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Corrosion Studies of Fly Ash and Fly Ash-Slag Based Geopolymer

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Abstract. This paper presents the results of corrosion studies between Fly Ash Geopolymer (FG) paste and Fly Ash-Slag Geopolymer (FSG) paste. Geopolymer was made from aluminosilicate inorganic polymers mixed with the alkaline activator in order to reduce the carbon dioxide (CO₂) to the ecosystem. Samples then were cured at 60°C for 24 hours in the oven. Reinforcement bar is placed at the center of the paste. The samples were examined after 7, 14 and 28 days in terms of Open Circuit Potential (OCP) test, phase analysis and morphology analysis. The potential values regarding OCP test for FSG paste from 7 days until 28 days are 0.464 V, 0.474 V and 0.498 V more positive than FG paste which the potential values are 0.087 V, 0.133 V and 0.206 V respectively. From the Pourbaix diagram, all the potential values for FG paste and FSG paste were located in the same Fe₂O₃, passivity region. Passive layer which is the oxide form exists in this region to protect the reinforcement bar from corrosion agents. It can be proved from phase analysis results which iron oxide hydroxide (FeOOH), hematite (Fe₂O₃) and magnetite (Fe₃O₄) peaks exist. The differences of morphological structures of these pastes were observed by Scanning Electron Microscope (SEM). It shows that FSG paste had good corrosion resistance and low corrosion rate compared to FG paste.

1. Introduction

An early degradation of reinforced concrete structures is caused by corrosion of steel in aggressive environments. Steel is passive due to the alkalinity of concrete, which the oxide formed to protect the steel concrete. However, the passive film could damage if the reinforcement bar exposed to the active environments such as carbon, chlorine, and acid conditions [1].

A few studies have been conducted to prevent the corrosion of reinforcement bar by improving the quality of the concrete. The uses of polymer to improve quality of concrete have attracted and obtained great attention nowadays.

Thus, geopolymer concrete is used as an alternative way to replace Ordinary Portland Cement (OPC) concrete. Geopolymer have a few advantages such as light weight, good tensile strength, durability and high corrosion resistance [1]. Geopolymer is inorganic materials produced by geosynthesis from alumina-silica materials with an alkaline activators and their chains or networks of inorganic molecules linked by covalent bonds. The precursors that always used as main raw material in geopolymer are volcanic ash, zeolite, kaolin, fly ash and granulated blast furnace slag [2].



2. Materials and Method

2.1. Materials and Sample Preparations

Fly ash geopolymer (FG) paste in this study was made from a mixture of class F fly ash, sodium hydroxide (NaOH) solution and sodium silicate (Na_2SiO_3) solution. While fly ash-slag geopolymer (FSG) paste was made from a mixture of 70% fly ash, 30% slag, NaOH solution and Na_2SiO_3 solution. The American Society for Testing and Materials (ASTM) defines fly ash as a residue resulting from the combustion of coal in power plants [3].

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 (SiO_2), alumina oxide (Al_2O_3) and iron (III) oxide (Fe_2O_3) [4].

NaOH solution was prepared by dissolving the NaOH pellets in distilled water. Then, the NaOH solution was mixed with Na_2SiO_3 solution. An alkaline activator was prepared 24 hrs prior to use with the ratio of the mixture of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ 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.

The prepared mixtures were placed in moulds and compacted. After that, the reinforcement bar was embedded in the geopolymer paste. The samples were kept at ambient temperature in the moulds until it becomes hardened. All the samples were taken out from the moulds after 24 hrs. Then, the samples were cured at 60°C in the oven for 24 hrs [5-7].

2.2. Experimental Methodology

2.2.1. Open Circuit Potential (OCP). In this research, the OCP test set up as shown in figure 1. The Sanwa digital multimeter CD 771 was used to measure the potential difference between the steel in the concrete and the metal in the reference electrode.

The positive side was connected to the reinforcement bar embedded in the geopolymer paste while the negative side was connected to the reference electrode. Reference electrode that was used in this research is Saturated Calomel Electrode (SCE) because this electrode is suitable to measure potential value in laboratory scale [8]. The potential value readings were taken accordingly with the time set.

