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INVESTIGATION OF CORROSION BEHAVIOUR OF MILD STEEL EMBEDDED IN GEOPOLYMER PASTE WITH CURING AND NON-CURING PROCESS

This research was conducted to examine the corrosion behaviour of mild steel bar embedded in geopolymer paste based fly ash Class F during curing and non-curing process. The geopolymer paste was fabricated by blending in the fly ash with alkaline activators (NaOH solution with molarity of 12 M, 2.5 ratio of solution Na2SiO3/NaOH). The paste was produced in 50 mm × 50 mm × 50 mm mould where the mild steel bar of 100 mm (length) × 12 mm (diameter) was embedded at the center of geopolymer paste. This is to comprehend the corrosion behaviour of mild steel embedded in geopolymer paste with and without curing process. Process of curing is carried out for 24 hours at a temperature of 60°C in oven. While on the contrary, the non-curing process will only be leave at room temperature. Both samples were tested after 28 days of curing to determine the corrosion behaviour, phase analysis and morphology analysis. In accordance with the morphology analysis, it shows that the fly ash was totally reacted with alkaline solutions in curing geopolymer paste sample while the non-curing geopolymer paste has shown the unreacted fly ash with high number of pores. The phase analysis of mild steel embedded in this geopolymer paste during curing and without curing process has proven that the presence of new crystallographic peak which also known as passive layer occurred. The potential values result by OCP testing shows the curing sample has highest potential values as compared to the non-curing sample ones.

Keywords: Corrosion behaviour; curing and non-curing; geopolymer paste; mild steel; passivity region

1. Introduction

Corrosion of steel or degradation process embedded in concrete structures is one of the major problems which effect such structures leading to their premature loss of serviceability, exorbitant cost for renovating or repairing, and worse, could affect structural collapse [1]. Therefore, in recent years issues regarding the corrosion behaviour has attracted attention to the industry key players specifically in structural of concrete structures. A good durability requirements of mild steel bar are not always possibly to achieve due deterioration in reinforced concrete structures cause by corrosion. This is also in agreement with Dhawan et al. [2] in which reported that one of the main contributions to durability problems in structural concrete is corrosion in mild steel bar.

The Australasian Corrosion Association (ACA) also stated that deterioration of the mild steel bar in concrete is a worldwide and major problem that causes a range of utilisation, economic and aesthetic issues. However, buildings can be built to last and protected for as long as possible if the design phase and right decision regarding the corrosion effects are considered at the first place before the construction start. Therefore, mild steel bar surface should form a passive film that could only be possible in the alkaline conditions (high in pH) in order to prevent or minimize the corrosion effects in the concrete structures [3].

Hence, geopolymer is being introduced as an alternative to Ordinary Portland Cement (OPC) that acts as a binder in building materials [4-8]. Geopolymer is inorganic materials that has similar chemical composition to zeolite. However, it possesses an amorphous and ceramic-like structure and properties which in favour for this project [9]. Furthermore, geopolymer was also preferred due to its advantages such as being environmentally friendly, high alkalinity environment, has excellent mechanical properties, good physical properties and has good corrosion resistance [10-12]. The geopolymer paste as a coating material become a greatest solution to reduce corrosion by preservation

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the mild steel bar from the corrosive agents and increased the strength of the materials with a low corrosion rate [1].

Curing is necessary to get the optimal results of geopolymer as geopolymer will undergo polymerization reaction during curing. Andi Arham et al. reported that 30°C to 90°C was the best range of curing temperature to assure the geopolymeric reaction will occur efficiently [13]. The effect of strength after curing have been reported elsewhere [14,15]. The temperature and duration of heat curing plays an important role for the strength development of fly ash based geopolymer paste [13].

This paper presents the corrosion behaviour of mild steel embedded in geopolymer paste during curing and without curing process. Apart from highlighting the corrosion behaviour of mild steel embedded in geopolymer paste, the morphology and phase analysis has also been examined for a better understanding purpose.

