

จีโอพอลิเมอร์จากดินขาวผสมน้ำยางพาราเพื่อป้องกัน

คอนกรีตเสริมเหล็กจากการกัดกร่อน

Geopolymer from Metakaolin containing Field Para Rubber Latex Coating for Protecting Reinforced Concrete against Corrosion

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บทคัดย่อ

ในการศึกษาครั้งนี้เป็นการศึกษาจีโอพอลิเมอร์พาสต์จากดินขาวผสมน้ำยางพารา และกระตุ้นด้วยโซเดียมซิลิเกตและโซเดียมไฮดรอกไซด์ ในการเคลือบตัวอย่างคอนกรีตเพื่อตรวจสอบการกัดกร่อนของเหล็กจีโอพอลิเมอร์พาสต์ถูกเตรียมด้วยดินขาวแทนที่ด้วยน้ำยางพาราร้อยละ 1, 3, 5 และ 10 โดยน้ำหนัก โดยมีกระบวนการผลิตคอนกรีตด้วยจีโอพอลิเมอร์พาสต์ 1 และ 2 ชั้น และมีการบ่มตัวอย่างที่อุณหภูมิเป็นเวลา 24 ชั่วโมง ทดสอบกำลังดึงผ่าซีกและการเกิดสนิมในเหล็ก จากการเร่งด้วยกระแสไฟฟ้า 12 โวลต์ โดยการให้กระแสไฟฟ้า 8 ชั่วโมง หยุด 8 ชั่วโมง เป็นเวลา 14 วัน ทดสอบก่อนตัวอย่างที่กำลังแช่ในน้ำทะเล ผลการทดสอบแสดงให้เห็นว่าตัวอย่างเคลือบจีโอพอลิเมอร์พาสต์ที่ผสมน้ำยางพาราร้อยละ 1, 3 และ 5 โดยน้ำหนัก สามารถลดการกัดกร่อนได้อย่างชัดเจน ทั้งการเคลือบ 1 และ 2 ชั้น พร้อมทั้งวิเคราะห์ตรวจสอบโครงสร้างระดับจุลภาคบริเวณรอยต่อระหว่างคอนกรีตและจีโอพอลิเมอร์พาสต์

คำสำคัญ: จีโอพอลิเมอร์, ดินขาวเผา, น้ำยางพารา, การเคลือบ, การกัดกร่อน

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ABSTRACT

In this study, geopolymers were synthesized from metakaolin containing field Para rubber latex and activated with sodium silicate and sodium hydroxide. The concrete samples were coated with these geopolymers to assess their corrosion resistance. Geopolymers were prepared by substituting metakaolin with field Para rubber latex at weight percentages of 1%, 3%, 5%, and 10%. The concrete specimens were coated with single or double layers of the geopolymers and then cured for 24 hours. Tensile strength and corrosion performance of the steel were evaluated through an accelerated corrosion test with a 12-volt electric current, applied for 8 hours and paused for 8 hours, over a duration of 14 days. Additionally, samples submerged in seawater were tested. The results demonstrated that the samples coated with geopolymers containing 1%, 3%, and 5% natural rubber latex by weight exhibited a significant reduction in corrosion, evident in both single and double-coated specimens. Structural analysis at the micro level was also conducted at the interfacial transition zone between concrete and geopolymers paste.

Key words: geopolymer, metakaolin, field Para rubber latex, coating, corrosion

INTRODUCTION

The corrosion of reinforcing steel in concrete structures causes severe deterioration of infrastructure in concrete elements exposed to chloride ions in seawater, brackish water, or waste water from factories. Several previous studies (Jacek *et al.*, 2018; Jhutan *et al.*, 2021; Marilene *et al.*, 2021; Mostafa *et al.*, 2020) have reported that geopolymer binders have excellent materials properties, especially acid resistance and durability. Therefore, geopolymers have been used as coatings in prior studies.

Coatings can effectively protect concrete structures against steel corrosion from chemical exposures. The coatings on concrete surfaces need to be designed for ease of application and good adhesion. Brenna *et al.* (2013) studied polymer modified mortar and polymeric coating of reinforced concrete with long-term chloride induced corrosion. Christodoulou *et al.* (2013) used silane coating of reinforced concrete in an assessment of performance of the surface. Geopolymer coatings in particular have been widely applied to coat steel or structural concrete for corrosion

protection (Shahedan *et al.*, 2014; Chindaprasirt and Rattanasak, 2016). Regarding geopolymer coatings, Aguirre-Guerrero *et al.* (2017) reported that geopolymers were prepared with fly ash and metakaolin as protective coatings against chloride-induced corrosion in reinforced concrete. The samples were immersed in a 3.5% NaCl solution with wetting/drying cycles and a constant 5 V potential. The results showed that metakaolin based geopolymer coating exhibited the best performance, reducing the corrosion rate compared to concrete without coating. Wiyono *et al.* (2015) used geopolymer coatings prepared from fly ash and calcined volcanic mud. The concrete substrates were exposed to 10% sulfuric acid in wet-dry cycles, and to chloride solution to evaluate its penetration depth. The results showed that geopolymer coating improved the durability of concrete samples.