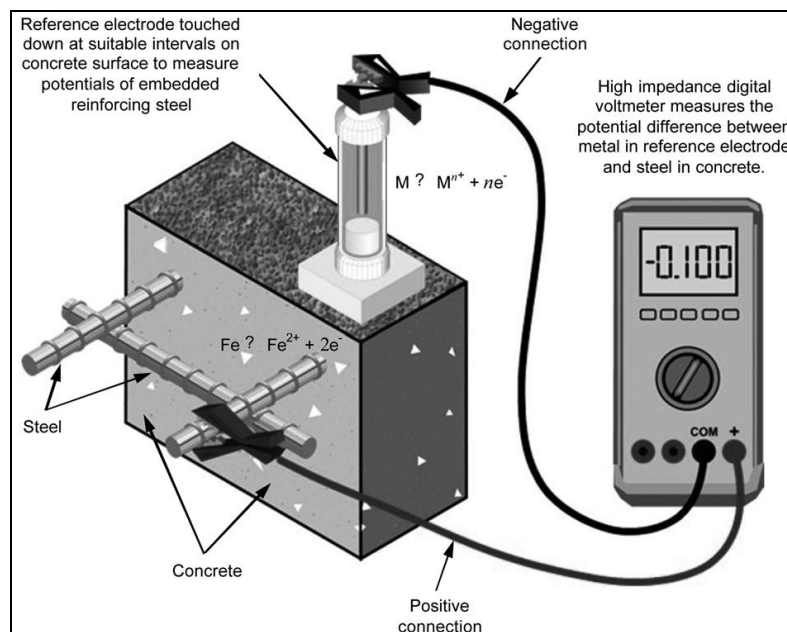


Figure 1. Reference Electrode Circuitry [8].

2.2.2. *Phase Analysis*. This test was set up to know the phase exist between reinforcement bar and geopolymer paste. The test was conducted by X-Ray Diffraction (XRD) method.

2.2.3. *Morphological Analysis*. This test was run to observe the fly ash geopolymer (FG) and fly ash-slag geopolymer (FSG) paste surface morphology and pore. Scanning Electron Microscope (SEM) was conducted by using JEOL machine JSM6460-LA.

3. Results and Discussions

3.1. Open Circuit Potential (OCP)

Figure 2 shows graph of time (day) against redox potential (volts) for open circuit potential (OCP) of reinforcement bar embedded in fly ash geopolymer (FG) and fly ash-slag geopolymer (FSG) paste at ambient temperature. From the graph, the FSG paste samples have higher potential values compared to the FG paste samples from day 7 until day 28.

The potential values of FSG paste samples were 0.464 V, 0.474 V and 0.498 V from day 7 until day 28 while FG paste samples potential values were 0.087 V, 0.133 V and 0.206 V respectively.