2. Experimental

2.1. Materials and sample preparation

In the experimental work, mild steel bars of 100 mm (length) × 12 mm (diameter) were used. These steel bars are firstly being cleaned to remove the contaminations on the surface. As for the geopolymer paste, fly ash Class F was prepared by mixing with alkaline activators (12 M molarity of sodium hydroxide (NaOH) solution and sodium silicate (Na_2SiO_3) solution). The ratio of Na_2SiO_3/NaOH solutions is 2.5. Then, the mixture was poured into the mould of 50 mm × 50 mm × 50 mm and mild steel bars were embedded in the middle of the geopolymer paste until the concrete harden. After being demolded, samples will undergo a curing and non-curing process. The curing process is conducted at 60°C in the oven for 24 hours while the non-curing specimens will be kept at room temperature. All the sample were tested after 28 days of curing.

2.2. Corrosion behaviour characterization

The corrosion behaviour testing by Open Circuit Potential (OCP) was performed in accordance to ASTM C876-09 [16,17] to determine the potential values of the mild steel bar in the absence of any current or potential suppliers. A multimeter that connected to the reference electrode (Saturated Calomel Electrode (SCE)) was used as negative connection, whereas the positive connection was linked to the mild steel bar (as a working electrode) that embedded in geopolymer paste as shown in Fig. 1. The OCP testing results are being elucidated using the Pourbaix diagram to determine the corrosion behaviour between mild steel bar and geopolymer paste. The Pourbaix diagram ofiron (Fe) at 25°C illustrates the E (standard hydrogen electronic potential) – pH (acidity) has been used to explain deterioration of metal. Referring to the Pourbaix diagram, there are three divided regions include of passivity, immunity and corrosion. Hence, the corrosion behaviour of mild steel bar was investigated and the Pourbaix diagram was used to interpret the thermodynamic tendencies for mild steel to deteriorate.

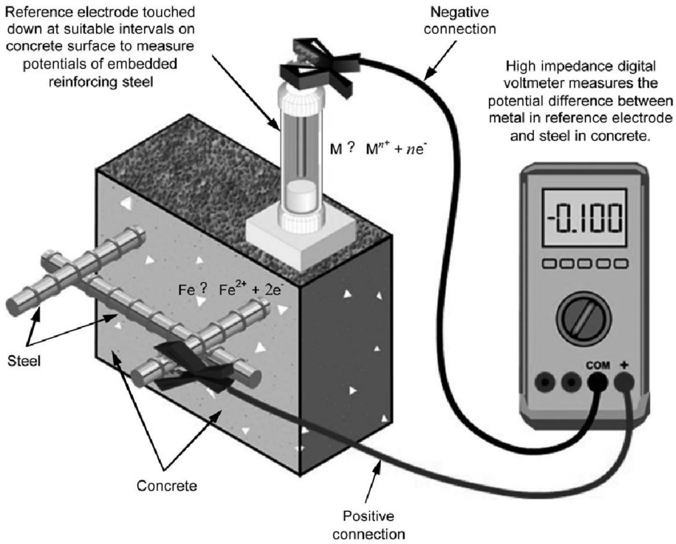

Fig. 1. Open Circuit Potential setup

2.3. Phase characterization

The curing and non-curing samples will undergo phase characterization using X-ray diffraction (D2 Phaser XRD Analyzer model) with voltage of 90-250 V under ambient temperature, concerning to analyse the presence of semi-crystalline and crystalline phases on the mild steel bar embedded in geopolymer. The scanning angle, 2ɵ with a scanning range of 20° to 90° was used during the phase characterisation. The XRD data analysis in this project was conducted qualitatively.

