

Corrosion of Steel Reinforcements in Fly Ash- and Kaolin-based Geopolymer Concrete Immersed in Distilled Water and ASTM Seawater

Astutiningsih, S.^{1*}, Rustandi, A.¹, and Noermalasari, D.²

Abstract: Corrosion behavior of steel bar in fly ash- and kaolin-based geopolymer concrete immersed in aggressive media of distilled water and ASTM seawater was compared to Portland cement concrete having similar mix design. An accelerated corrosion by applying 3 V potential on the steel bar was performed to obtain reasonable test results in a relatively short time. The potential and pH of the immersing media were measured from day 1 to day 10 and then plotted on Pourbaix diagram to predict passivation or corrosion state. At day 10, steel bar in Portland cement concrete were in corroded state both in distilled water and seawater. The best corrosion performance was for kaolin- based geopolymer concrete in which at day-10 the steel bar was passivated in both media. Steel bar in fly ash- based geopolymer concrete was passivated in distilled water but corroded in seawater.

Keywords: Corrosion potential, pH, geopolymer concrete, ASTM seawater, distilled water.

Introduction

Geopolymer is a new type of material introduced by Davidovits in the 1970s [1]. Study on geopolymer has been published since the end of 1990s. Geopolymer can be used to substitute ordinary Portland cement due to the similar mechanical and physical properties as the Portland cement. However, geopolymer has better chemical resistant than Portland cement due to the absence of calcium compounds [2]. The utilization of geopolymer as Portland cement substitute in concrete requires that geopolymer be compatible with the steel bars reinforcement in concrete.

Fly ash has been used as raw material or precursor for geopolymer cement. The utilization of fly ash makes geopolymer an environment friendly alternative, besides that the low temperature processing itself does not generate CO₂ emission. Fly ash is a residue from coal burning in power plants. Due to the high temperature exposure, fly ash is glassy and hence reactive to alkali. Alkaline silicate solutions are used to activate the precursors to form geopolymer. For that reason, geopolymer can be classified as alkali-activated cements.

Ordinary Portland cement concrete is known to protect steel reinforcement due to the alkalinity of the materials that encourage the formation of a passive adhering film protecting metals from corrosion [3]. This passive film can be formed in pH range of 12.5–13.8 [4], however the film may fail due to (1) the decomposition of CO₂ and SO₂ gases in the pore solutions which then decrease pH, and (2) chloride penetration which can break the film causing pitting corrosion of the metals [5]. Geopolymers is synthesized under a high alkaline environment which tends to be corrosive to metals, however after curing, geopolymer should be neutral. Geopolymerisation includes dissolution of the precursors into oligomers of tetrahedral alumina and silica, followed by polycondensation of the oligomers into a 3D network structure [1] which is different from the molecular structure of Calcium Silicate Hydrate in hardened Portland cement. The alkali metals in geopolymer structure serve as charge balancers for the tetrahedral alumina. If not all of the alkali incorporates into the geopolymer structure, the remaining alkali will be dissolved in the polycondensation water [6] and would increase pH/alkalinity.

Corrosion behavior can be predicted using Pourbaix Diagram, devised in 1938 by Marcel Pourbaix, at that time a PhD candidate at the Faculty of Applied Sciences, Université Libre de Bruxelles. Pourbaix diagram is a graphical representation of the thermodynamic equilibrium of a metal – electrolyte, commonly aqueous solutions, in which the axes represents potential of the metal (electrode) and pH of the aqueous solution. Figure 1 is a simplified Pourbaix Diagram for iron – water at 298K [7]. The

¹ Department of Metallurgy and Engineering Materials, Universitas Indonesia, Jakarta, INDONESIA

² Alumni, Department of Metallurgy and Engineering Materials, Universitas Indonesia, Jakarta, INDONESIA.

* Corresponding Author e-mail: sotya.astutiningsih@ui.ac.id

Note: Discussion is expected before November, 1st 2013, and will be published in the “Civil Engineering Dimension” volume 16, number 1, March 2014.