In 2019, global production of Para rubber was approximately 13,841,000 tons. Thailand produced approximately 4,852,000 tons or 35% of this total (Rubber Economic Research and Development Division, 2020). In fresh or field state, the latex raw form of rubber (field Para rubber latex,

FPRL) annual amount in Thailand is estimated as 10.78 to 19.41 million tons, because FPRL contains also 55-75% water and non-rubber substances such as sludge, proteins, and some inorganic materials, along with 25-45% of rubber particles (Knowledge Management, 2017). In FPRL the rubber particle size ranges within 0.04-4.0 micron with mean particle size of 1.0 micron (Blackley, 1997), for the clonal variety RRIM 600 (Rubber Research Institute of Malaysia 600) of rubber trees.

Limited research exists concerning the utilization of FPRL in geopolymer binders, particularly regarding the application of geopolymer coatings for safeguarding against steel corrosion. Consequently, further investigation is necessary to thoroughly assess the impacts of FPRL in geopolymer composite materials. This present study seeks to examine the splitting tensile strength, steel weight loss, and the microstructure of the interfacial transition zone between the concrete surface and geopolymer binder paste coatings containing field Para rubber latex. The samples, incorporating FPRL contents of 0%, 1%, 3%, 5%, and 10% by weight, were submerged in seawater at ambient temperature for 14 days, with 12 V excitation alternating every 8 hours. These samples featured either one or two coating layers on concrete substrates, characterized by either a flat or rugged surface. The objective of this study is to analyze the influence on splitting tensile strength and

steel weight loss, while evaluating the ITZ microstructure through scanning electron microscopy (SEM). Significantly, this paper offers a candid discussion on the challenges encountered during the geopolymer application process.

MATERIALS AND METHODS

1. Materials

Metakaolin (MK) and field Para rubber latex (FPRL) were sourced from the Narathiwat province in southern Thailand. The kaolin used in this study was calcined at 750 °C for 2 h. The chemical compositions and particle size distribution are presented in Table 1 and Figure 1, respectively. The chemical composition of MK was analyzed via X-ray fluorescence (XRF). According to ASTM C618 (2019), the MK utilized in this study falls under class F. The total proportion of the major oxides (SiO₂ and Al₂O₃) amounted to 91.32%, while the CaO content stood at 0.33%. Figure 1 illustrates the particle size distribution of MK, revealing an average particle size of approximately 9 μm, with 96% of the MK passing through the 45-micron sieve.

Field Para rubber latex (from rubber tree clonal variety RRIM 600) used in this study was collected from Narathiwat province in Thailand. The FPRL is a suspension with 35-40% total solids content. The particle sizes in FPRL are in the range 0.04-4.0 μm. Kaesaman *et al.* (2014) reported that the total solids content in FPRL was 36.9%.

Table 1 The chemical composition of MK

Component	% by weight
SiO ₂	50.30
Al ₂ O ₃	41.02
Fe ₂ O ₃	1.05
CaO	0.33
TiO ₂	1.05
K ₂ O	4.08

The alkaline activators for geopolymerization reactions were prepared using sodium silicate ($\text{Na}_2\text{O} = 14.85\%$, $\text{SiO}_2 = 29.45$ and $\text{H}_2\text{O} = 55.7\%$), sodium hydroxide (NaOH) flakes of 99% purity, and water for the geopolymer paste. Natural river sand for ordinary Portland cement concrete passing

through ASTM sieve No. 4, with particle size below 4.75 mm, specific gravity 2.56, and fineness modulus 2.59, was used to prepare the concrete substrate. The coarse aggregate used in this work was compliant with ASTM C33/C33M (2013).

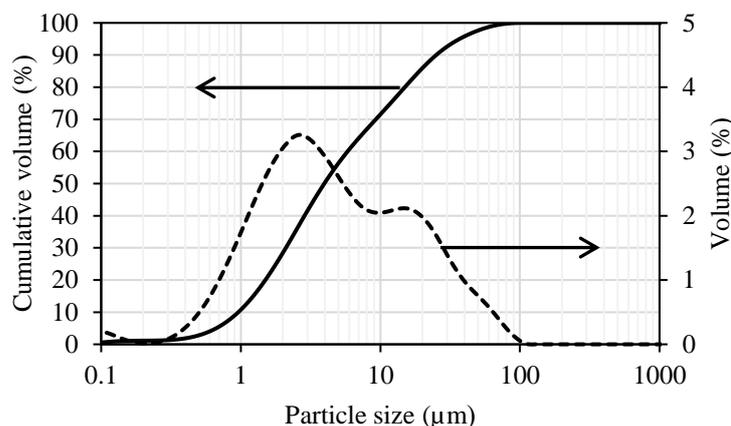


Figure 1 Particle size distribution of matekaolin

2. Mixture proportion and curing of samples

Geopolymer paste was created by blending MK with FPRL and mixing them with sodium silicate, sodium hydroxide, and water in accordance with the experimental design. Field Para rubber latex was incorporated as a stabilizing agent. The specific mixture proportions are detailed in Table 2. These proportions were selected to optimize both the enhancement and reduction of corrosion in the reinforcing steel with coatings. The ratios of powder and alkaline activator were kept constant for a robust formulation. MK, sodium silicate, sodium hydroxide, and water were mixed for 3 minutes until achieving a homogenous, viscous liquid. Geopolymer

binders were mixed for 3 minutes before being applied onto the concrete substrate using a paintbrush. The concrete samples received either 1 or 2 coating layers, were not enveloped in polyvinyl, and were cured at ambient temperature for 24 hours for a single-layer coating. In the case of a double-layer coating, after the initial layer was applied, the samples were cured at ambient temperature for 7 days. Subsequently, the second layer was applied and allowed to cure at ambient temperature for an additional 24 hours. Following this, the samples were immersed in seawater for the steel corrosion test.