2.4. Morphology characterization

Scanning Electron Microscope (SEM) equipped with Energy Dispersive X-ray (EDX) (JEOL JSM-6010LV) were performed on the curing and non-curing samples for morphology characterisation of geopolymer paste based fly ash class F on the mild steel surface. There are two magnifications used in this work; ×1000 and ×2000.

3. Results and discussion

3.1. Corrosion behaviour analysis

Fig. 2 shows the potential values result of Open Circuit Potential (OCP) for curing and non-curing samples of mild steel bar embedded in geopolymer paste after 28 days. The potential value which undergoes curing sample was 0.105 V, which slightly higher than the potential value of non-curing sample which is 0.052 V. This is due to heat applied during the curing process that causes the sample to harden faster and more crystalline compared to non-curing sample. The workability and durability of the curing sample also improved hence the potential values became more positive.

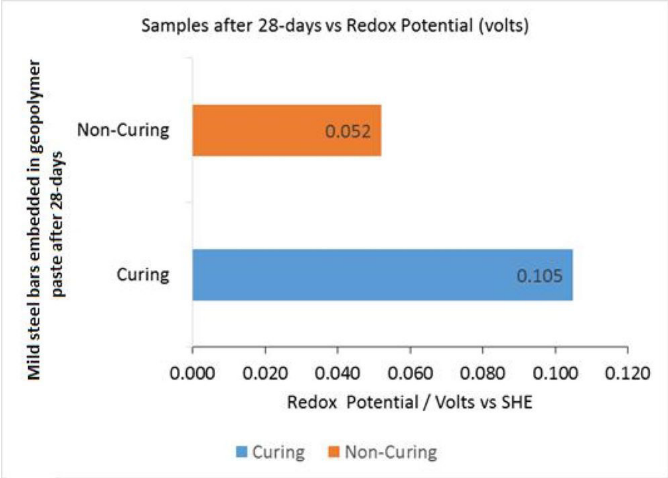

Fig. 2. Bar chart of curing and non-curing mild steel bar embedded in geopolymer paste after 28 days against redox potential (volts) by OCP testing

The potential values of curing and non-curing samples are 0.105 V and 0.052 V respectively. By referring to the Pourbaix diagram of iron (Fe) at 25°C as shown in Fig. 3, their potential values were in the passivity region at pH 12 for both samples. The pH value for this geopolymer paste is 11.5 to 12 as the concrete was always in an alkaline environment. In accordance with Davidovits [18], depending on the formulations of geopolymer paste, the pH shall be in the range of 11.5 to 12.5 which in agreement with this finding. In this passivity region, the iron reacts to form protective oxide films which results to a stable phase which is Fe_2O_3. This oxide acts as a protective film or passive layer in this region that has been expected to provide some protection against corrosion on the samples. The use of the fly ash based geopolymer had good corrosion resistance and it could preserve the mild steel bar in the geopolymer paste from corrosion.