Received 24 August 2011; revised 29 November 2012; accepted 04 June 2013.

y axis of the diagram denotes the potential of the electrode/metal. A certain metal (electrode) in an aqueous solution will oxidize if its potential is highly positive, i.e. at the upper part of the diagram. In the opposite, a certain metal will be reduced (reduction of the oxygen atoms) at highly negative potentials, i.e. the lower part of the diagram. The x axis of the diagram refers the pH of the electrolyte/aqueous solution, in which pH below 6 (the left part of the diagram) is considered acidic and the right part of the diagram, pH larger than 6 is considered alkaline/basic. The electrolyte is neutral if the pH is ~ 7 . The area below line a-b-j, i.e. the area at highly negative potentials, refers to the immunity zone. At this condition, a metal will not corrode. The area at highly positive potentials, above line g-f-h, is the corrosion zone. At this zone, the metal will oxidize (dissolve) into FeO_4^{2-} in an aqueous solution. While at the area e-f-g-k, the metal will oxidize into Fe^{3+} . At less positive potentials, in the area a-b-n-c-d-e, the metal will oxidize into 2-valenced Fe^{2+} . Inside area c-d-f-h-i, the metal oxidizes forming an oxide of Fe_2O_3 which will then acts as barrier for further reaction. This zone is referred to as passivation. At this zone metals will not corrode due to the formation of solid oxide film. However the presence of aggressive ions of Cl, for example in seawater, will cause the destruction of the film. Chloride ions are known to cause pitting corrosion in steels. Other passivation zones are e-i-p-n and n-p-j-b; at these zones solid oxides formed are Fe_3O_4 and $\text{Fe}(\text{OH})_2$, the latter of which is known as green rust.

Durability which is interpreted as resistance of geopolymer itself towards reaction with the environment has been studied in the last decade [8-13]. In summary, the media used to study the environmental interaction of geopolymer are acetic acid, sulphuric acid, deionized or distilled water, ASTM seawater, sodium sulphate, nitric acid and hydrochloric acid.

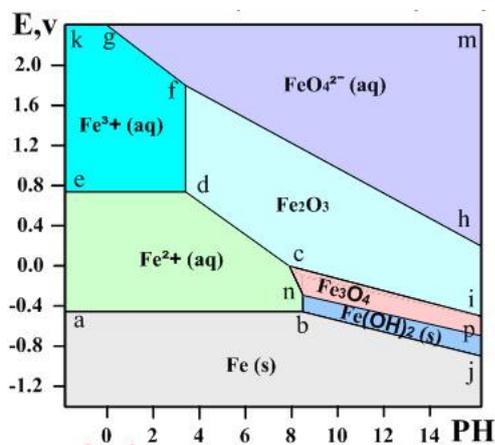


Figure 1. Simplified Pourbaix Diagram for iron–water system at 25°C [7]

Besides the chemical and/or corrosion resistance of the geopolymer itself, the behavior of the steel reinforcement within the geopolymer matrix has also been studied, although less in number [14-16]. Methods used in these experiments are by measuring half-cell potential [14, 15], corrosion potential and polarization resistance [16] in accelerated corrosion condition in laboratory. The steel was embedded within geopolymer matrix made from fly ash under atmospheric condition [14, 16] and in the presence of chloride ions by immersing in natural seawater [15] or by the addition of CaCl_2 in the geopolymer mortar [16]. Many research has been done on fly ash based geopolymer but none of the studies, whether on the chemical resistance of the geopolymer itself [8-13] nor on the corrosion behavior of the steel reinforcement [14-16], have been performed for kaolin-based geopolymer. In the present study, half-cell potential measurement of the steel reinforcement in kaolin-based geopolymer concrete immersed in distilled water and ASTM seawater was performed. The corrosion behavior of the steel reinforcement in kaolin-based geopolymer concrete was compared to both fly ash-based geopolymer and Portland cement concrete under the same condition.