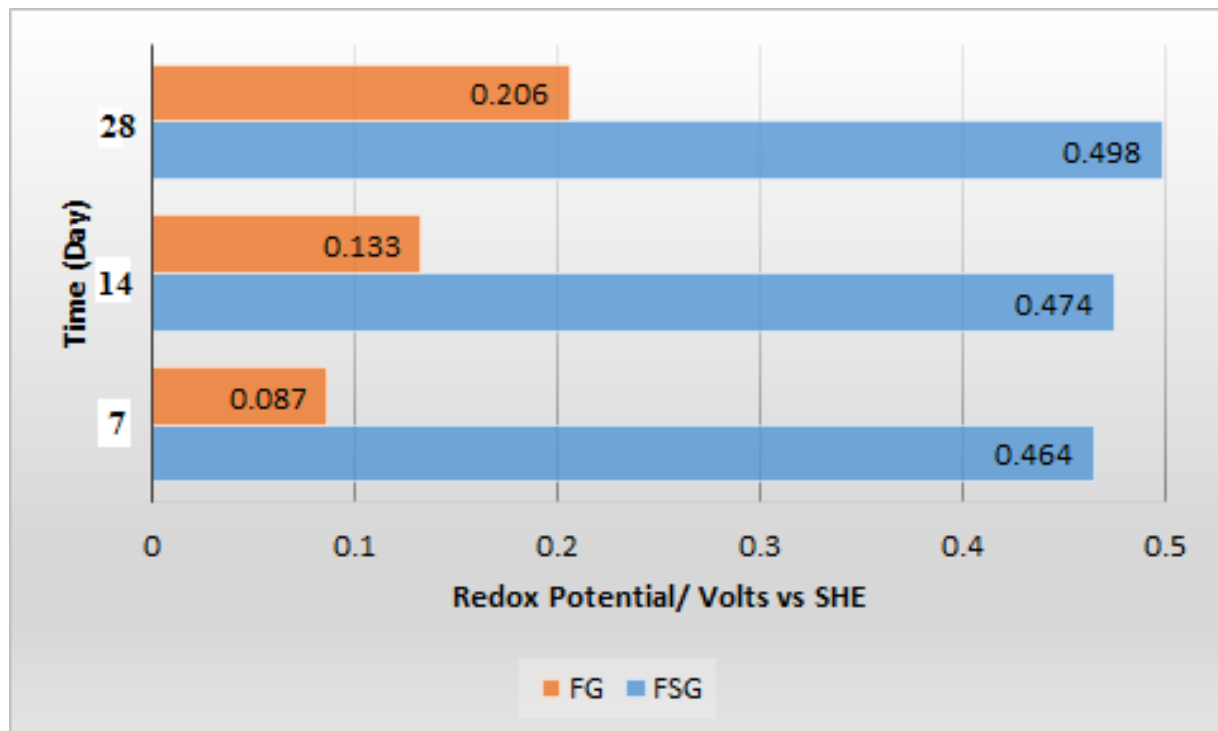


Figure 2. Graph of time (day) against redox potential (volts) for OCP of reinforcement bar embedded in FG and FSG paste at ambient temperature.

The potential values obtained from OCP test were interpreted in Pourbaix diagram to determine which region these value located. The pH value for these FG and FSG paste were observed by using the pH paper.

The pH value for these paste is 12 as the concrete were always being alkaline in nature [9].

Figure 3 shows potential-pH diagram or Pourbaix diagram for Fe-H₂O at 25°C. When referring the Pourbaix diagram at pH 12, all samples of FG and FSG paste were in the passivity region as labelled. The passive layer was formed to protect the reinforcement bar from corrosion in this region.

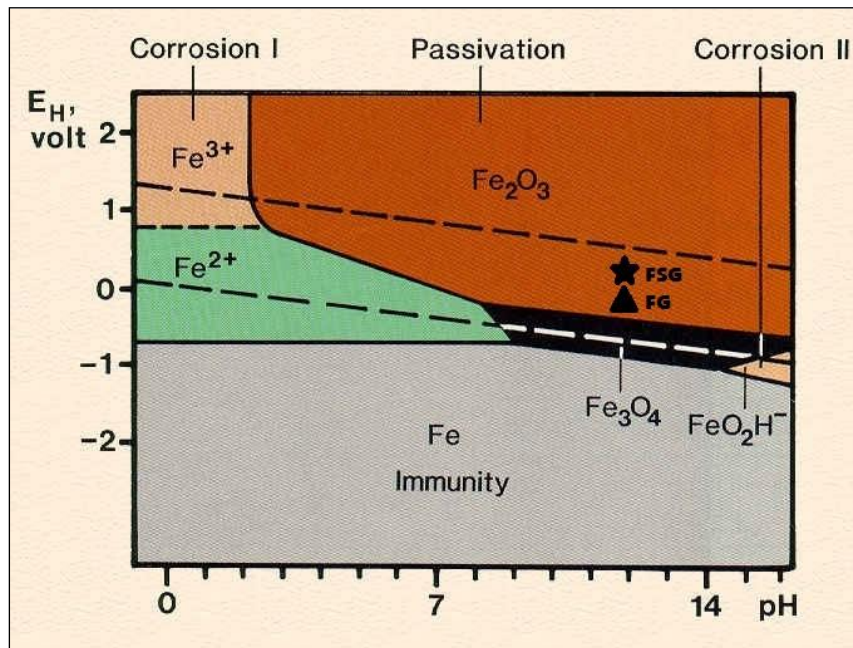


Figure 3. Pourbaix diagram for Fe-H₂O at 25°C.