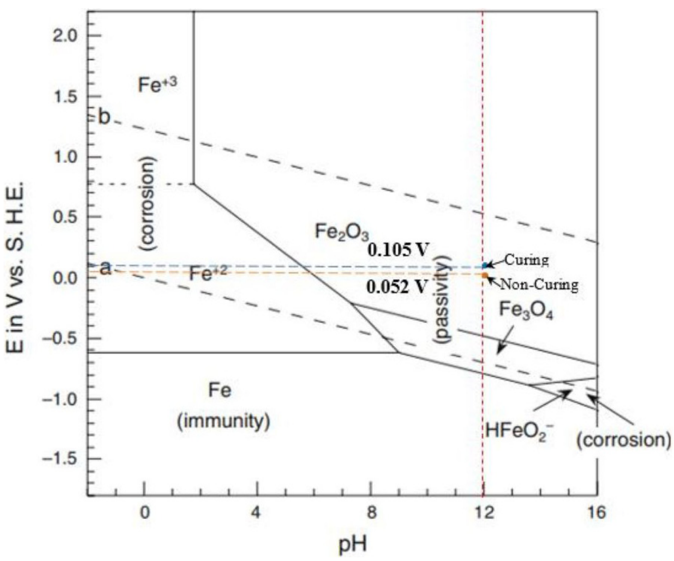

Fig. 3. Both samples located at passivity region

TABLE 1 shows an ASTM criterion for different standard reference electrodes for corrosion of steel in concrete. In this study, SCE was used as reference electrode and the potential values from this OCP testing were then convert to Standard Hydrogen Electrode (SHE) results interpretation in Pourbaix diagram. The potential values were in the low category range, between 0.052 V to 0.105 V which mean there is only 10% risk of corrosion will occur to the mild steel bar. This proved that the geopolymer paste had good corrosion protection on mild steel bar against the corrosion.

TABLE1

ASTM criteria for corrosion of steel in concrete for different standard reference electrodes

$Cu/CuSO_4$	Ag/AgCl	SHE	SCE	Corrosion condition
>–200 mV	>–100 mV	+120 mV	>–80 mV	Low (10% risk of corrosion)
–200 to –350 mV	–100 to –250 mV	+120 to –30 mV	–80 to –230 mV	Intermediate corrosion risk
<–350 mV	<–250 mV	<–30 mV	<–230 mV	High (>90% risk of corrosion)
<–500 mV	<–400 mV	<–180 mV	<–380 mV	Severe corrosion

3.2. Phase analysis

Fig. 4 represents the phase analysis of mild steel bar embedded in geopolymer paste which have go through a curing and non-curing processes after 28 days. Based on the XRD results, both XRD patterns of curing sample and non-curing sample show the existence of hematite (Fe_2O_3) and magnetite (Fe_3O_4) qualitatively. These two phases were found to be located at the passivity region in Pourbaix diagram (refer Fig. 3). The reaction between geopolymer paste and mild steel will form passive layer which are Fe_2O_3 and Fe_3O_4. This passive layer act to protect a mild steel from corrosion [12].

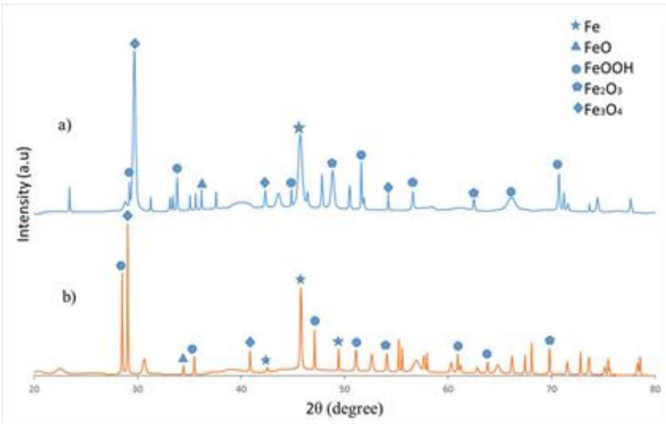

Fig. 4. XRD patterns of mild steel bar embedded in geopolymer paste that undergoes (a) curing and (b) non- curing process after 28 days

At the same time, there are also presence of other phases like iron oxide (FeO) and iron oxide hydroxide (FeOOH). Fe peaks were detected with lower intensity on curing of mild steel bar embedded in geopolymer paste sample as compared to non-curing ones. This is possible due to the iron (Fe) element in mild steel bar has transformed to hematite (Fe_2O_3) and magnetite (Fe_3O_4). Even though both samples were observed to have passive layer, non-curing sample tends to corrode in a short time. This is because of the geopolymer structure on non-curing sample which has high number of pores due to the unreacted fly ash with alkaline activators. This will affect the corrosion performance of the sample.