Experiments

Corrosion proceeds in a slow rate that the effect cannot be observed in a relatively short time. Accelerated corrosion for steel reinforcement in concretes was performed by applying 'artificial' 3V potential and measuring the half-cell potentials, which is the decrease in potential between the steel and a standard electrode placed on the concrete surface [17]. The steel reinforcement as the anode and a copper cathode are connected through an electrolyte. By applying electric current via a rectifier and using calomel as the standard electrode, the half-cell potential of the steel can be measured. Figure 2 shows the arrangement of the apparatus. Accelerated corrosion allows us to observe the effect of corrosion in a relatively short period; however it only provides a qualitative analysis and does not provide information on the actual corrosion rate to predict service life of a component.

In each of the experiment set, three corrosion test pieces were immersed in distilled water and ASTM seawater and three compression test samples of fly ash-based geopolymer, kaolin-based geopolymer and Portland cement concrete were prepared. Portland cement concrete samples were immersed in water for 28 days for complete hydration before any measurement or test, while geopolymer samples were tested 1 day after casting. The ASTM seawater composition is presented in Table 1.

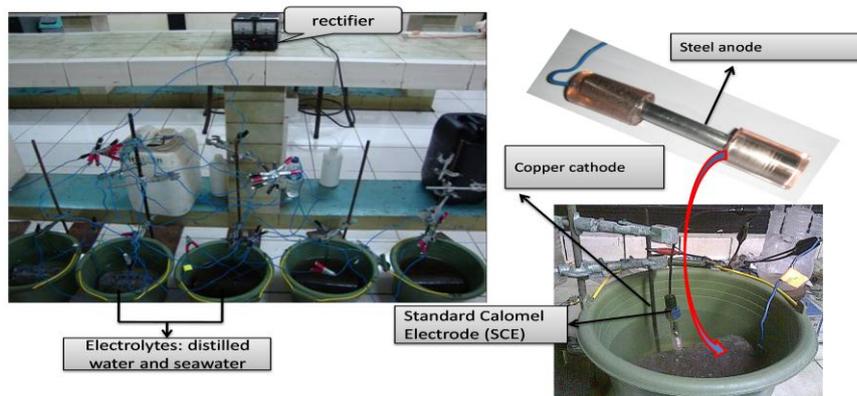


Figure 2. Arrangement of the Apparatus and Samples for Measuring Half-cell Potentia

The corrosion test piece was made up from a concrete cylinder of 10 cm diameter and height of 20 cm. In the center of the cylinder, a steel bar of 14 mm diameter and length of 14 cm was studded. Only 6 cm area in the middle of the bar was exposed to the concrete.

Samples for compression test were of 15 cm x 15 cm x 15 cm cubes. Each value of compressive strength was an average of 3 samples. The concrete mix was for a designated compressive strength of 40 MPa and that the ratio of cement paste, sand and coarse aggregates was the same for both Portland cement and geopolymer concrete. The water to cement ratio for Portland cement paste was 0.37 while the activator solution to ash ratio for geopolymer paste was 0.43. The composition of the fly ash used was determined using x-ray fluorescence and is presented in Table 2. The activator used was sodium silicate solution. Compositions of the geopolymer pastes are presented in Table 3.

Table 1. ASTM Seawater Composition

| Ingredients | Amount |
|-------------------------------------------|--------------------------------------------------|
| Volume total | 10L |
| NaCl | 245,34 gr |
| Na ₂ SO ₄ unhydrate | 40,940 gr |
| Stock no.1 | |
| MgCl ₂ .6H ₂ O | 111,114 gr |
| CaCl ₂ unhydrate | 11,588 gr |
| SrCl ₂ .6H ₂ O | 0,422 gr |
| Water | Was added until the mixture volume reached 200ml |
| Stock no.2 | |
| KCl | 6,945 gr |
| NaHCO ₃ | 2,01 gr |
| KBr | 1,005 gr |
| H ₃ BO ₃ | 0,271 gr |
| NaF | 0,03gr |
| Water | Was added until the mixture volume reached 100ml |