Table 2 Mix proportions of geopolymer binder pastes (by weight)

MK (%)	FPRL (%)	(MK+FPRL) : SS (g)	SS : SH (g)	B : W (g)
100	0			
99	1			
97	3	1 : 0.75	2.5 : 1	1 : 0.3
95	5			
90	10			

Abbreviations used: MK = metakaolin; FPRL = field Para rubber latex; SS = sodium silicate; SH = sodium hydroxide; B=binder (MK+FPRL) ; W = water

3. Methods

3.1 Reinforced concrete substrate

In order to create concrete samples (substrates), commercially available raw materials from Thailand were chosen, which encompassed a general-use Portland cement, reinforcing steel bars, river sand, and rocks. Two distinct types of concrete samples were prepared, distinguished by water-to-cement ratios of 0.5 and 0.65, and a cement-to-river sand-to-rock ratio of 1:2:4 by weight. These samples were subsequently submerged in water for a 28-day curing period. Each specimen contained a centrally embedded reinforcing steel bar, positioned 5 cm away from the bottom. The samples featuring a water-to-cement ratio of 0.5 exhibited sizable pores and rugged surfaces, whereas those with a water-to-cement ratio of 0.65 displayed a uniform matrix and smooth surfaces. The strength and surface conditions of these samples are concisely outlined in Table 3.

3.2 Geopolymer coating

The coatings were manually applied using a paintbrush. Initially, the concrete surface was wiped with a damp cloth to saturate the pores and prevent or limit the undesired absorption of water from the coating geopolymer paste. Following this, the geopolymer coating was prepared and subsequently applied.

In the context of concrete coatings, there are at least two primary factors that significantly contribute to the permeability of seawater, primarily chloride ions, as illustrated in Figure 2. The mechanism involving chloride ions entails the dispersion of seawater through the geopolymer coating layer and into the concrete substrate.

3.3 Steel corrosion

The concrete samples underwent steel corrosion testing. These samples had a diameter of 100 mm and a height of 200 mm, featuring an embedded steel bar with a diameter of 12 mm and a length of 200 mm. The weight of the steel bars was measured

to ascertain the extent of weight loss caused by corrosion.

3.4 Imposed voltage

The imposed voltage test was conducted 24 hours after the application of the geopolymer paste coating. The aim of this test was to evaluate the corrosion of the steel bars embedded within the concrete samples in the presence of chlorides, as well as to determine the impact of the coatings on this corrosion process. The concrete specimens were immersed in seawater, and a voltage of 12 V was intermittently applied every 8 hours from an external power source (as shown in Figure 3), between the steel bar and a steel sheet placed in the seawater, over a duration of up to 14 days. Subsequently, the samples were subjected to a splitting tensile strength test after undergoing a 24-hour drying period at ambient temperature.

3.5 Splitting tensile strength

The splitting tensile strength of the concrete coating was determined through the application of the test method outlined in ASTM C496/C496M (2017). In the splitting tensile strength test, a 100 mm x 200 mm cylinder is subjected to axial compression. The results of the splitting tensile strength test for the samples are shown in Figure 4. To calculate the splitting tensile strength of the specimen, follow the steps below:

$$T = \frac{2P}{\pi ld} \quad (1)$$

T = Splitting tensile strength, (MPa)

P = Maximum applied load indicated by the testing machine, (N)

l = Length, (mm)

d = Diameter, (mm)

3.6 Scanning electron microscopy (SEM) analysis

For microscopic analysis, small fragments of the samples from the steel

corrosion tests (conducted subsequent to the splitting tensile test) underwent examination via scanning electron microscopy. The JMS-5800 LV model scanning electron

microscope (JEOL, Japan) was employed to scrutinize the microstructure within the concrete samples.

Table 3 The two types of concrete substrate

W/C	Ambient temperature	Curing (days)	Surface	Compressive strength (MPa)
0.50	30±3	28	rough	26.87
0.65	30±3	28	smooth	19.81

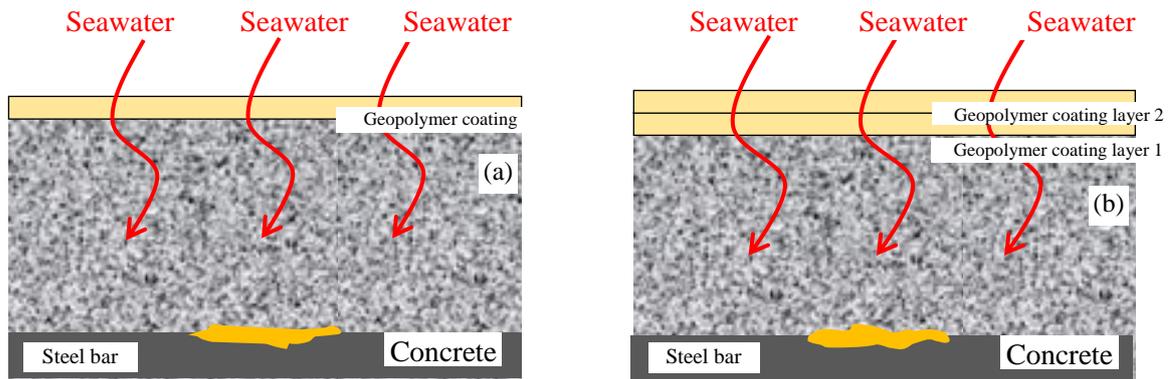


Figure 2 Illustration of the seawater penetration mechanisms with geopolymer coatings on concrete substrates (a) 1 coating layer, and (b) 2 coating layers