3.3. Morphology analysis

The morphology analysis was conducted using SEM for both curing and non-curing samples 28 days later as shown in Fig. 5 and Fig. 6. The SEM micrograph for curing sample shows highly homogeneous structure due to completed geopolymerization that contributed to complete reaction between fly ash particles and the alkaline activators, as shown in Fig. 5. As for the geopolymer paste with 70% of fly ash Class F and alkaline activators mixtures (NaOH solution with molarity of 12 M, solution of Na_2SiO_3/NaOH solution ratio of 2.5), the sample that undergoes curing process at 60°C for 24 hours will obtain a geopolymer paste with no presence of pores, higher impermeable properties and good corrosion resistance due to the good reaction between fly ash and alkaline activators achieved a high degree of geopolymerization after 28 days of cured.

Fig. 6 shows a SEM micrograph of non-curing sample fly ash particles that were not fully reacted to alkaline activators. This is because the sample was not exposed to the heat of curing and retained higher moisture content which may reduce corrosion resistance and lead to corrosion in a short time span for the mild steel bars compared to curing samples.

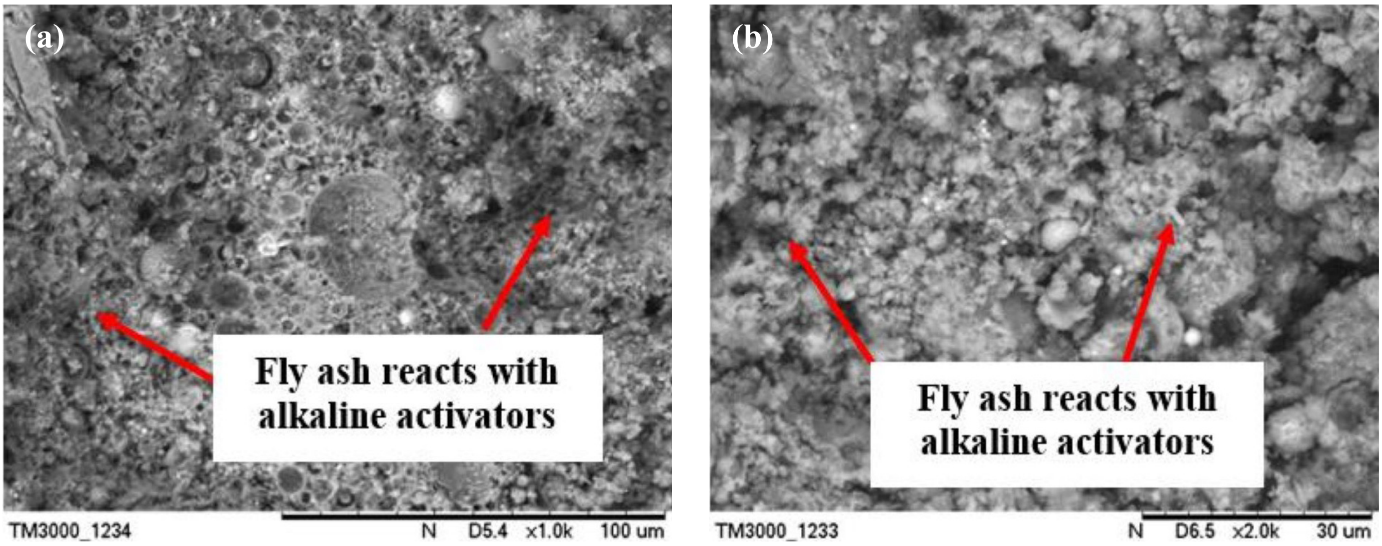

Fig. 5. SEM micrographs of curing geopolymer paste based fly ash after 28 days (a) ×1000 and (b) ×2000