3.2. Phase Analysis

Figure 4 (a) shows the X-Ray Diffraction (XRD) pattern for reinforcement bar and reinforcement bar embedded in fly ash geopolymer (FG) paste and figure 4 (b) was XRD pattern for fly ash-slag geopolymer (FSG) paste. Both figures shows that peak iron oxide hydroxide (FeOOH), hematite (Fe₂O₃) and magnetite (Fe₃O₄) were exist as stated in OCP analysis which mean in passive region.

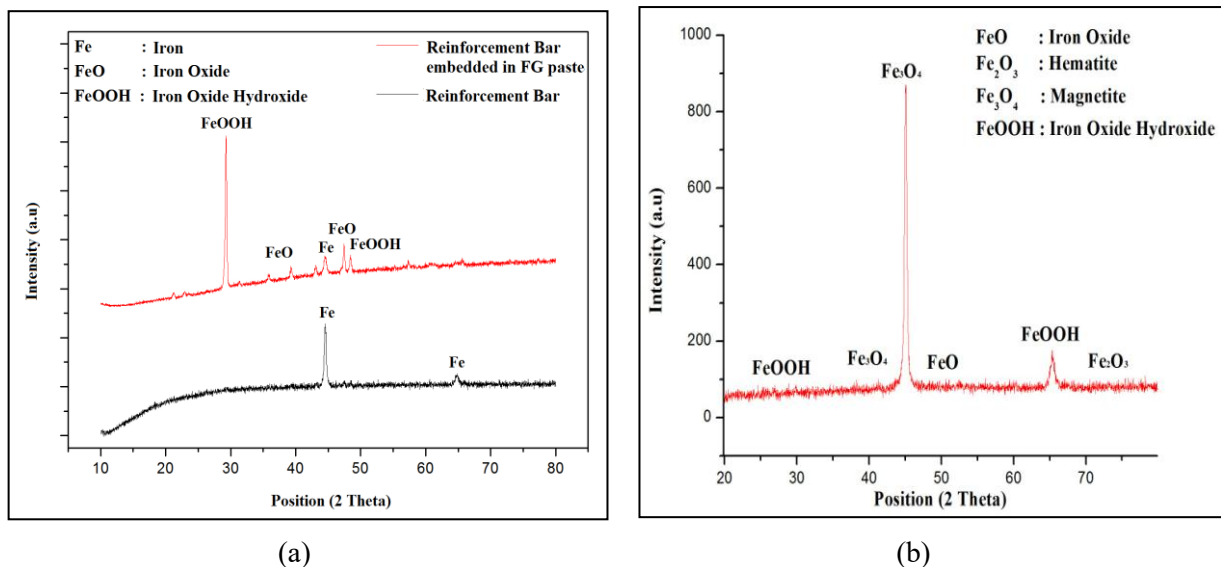


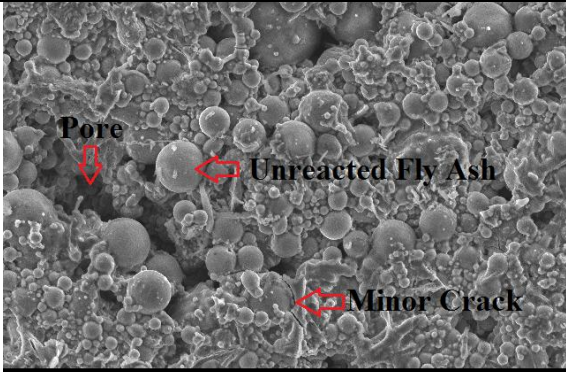

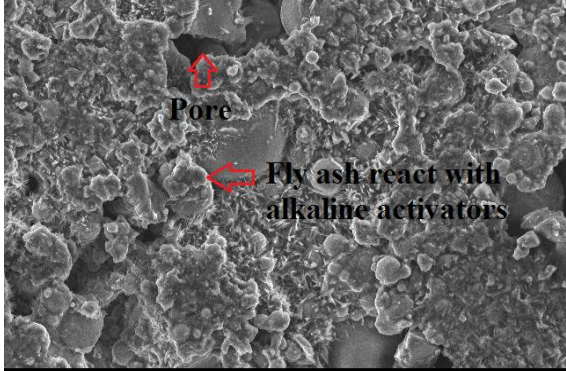

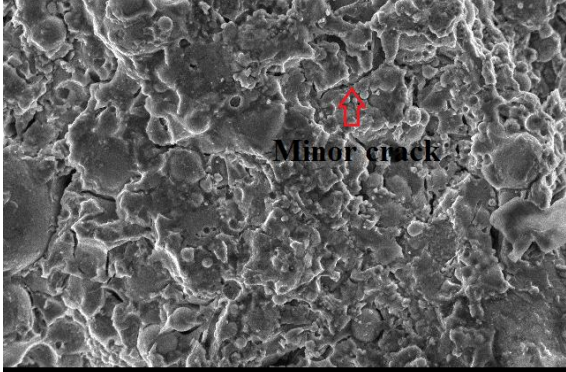

Figure 4. XRD pattern for (a) Reinforcement bar and reinforcement bar embedded in FG paste; (b) Reinforcement bar in FSG paste.

3.3. Morphology Analysis

Table 1 shows micrographics Scanning Electron Microscope (SEM) of fly ash geopolymer (FG) paste and fly ash-slag geopolymer (FSG) paste for day 7 until 28. For the FG paste in day 7, it shows a lot of

pore, unreacted fly ash and micro crack. It was determined that the existence of unreacted fly ash and microcracks had caused the increasing of pores and reduced strength [10]. The corrosive agents will easily penetrate to the reinforcement bar with these pores. When comparing with FSG paste for day 7, most of the fly ash and slag has already reacted with alkaline activators and only have a few pore. For day 14, the reaction at FSG paste was almost done, only have slightly unreacted slag. Different with FG, there