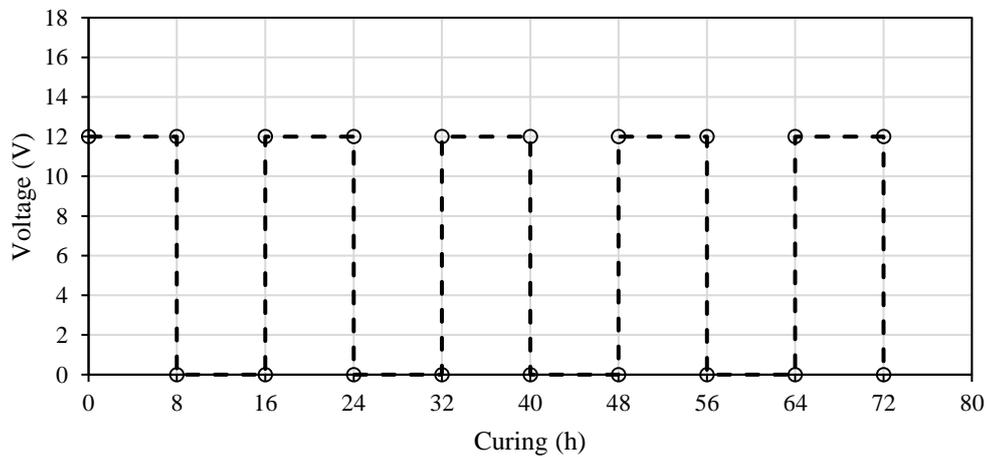


Figure 3 Impose voltage of samples



Figure 4 Splitting tensile strength of samples

RESULTS AND DISCUSSION

1. Physical characteristics

The application of coatings was done manually with the experimental mixtures. The physical characteristics of the coatings were determined after 12 V excitation under seawater for 14 days, with on/off switching each 8 hours. Figure 5 shows the results for concrete specimens without coating having both types of surfaces (rough or smooth) and clear rust stains are seen in Figures 5(a) and 5(b). On the other hand, single or double layers of geopolymer paste coating protected the steel bars in the concrete specimens, especially with 2 coating layers.

2. Splitting tensile strength

The splitting tensile strength of concrete samples was determined for cases coated with double layers. The samples were measured in applied test with ASTM C496/C496M (2017), except for using 100 mm x 200 mm sample size with steel bar embedded in the axial center. Only small amounts of steel loss were observed in this study because of the relatively short corrosion period of 14 days. The condition of steel in concrete after the corrosion test is shown in Figure 6, with subjectively obvious corrosion. While samples without coatings had clear corrosion on surfaces, those with 2 coating layers had only slight surface corrosion, both with rough and smooth concrete cases. The results of splitting tensile strength for the two surface types are shown in Figures 7 and 8. The splitting tensile

strengths ranged in 2.66-3.34 MPa (see Figure 7) and in 1.49-2.29 MPa (see Figure 8) for rough and smooth cases, respectively. It is observed that rough surface cases gave higher splitting tensile strength than smooth surface cases. This is because cases with rough surfaces had water-to-cement ratio of 0.5, lower than the 0.65 for smooth surfaces. It is known that concrete with lower water to cement ratio has higher strength. Moreover, the increase of split tensile strength due to the geopolymerization process, where aluminosilicate oxides react with alkali polysilicates to produce Si-O-Al polymeric bonds. Aluminosilicate polymeric bonds were more stable when submerged in seawater (Zhang *et al.*, 2010a). The splitting tensile strength of the specimens was determined at the age of 14 days. In Figure 8, the results indicate that the splitting tensile strength of samples with geopolymer coating was slightly increased relative to cases without coating. The cases with smooth surfaces had approximately similar splitting tensile strengths with and without coating. The test results show that geopolymer paste coatings with and without FPRL on concrete substrate gave only slight differences in splitting tensile strength after immersion in sea water. This is because of the short curing/corrosion time (14 days) under seawater. Nastiti and Ekaputri (2020) reported that the concrete samples were coated with fly ash geopolymer mortar of the difference thickness. The results shown that immersion in seawater for 30 days slightly affected the compressive strength.



Figure 5 Geopolymer concrete samples (a) without coating on rough surface, (b) without coating on smooth surface, (c) 5% FPRL single layer, rough surface, (d) 5% FPRL single layer, smooth surface, (e) 5% FPRL double layers, rough surface, and (f) 5% FPRL double layers, smooth surface