Table 2. Fly Ash and Kaolin Composition

| Chemical Formula | Wt. (%) | |
|--------------------------------|---------|---------|
| | Fly ash | Kaolin |
| Al ₂ O ₃ | 25.2600 | 42.3029 |
| SiO ₂ | 47.2992 | 55.1546 |
| S | 0.2969 | - |
| K ₂ O | 0.7009 | 1.2419 |
| CaO | 5.1482 | - |
| TiO ₂ | 1.7579 | 0.2068 |
| MnO | 0.1238 | - |
| Fe ₂ O ₃ | 16.5277 | 1.0902 |
| SrO ₂ | 0.1743 | - |
| MgO | 2.7074 | - |
| V ₂ O ₃ | - | 0.0037 |

Table 3. Geopolymer Paste Composition

| Atom / compound | Molar ratio | |
|-------------------------------------|-------------|--------|
| | Fly ash | Kaolin |
| Na/Al | 1.1032 | 1.1032 |
| Si/Al | 2.2126 | 2.0174 |
| H ₂ O/Na ₂ O | 10.1638 | 9.9618 |
| SiO ₂ /Na ₂ O | 2.2985 | 1.6321 |

Results and Discussion

Table 4 shows the compressive strength of fly ash-based geopolymer concrete after curing for 1 day. The average strength of geopolymer concrete was 45.3 MPa, achieving the target strength and is higher than the average compressive strength of 39.49 of the 28 day-cured Portland cement concrete having the same mix design. The compression test results of Portland cement concrete is shown in Table 5. Unlike fly ash geopolymer concrete, kaolin based geopolymer concrete did not achieve the designated strength. This type of concrete set quickly and achieved the optimum strength earlier. Table 6 presents the compressive strength of kaolin based geopolymer concrete after curing for 4, 8 and 24 hours.

Tabel 4. Compressive Strength of Fly Ash Geopolymer Concrete after 24 Hour Curing at 60°C

| No | Mass (kg) | Compressive strength (MPa) |
|---------|-----------|----------------------------|
| 1 | 7844 | 38,33 |
| 2 | 7823 | 46,60 |
| 3 | 7780 | 50,96 |
| Average | | 45,30 |

Tabel 5. Compressive Strength of Portland Cement Concrete after 28-day Curing

| No | Mass (kg) | Compressive strength (MPa) |
|---------|-----------|----------------------------|
| 1 | 7864 | 40,94 |
| 2 | 7874 | 38,33 |
| 3 | 7860 | 39,20 |
| Average | | 39,49 |

Tabel 6. Compressive Strength of Kaolin Geopolymer Concrete Cured at 60°C

| No | Curing time (hours) | Mass (kg) | Compressive strength (MPa) |
|----|---------------------|-----------|----------------------------|
| 1 | 4 | 12.154 | 15.25 |
| 2 | 8 | 12.200 | 16.08 |
| 3 | 24 | 11.856 | 17.13 |

Figures 3 and 4 show the half-cell potential measured every day since charging from day-1 to day-10 for the steel reinforcement in, respectively, fly ash-based and kaolin-based geopolymer concrete immersed in ASTM seawater and distilled water. The potential values range between -300 mV to -450 mV in seawater and -150 mV to -400 mV in distilled water. It is shown from the two graphs that the potential values from day-8 to day-10 are in a steady state condition. Compared to the fly ash-based geopolymer concrete, the kaolin-based geopolymer concrete showed a much more steady condition from the first day of measurement. The potential values ranges of the latter were between -250 to -300 mV in distilled water and between -500 to -550 mV in ASTM seawater. The fluctuations in the potentials measured referred to the conditioning of the samples before being established, which is when the surface of the samples has been thoroughly wetted. The steady state or corrosion potential equilibrium condition is established at a more positive potential forming a 'plateau'.

In a previous study by Yodmune and Yodsudjai [14] a high potential (3 V) was applied in order to accelerate corrosion. At this experiment, concrete was cured for 7 days to allow hardening and then immersed in water and charged until day-3. It was found that the half-cell potential decreased with day of charging. The potential measurement up to day-3 since charging was not adequate since the constant 'plateau' stage of potential had not been reached. It is the value of half-cell potential at the plateau stage that represents the real condition.