were still many pores and unreacted fly ash. There were few minor cracks on FG and FSG paste on day 28. The reactions of FSG paste are already done while FG paste microstructure is still changing. It shows that the geopolymerization process occurred in FSG paste was better than FG paste.

Table 1. Micrographics SEM of FG and FSG paste for 7, 14 and 28 days.

Days	Fly Ash Geopolymer (FG) Paste	Fly Ash-Slag Geopolymer (FSG) Paste
7 Days	 <p>Pore Unreacted Fly Ash Minor Crack</p> <p>10kV X1,000 10µm 09 35 SEI</p>	 <p>Unreacted slag Pore Unreacted fly ash</p> <p>10kV X1,000 10µm 12 33 SEI</p>
14 Days	 <p>Pore Fly ash react with alkaline activators</p> <p>10kV X1,000 10µm 08 32 SEI</p>	 <p>Unreacted slag</p> <p>10kV X1,000 10µm 14 33 SEI</p>
28 Days	 <p>Minor crack</p> <p>10kV X1,000 10µm 08 30 SEI</p>	 <p>Minor crack</p> <p>10kV X1,000 10µm 09 34 SEI</p>

4. Conclusions

Based on this research, the following conclusions were drawn:

- Fly ash-slag geopolymer (FSG) paste had good corrosion resistance and low corrosion rate compared to fly ash geopolymer (FG) paste.
- From the Pourbaix diagram, all the potential values for FG paste and FSG paste were located in the same Fe_2O_3 , passivity region. Passive layer which is the oxide form exists in this region to protect the reinforcement bar from corrosion agents.

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Acknowledgments

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