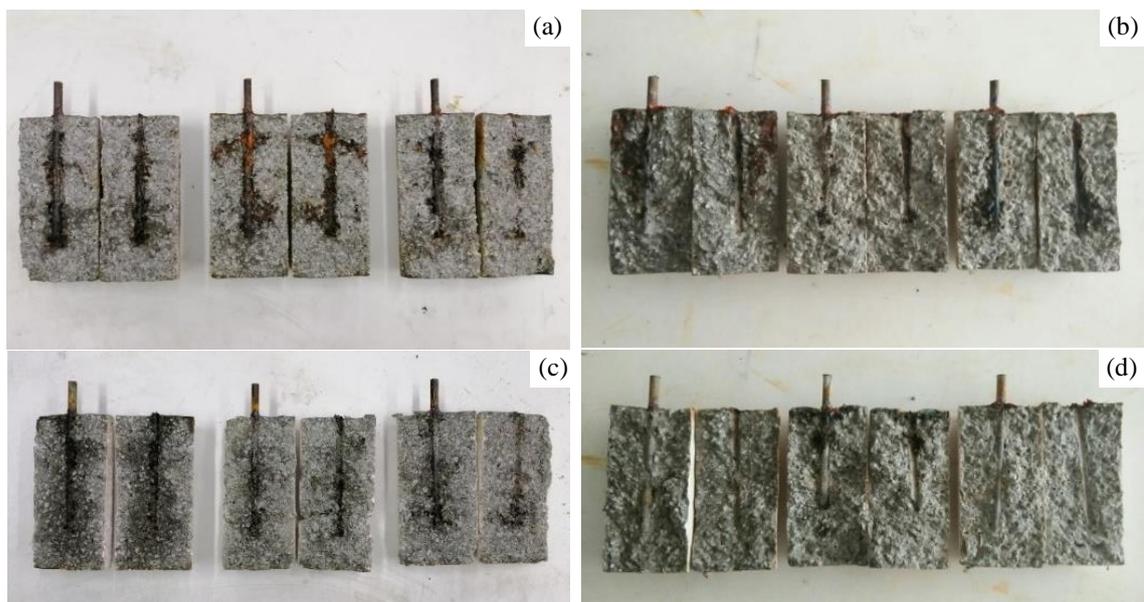


Figure 6 The splitting tensile strength of concrete samples (a) without coating on rough surface, (b) without coating on smooth surface, (c) 5% FPRL double layers, rough surface, and (d) 5% FPRL double layers, smooth surface.

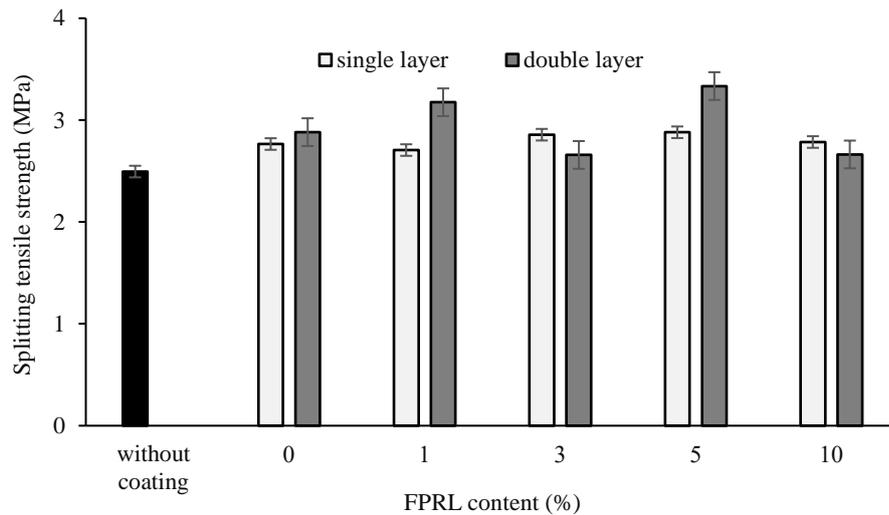


Figure 7 Effect of FPRL content on splitting tensile strength, rough surface.

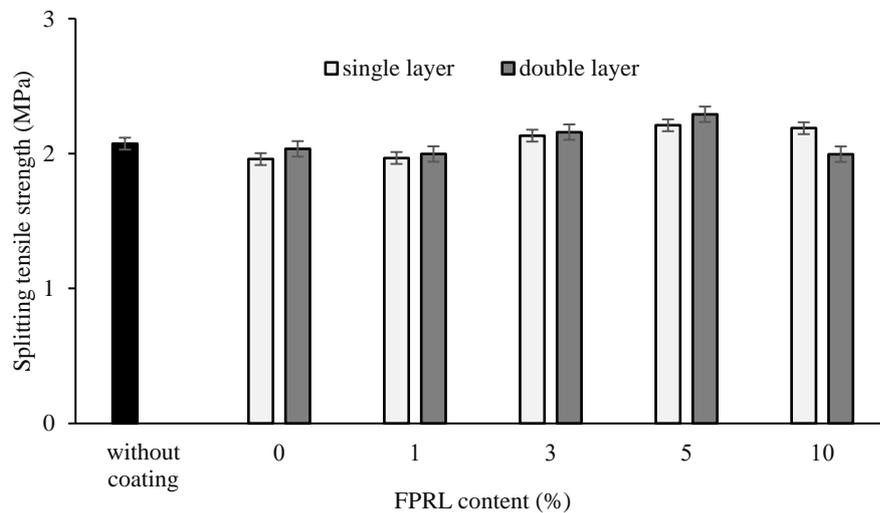


Figure 8 Effect of FPRL content on splitting tensile strength, smooth surface.

3. Imposed voltage

Figures 9 and 10 show the steel bar weight loss from corrosion during immersion in seawater with 12 V cycled on/off every 8 hours. The effects of FPRL content on the steel weight loss from the samples are shown in Figure 9 for rough surface cases and in Figure 10 for smooth surface cases. The steel weight loss by FPRL content in the geopolymer coating on rough surface is presented in Figure 9. The specimens with 1%, 3%, and 5% FPRL reduced corrosion of the reinforcing bars in the concrete samples, while geopolymer binder pastes without FPRL gave poorer protection against corrosion. This is because FPRL had fine

particles of 0.4-4 micron sizes that plugged pores in the matrix, reducing its permeability and preventing the penetration by chloride ions. However, it was observed that geopolymer binder pastes containing 10% FPRL had increased weight loss, due to the large FPRL content in geopolymer system inducing large pores in the coating. Hawa *et al.* (2017) assessed fly ash geopolymer mortars containing field Para rubber latex. SEM imaging showed that high content of FPRL gave a highly porous microstructure. Both concrete surface types (rough and smooth) behaved similarly in this respect. A similar phenomenon was presented by Tittarelli *et al.* (2018) who found that the higher total

porosity of geopolymer compared other geopolymeric matrices favors the ingress and thus the attack of chloride ions. Zhang *et al.* (2010b) investigated the use of geopolymers as coating materials for the

anticorrosion protection of reinforce concrete. The results shows that geopolymer had low permeability and excellent anticorrosion.