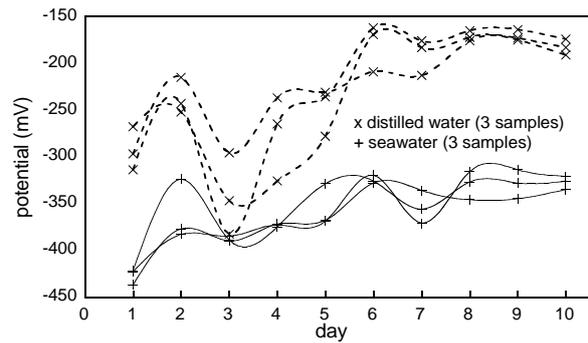


Figure 3. Half-Cell Potential of Steel Reinforcement in Fly Ash-Based Geopolymer Concrete Immersed in Seawater (+) and in Distilled Water (x) Measured Every Day from day-1 to day-10

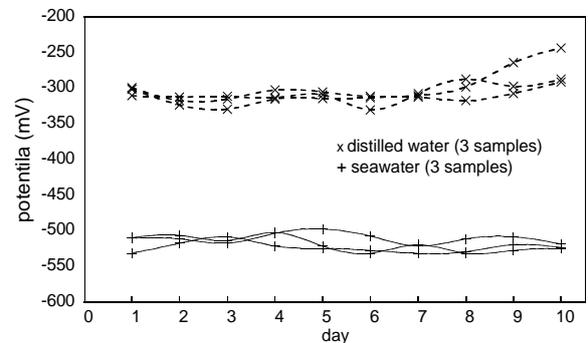


Figure 4. Half-Cell Potential of Steel Reinforcement in Kaolin Based Geopolymer Concrete Immersed in Seawater (+) and in Distilled Water (x) Measured Every Day from day-1 to day-10

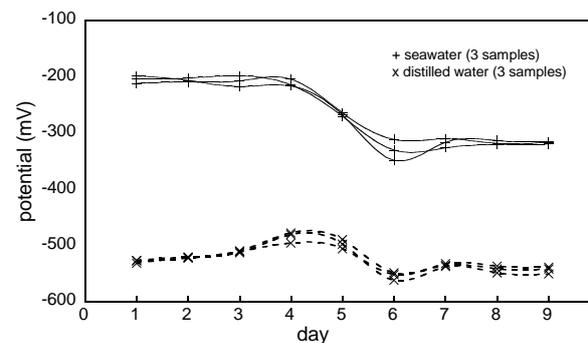


Figure 5. Half Cell Potential of Steel Reinforcement in Portland Cement Concrete Immersed in Seawater (+) and in Distilled Water (x) Measured Every Day from day-1 to day-9

Figure 5 shows the half-cell potential of steel reinforcement in Portland cement concrete from day-1 to day-9 of charging. It was shown from the graphs that starting at day-7, the potential has come to a constant potential or 'plateau' stage. Compared with the potential measurement of the steel in geopolymer concrete, the deviations between the samples of the same condition are much less for Portland cement concrete. This might be due to the long 28 day curing time where hydration had nearly

been completed in Portland cement concrete, whereas curing in geopolymer concrete might not have been completed although the strength has outstood the strength of Portland cement concrete. To confirm the results, charging of 'aged' geopolymer should be performed. Furthermore longer charging period is required especially for the fly ash based-geopolymer system to confirm that the steady state 'plateau' condition has been achieved.

Table 7. pH of the Medium in Geopolymer and Portland Cement System

| Medium /Day | Distilled water | | Seawater | |
|-----------------------------------|-----------------|------|----------|------|
| | First | Last | First | Last |
| Geopolymer system (fly ash based) | 7.2 | 10 | 8.2 | 9 |
| Geopolymer system (kaolin based) | 7.2 | 10 | 8.2 | 10.9 |
| Portland cement system | 7.2 | 7.2 | 8.2 | 8.2 |

Table 7 shows the pH measured at the beginning and end of charging. pH of both distilled water and ASTM seawater at the first day were the same for both the geopolymer and Portland cement system as no reaction has occurred. The pH of media in geopolymer system increases with increasing time of immersion due to the release of unreacted alkali in geopolymer that can be explained as follows.

