then, the content of Na$_2$O and the SiO$_2$/Na$_2$O ratio of the activator has a profound effect on phases formed during activation and that reflected on the mechanical properties of alkali activated materials [7]. Hence, it will also affect the physical properties of geopolymer paste. In contrast to OPC, Fig 3(b) demonstrates that there were still many pores that were unreacted. In this way, corrosion in reinforced OPC concrete structures occurs in hostile environments due to the early deterioration process and has an adverse effect on structural durability.

Fig. 3. SEM image for (a) Geopolymer paste and (b) OPC paste at 14 days.

On day 28, there were a few minor cracks on the OPC compared to the geopolymer paste, as illustrated in Fig 4(a). The reactions of the paste have already taken place. However, the microstructure of the paste is still changing. According to the results, the geopolimerization process in geopolymer paste performed much better than the hydration process in OPC paste. As an added benefit, less unreacted fly ash results in higher compressive strength and denser geopolymer paste. Less alkaline activator leads to less geopolymer paste, which is rapidly converted into the hardening of paste as the amount of alkaline activator is decreased.
The OPC demonstrates that the cement particles progressively disintegrate and that a porous shell of hydration products forms around the particles. In addition, the porous nature of the paste considerably promotes the transportation and spread of aggressive mediums, such as chloride, into and through the paste from the surrounding environment. Furthermore, the content of portlandite Ca(OH)$_2$ in OPC paste is higher, whereas the content of ettringite is lower. As a result, cement paste’s mechanical characteristics and durability may be compromised because calcium chloride may react with the aluminates in cement to generate insoluble reaction products. The formation of this material occurs at a slower rate than the formation of ettringite. Following the appearance of ettringite, the chloride aluminate complex is generated. It has the potential to avoid future sulphate reactions with the residual aluminates in cement if appropriately used. It is owing to the fact that the hardened cement paste has a different microstructure and composition than the fresh cement paste, as illustrated in Fig 5.

In contrast to the hydration process by OPC, the polymerisation process results in the hardness of the material. The geopolymerization process will go through a mechanism in which a portion of the fly ash combines with the alkaline solution and produces a new geopolymer structure in the solution. The gel formed during this polymerisation process is called colloid and comes in a variety of sizes. Compared to OPC paste, the hardening fly ash geopolymer is heterogeneous, with some fly ash acting as an alkali activator throughout the gelation process.
Fig. 6. (a) SEM image of Geopolymer paste, (b) EDX analysis of the selected area in the geopolymer paste at 28 days. 

Fig 6(a) depicts the major constituents of the geopolymer, which are sodium, silicon, aluminium, and oxygen (b). In addition to the main elements (Na, Al, O, Si) present in the geopolymer binders, the elements of Ca and Fe are also present in the geopolymer. The Si/Al ratio is near in the gel matrix of the chosen region, which alters the structure of the polysialate crystals. The reason for this is that fly ash is a pozzolanic material. The active ingredients contained within it, SiO$_2$ and Al$_2$O$_3$, react to the alkaline solution to significantly reduce the porosity of the concrete and change the pore structure. Compared to OPC, the hydration products of cement and lime produce hydrated calcium silicate and hydrated calcium aluminate, which further react with gypsum to produce hydrated calcium alumina [3]. There is a linear relationship between sodium hydroxide concentration and heat generation; more acids are in the same volume as the acid concentration increases. Thus, more heat energy is required to react to the acids in the same volume as the acid concentration increases [3].

4 Conclusion

The major advantage of geopolymer paste is its excellent early strength and corrosion resistance. Geopolymers may improve corrosion protection by slowing the corrosion of steel in concrete. Geopolymers with a pH 12 to 13 have an alkaline environment that protects steel-reinforced surfaces by creating a passivation layer. When the alkalinity of the concrete is decreased, or the chloride content in the concrete reaches a particular level, the passivating layer is destroyed. The development of microstructure during the geopolymerization process, which results in producing an aluminosilicate gel, is crucial. The geopolymer’s stability and high compressive strength indicate that the geopolymer matrix is dense, implying that permeability was limited, increasing paste durability.

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