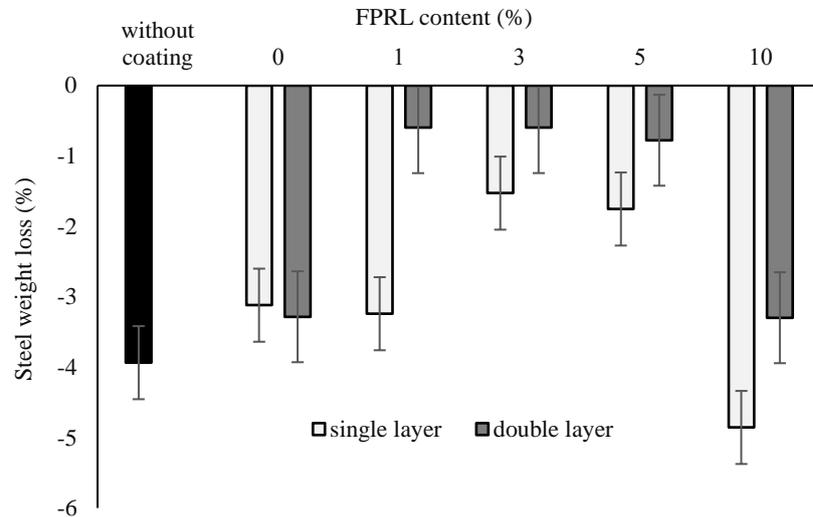


Figure 9 Effect of FPRL content on steel weight loss, rough surface.

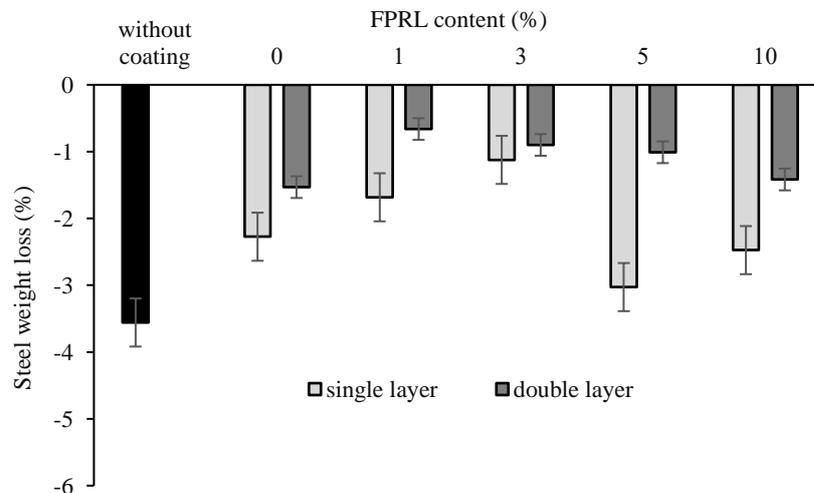


Figure 10 Effect of FPRL content on steel weight loss, smooth surface.

When comparing 1 and 2 coating layers, double layers protected steel well against weight loss, because the geopolymer had a dense compact microstructure that protected against seawater penetration and chloride exposure. SEM images were taken for morphological and elemental analysis of the interfacial transition zone. The smooth surfaces resulted in less steel weight loss

than the rough surfaces, because the smooth texture of concrete was associated with a dense and compact homogeneous matrix, i.e. low permeability in the concrete. For cases with rough surfaces, double layers of coating clearly provided better protection than a single layer.

The samples were coated with geopolymer pastes containing metakaolin

as a partial replacement for field Para rubber latex with single or double layers, showing a slight increase in splitting tensile strength. It was shown that the coating of metakaolin-based geopolymer paste had no effect on the splitting tensile strength of the concrete substrate. However, in this study of corrosion with immerse samples in seawater, it is clear that the steel corrosion resistance is improving. Especially, the samples were coated geopolymer paste containing FPRL ranging from 1 to 5%. Zhang *et al.* (2012) reported that geopolymer coating materials have been proposed for protecting concrete structures exposed to marine environment. The systematical experiments from laboratory and field applications have demonstrated that the coating possesses excellent anti corrosion properties.

Yoon *et al.* (2018) presented that fly ash and blast furnace slag were used as binder materials, and sodium hydroxide and sodium silicate were used as alkali activators. The results showed that the chloride penetration depth of alkali-activated sample was much lower than that of the OPC sample. The presence of alkali activated was effective in improving the chloride penetration resistance. Hence, the samples coating with geopolymer paste can protect against corrosion in the steel bars. The chloride binding capacity of geopolymer samples under standard curing is better than that of OPC sample under the same conditions. It was reported that the high alumina content promoted the chloride binding capacity (Tong *et al.*, 2021). Moreover, Ross *et al.* (2022) reported that geopolymer with fly ash, when tested, was found to have low permeability compared to portland cement paste and slurries. The chloride diffusion coefficient of alkali activator materials was generally lower than that of ordinary Portland cement due to the increased tortuosity, lower total porosity and water absorption properties, which could be also affected by precursor chemistry (Tahri *et al.*, 2021).