Fly ash which is actually a solid solution of its oxides make up, dissolve into Si(OH)_4 and Al(OH)_4 oligomers upon reaction with high alkaline solution. Polycondensation or curing of the oligomers resulted in the formation of alkali aluminosilicate having 3D network structure and water as by product. The fact that Al atoms are covalently bonded to four O atoms requires that alkali ions, in this case the sodium ions, neutralize every $[\text{AlO}]$ tetrahedron. However, not all of the sodium ions are integrated into the aluminosilicate network structure. Excess sodium will be dissolved in water originated from the reactant and from the polycondensation reaction. The excess sodium will eventually increase pH much stronger than the Calcium Silicate Hydrates in Portland cement. The combination of high alkalinity and the soluble silicates present in geopolymer system promotes the formation of protective film on the reinforcing steel surface.

Distilled water is water with very low amount of dissolved solids and ions. Unlike normal potable water, distilled or pure water acts aggressively towards Portland cement concrete as it will dissolve calcium compounds in concrete [17]. Hardened Portland cement paste contains several types of calcium compounds. The first compound that will be dissolved by distilled water is calcium hydroxide Ca(OH)_2 . The removal of calcium hydroxide results in a more open matrix making the concrete

penetrable to aggressive solution. Other calcium compounds which is less soluble than Ca(OH)_2 will then also be dissolved.

The effect of seawater exposure upon concrete can be of several types: superficial erosion, swelling, chemical attack and freeze-thaw cycles [18]. Immersion of concrete in ASTM seawater in laboratory excludes the effect of other types of attacks except the chemical attack. For that reason, permanent submersion is less severe than tidal or splash-zone exposure. Seawater contains sulphate and chloride ions. The concrete itself are prone to sulphate ions while chloride ions attack the steel reinforcement causing pitting corrosion. Phases in Portland cement susceptible to sulphate ions in seawater are calcium hydroxide and hydrated calcium aluminates. However, examination using polarizing and fluorescence microscopy on concrete submerged in North Sea for 16 years showed a good and unchanged microstructure apart from the formation of discontinuous brucite, Mg(OH)_3 , layer on the outer few milimetres [Nijland and de Bruijn in 18]. Brucite layer is regarded as protective. Chloride ions, on the other hand, are aggressive to the steel reinforcement causing pitting corrosion and may penetrate through concrete. For those reasons techniques to develop non-destructive monitoring on the penetration of chloride ions is important [19].

The potential and pH at final day of both fly ash and kaolin based- geopolymer system and Portland cement concrete were plotted on Pourbaix diagram [20] to determine the corrosion condition of the steel reinforcement and the plot is presented in Table 8. The potential and pH plot of steel in Portland cement concrete immersed both in distilled water and seawater were in the corrosive area. Within the time of measurement, it seemed that protective film had not been formed or stabilized. For that reason, further experiment is being carried out for longer time of measurement. Yalcyn and Ergun [21] showed that corrosion rate of steel in Portland cement concrete in atmospheric condition decreased exponentially from day-1 to day-90 due to the formation of passive film. They observed that corrosion rate of concrete containing chloride ions, by adding sodium chloride into the mixture, was higher than the one without admixture.

On the other hand, fly ash-based geopolymer concrete immersed in distilled water was in the passive area while the one in seawater were in the corrosive region. Kaolin-based geopolymer concrete was passivated both in distilled water and seawater. Passive film, which is thin non porous and stable layer of Fe_2O_3 adhering to the steel surface, will be stable as long as there is no chloride ion penetration. Distilled water does not contain chloride ion while seawater contains abundance of this ion.