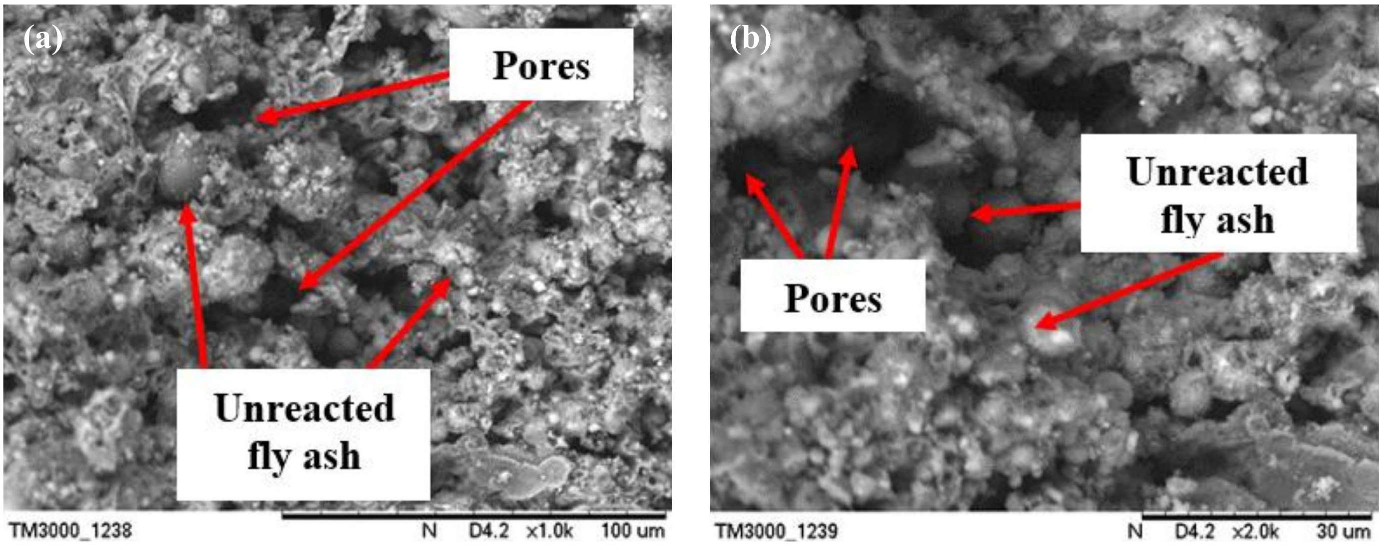

Fig. 6. SEM micrograph of non-curing geopolymer paste based fly ash after 28 days (a) ×1000 and (b) ×2000

4. Conclusion

The experimental investigation was done to attain the objectives of this research. The following conclusions were reported by referring to the experimental investigation:

1. The fly ash Class F mixing with alkaline activators which 12 M of NaOH solution with of Na_2SiO_3/ NaOH solution ratio is 2.5 and undergoes a curing process at 60°C for 24 hours will produce geopolymer paste with advantages of good corrosion resistance and undeniably, able to preserve the mild steel bar from corrode.
2. In accordance with the Pourbaix diagram of iron (Fe) at 25°C, both samples' potential values of curing and non-curing sample are 0.105 V and 0.052 V respectively. Those potential values were in the passivity region at pH 12.
3. A passive layer which are Fe_2O_3 and Fe_3O_4 have been formed as a result from the reaction between geopolymer paste and mild steel. The role of this passive layer is to protect a mild steel from corrosion. This has been proven by the phase analysis using XRD.
4. The sample that undergoes curing process at 60°C for 24 hours will obtain a geopolymer paste with no presence of pores, high impermeable properties and good corrosion resistance because the fly ash was well reacted with alkaline activators and achieved a high degree of geopolymerization after 28 days of cured. The non-curing samples exhibited a high number of pores in the geopolymer paste as the fly ash was unreacted to alkaline activators. This will later reduce the resistance to corrode and lead the corrosion to occur in a short time on mild steel bars.
5. It can be concluded that corrosion behaviour during curing and without curing process of mild steel embedded in geopolymer paste was successfully investigated. The results have proven by the analysis of its phase, morphology and corrosion behaviour 28 days later.

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