4. Morphological and elemental analysis of interfacial transition zone

Microstructural characteristics of the interfacial transition zone (ITZ) between geopolymer paste and the concrete surface were assessed using scanning electron microscopy (SEM). SEM images were taken of cross sections of geopolymer coatings from low FPRL content to high FPRL content, and with 1 or 2 coating layers. Figures 11 and 12 show representative images of ITZ and geopolymerization product morphology. In Figure 11, geopolymer coating with 5% FPRL and 1 coating layer had small lacunae in the ITZ between the geopolymer binder paste and the concrete surface. The binder had small pores in the cross section. It was observed that the samples were not connected in a matrix concrete surface and geopolymer paste. This is because the samples with 1 coating layer of viscous geopolymer were cured at ambient temperature for 24 hours, and the short curing time cannot produce geopolymerization products on the concrete surface, resulting in the lack of connection in the matrix structures. Figures 11(b) and 11(c) clearly show unreacted raw materials. On the other hand, the samples with 2 coating layers show geopolymerization products at the interfacial transition zone between the concrete surface and geopolymer paste. The SEM imaging shows a homogenous microstructure and continuous matrix with concrete surface and geopolymer paste in the microstructure (see Figure 12(c)). The MK particles were combined through cementitious reactions that provided a dense compact microstructure, offering good protection against chloride in seawater (see Figure 9 and 10). The reaction extent in geopolymer binder paste was higher in samples with 2 coating layers, because after first coating, they were cured for 7 days at ambient temperature. Samples with 1 coating layer only were cured for 24 hours. However, it was observed that the geopolymer binder paste containing 10% FPRL couldn't protect against weight loss of steel. This is because high FPRL in the geopolymer

causes high porosity. In a study on geopolymer with fly ash partial replacement by field Para rubber latex, Hawa *et al.* (2017) showed that geopolymer with a high FPRL content had large pores in the matrix. The large pores in the geopolymer matrix allow

seawater to penetration the concrete. Moreover, Hawa and Prachasaree (2020) reported that fly ash-based geopolymer partial replacement of FPRL at 1% and 3% had few pores in the matrix.

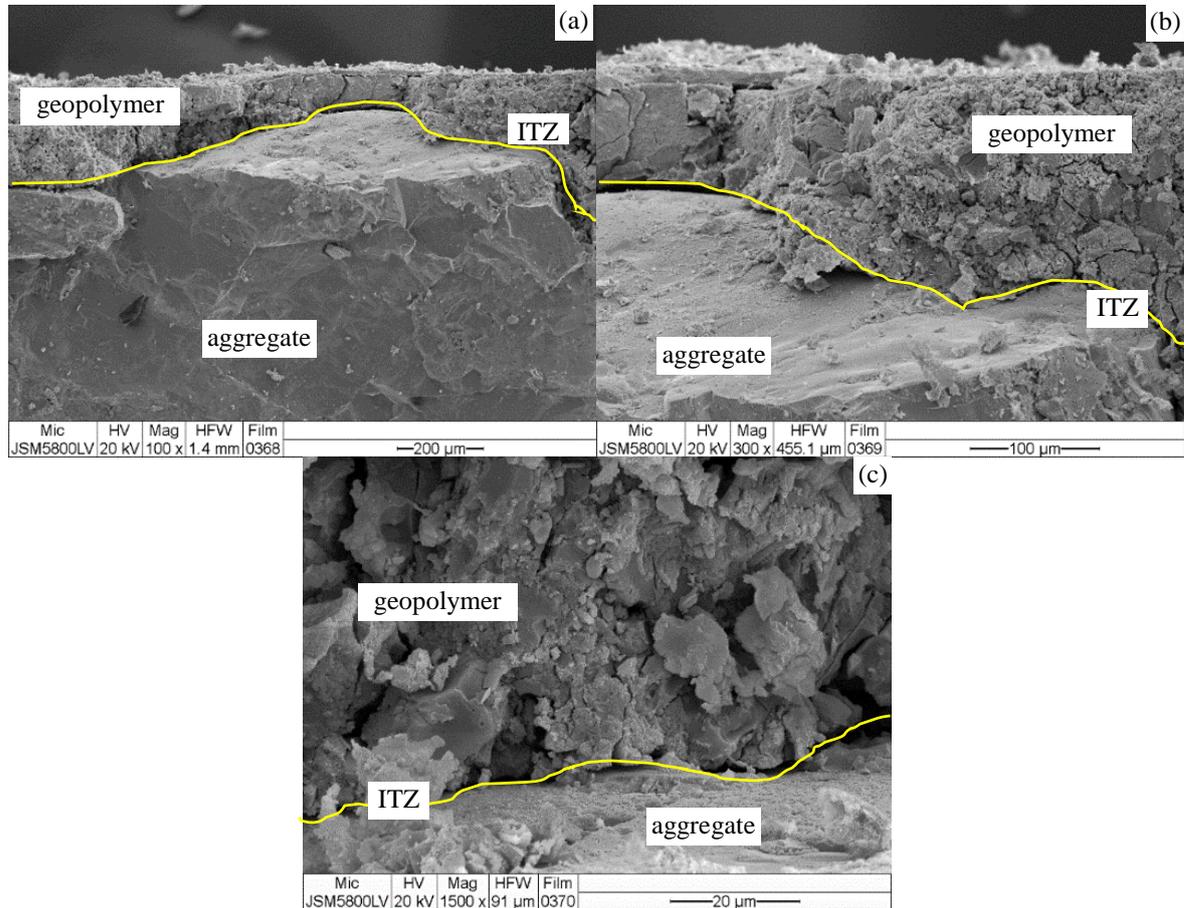


Figure 11 SEM images of geopolymer coatings with 5% FPRL single layer (a) 100x, (b) 300x, and (c) 1,500x