Table 8. Corrosion Behaviour of Steel in Geopolymer and Portland Cement Concrete

| Concrete type | Media | Day- | | Pourbaix Diagram Plot at final day |
|--------------------------|-----------------|-----------------|------------------|------------------------------------|
| | | Initial | Final | |
| Portland | Distilled water | -0.529V; pH 7.2 | -0.543 V; pH 7.2 | corrosion |
| | seawater | -0.205V; pH 8.2 | -0.319; pH 8.2 | |
| Fly ash based-geopolymer | Distilled water | -0.293V; pH 7.2 | -0.183V; pH 10 | passivation |
| | seawater | -0.427V; pH 8.2 | -0.327V; pH 9 | |
| Kaolin based-geopolymer | Distilled water | -0.299V; pH 7.2 | -0.243V; pH 10 | passivation |
| | seawater | -0.531; pH 8.2 | -0.523; pH 10.9 | |

The destruction of the passive film can be detected by measuring corrosion current or polarization resistance which was not performed in this work. The present work only confirmed the passivation of steel reinforcement in kaolin geopolymer in distilled water. The corrosion resistance in the geopolymer system was very likely due to the alkaline environment provided by the excess alkali from geopolymer synthesis that encourage the formation of passive film protecting the steel. The excess sodium had made the concrete environment alkaline and thus increased the pH of the medium, especially distilled water. The pH of seawater was not as high as distilled water because part of the excess sodium reacted with chloride ions abundant in seawater forming salt of sodium chloride, and was also due to the formation of calcium chloride in the case of fly ash containing calcium [22], thus decreasing the pH. The formation of salt was confirmed in this experiment during visual observation of geopolymer samples in seawater. The fact that pH of seawater where the kaolin-based geopolymer had been immersed was more alkaline compared to the fly ash-based system was due to the higher concentration of the unreacted sodium silicate in the kaolin system. The quantity of unreacted sodium silicate in geopolymer system was suspected to be related to carbonation and strength of the material [23], which is beyond the scope of this study.

Besides alkalinity, geopolymer has an inherent characteristics that makes this material superior to Portland cement in term of chemical resistance. This inherent characteristic is the absence of calcium compounds in the strength contributing phase(s) in the material to the opposite to the fact that calcium compounds are the strength contributing phase(s) in Portland cement. In term of chemical resistance calcium is unwanted in geopolymer system, however precursors or raw materials for geopolymer may contain calcium, for example fly ash and blast furnace slag. In term of mechanical strength, the

effect of calcium silicate in geopolymer depends on the crystallinity of the calcium silicate source and the alkalinity of the activator [24]. At lower alkalinity, amorphous or glassy calcium silicate formed C-S-H gel together with the aluminosilicate geopolymer structure enhancing the strength of the overall matrix. On the other hand, crystalline calcium silicate was not reactive at low alkalinity thus would not form CSH gel and therefore disrupted the geopolymer network resulting in strength decrease. At high alkalinity, geopolymer formation was predominant resulting in similar strength regardless of the calcium silicate crystallinity.

Summary

Two types of geopolymer concrete had been made using fly ash and kaolin as the raw materials. Half-cell potential measurement of the steel bars reinforcing geopolymer concrete and ordinary Portland cement concrete were compared. The reinforced concrete were immersed in aggressive media of distilled water and ASTM seawater and charged with high potential of 3 V to accelerate corrosion for the duration of 10 days. Along with half-cell potential, pH of the media was measured at the first day to last day of charging. The half-cell potential and pH of each system was plotted on a Pourbaix diagram. It was found that steel in Portland cement concrete both in distilled water and ASTM seawater were corroded until last day of charging. Steel bars in both types of the geopolymer concrete were already in the passivation state at day 10 of immersion in distilled water. Upon immersion in ASTM seawater, the steel in fly ash concrete was corroded while the one in kaolin concrete had been passivated only temporarily. In the presence of chloride ions, i.e. in seawater, passive film tends to be destroyed. Corrosion potential, which was performed in this work, cannot predict the stability of the passive film. Polarization resistance should be measured to ensure the stability of the passive film in the kaolin-based geopolymer system in seawater. Furthermore, longer time of observation until the steady state 'plateau condition' achieved is necessary and is in progress. The fact that pH of distilled water and seawater of the geopolymer system increase more than those of the Portland cement system during immersion was due to the release of excess sodium in the geopolymer structure.

Acknowledgement

This work was part of a project funded by Ditlitabmas DIKTI, Ministry of Education under the scheme Hibah Bersaing 2009/2010 contract no. 408D/DRPM-UI/A/N1.4/2009 and 1705/H2.R12.3/PPM.00 Penelitian/2010.

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