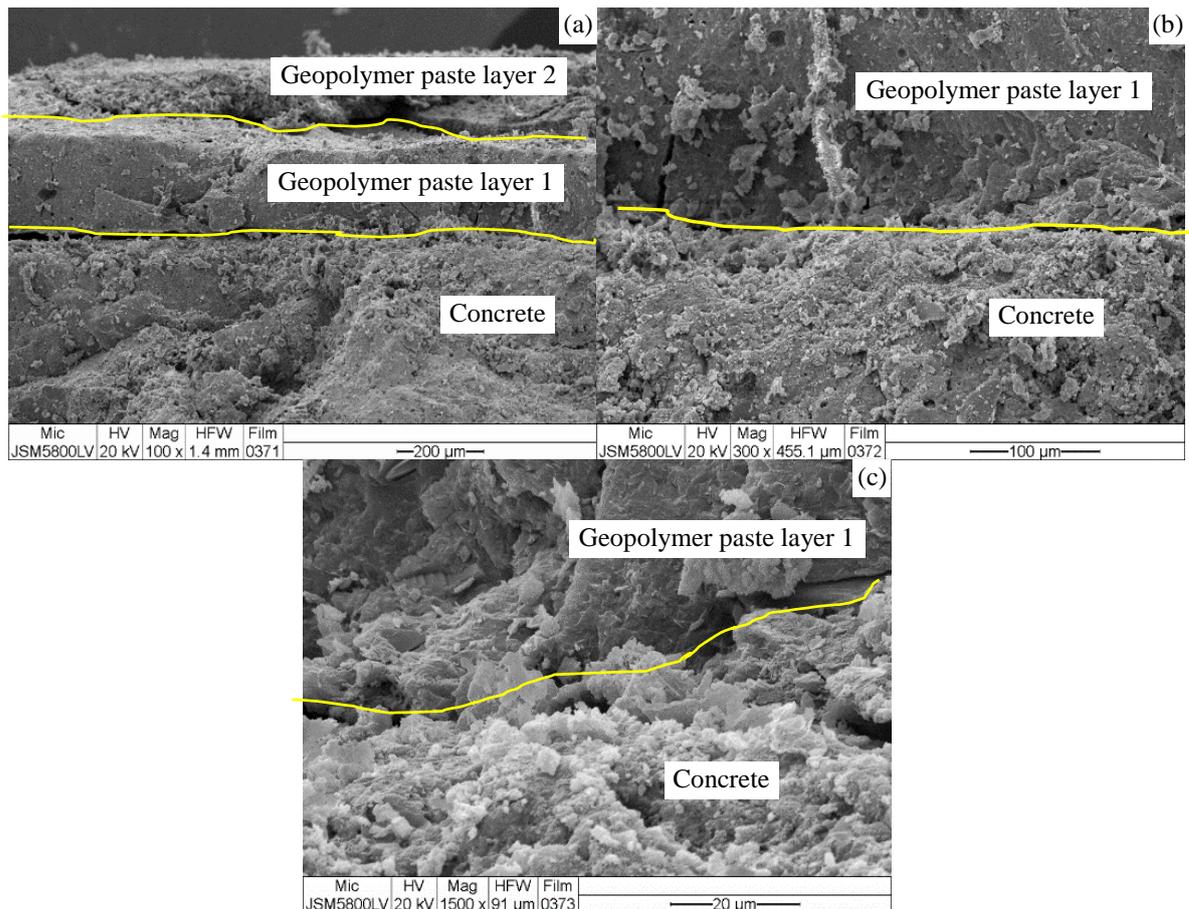


Figure 12 SEM images of geopolymer coatings with 5% FPRL double layers (a) 100x, (b) 300x, and (c) 1,500x

CONCLUSION

The use of geopolymer coatings with added field Para rubber latex was proposed for protecting reinforced concrete against corrosion from exposure to seawater with chloride ions. Based on the results presented in this study, the following conclusion can be stated:

1. The results indicate that the splitting tensile strength of concrete samples with geopolymer coating was higher than of concrete samples without coating.

2. The metakaolin-based geopolymer binder paste containing 1-5% field Para rubber latex gave slightly higher splitting tensile strength than the other cases tested.

3. The corrosion protection against steel weight loss by coatings with 1-5% field Para rubber latex in geopolymer was superior to that of the other cases.

4. The concrete specimens with 2 geopolymer coatings protected steel better than a single coating layer. This is partly due to each coating acting as a barrier, and partly due to the different curing required with two coatings compared to one coating.

5. Evaluation of accelerated corrosion tests suggests that geopolymer binder pastes can be used as protective coatings for concrete substrates exposed to seawater (chloride ions). Among the coatings evaluated, metakaolin powder combined with 1 to 5% field Para rubber latex exhibited the best performance. The accelerated testing for up to 14 days involved switching the 12 V potential on/off every 8 hours under seawater.

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