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Effect of Primer Layer and Curing Method on Geopolymer Paste Coating Properties

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ABSTRACT

This technical paper presents a comprehensive study on the properties of geopolymer coating applied to mild steel pipelines as a potential alternative to Ordinary Portland cement (OPC) concrete structures. The geopolymer paste was formulated using a mixture of fly ash and alkaline activators, specifically sodium hydroxide (NaOH) solution and sodium silicate (Na2SiO3) solution. Two types of primers, epoxy metal primer and self-etch primer were applied before the geopolymer coating and various curing conditions were investigated. The geopolymer samples were subjected to two different curing processes: one set was cured in an oven at 60°C for 24 hours while the other set was left to cure under ambient conditions. After the curing period, the samples were aged at ambient conditions for 28 days. The properties of the fly ash and geopolymer paste were evaluated through a range of tests including phase analysis, morphology analysis, optical emission spectroscopy (OES) analysis, chemical composition analysis and adhesion strength testing. The results revealed that the geopolymer coatings exhibited surface cracks and efflorescence attributed to unreacted sodium oxide. X-Ray diffraction (XRD) analysis confirmed the presence of quartz, hematite, magnetite, aluminum oxide and mullite in the geopolymer coating. The self-etch primer-coated samples demonstrated improved adhesion and corrosion resistance properties with a denser and more cohesive microstructure. The geopolymer coating when applied with a self-etch primer and cured at 60°C for 2 hours, achieved the highest adhesion strength of 2.2 MPa, indicating strong bonding with the mild steel pipelines. These findings contribute to the understanding of geopolymer coatings and their potential application in enhancing the performance and durability of mild steel pipelines, offering a sustainable alternative to conventional concrete coatings with improved corrosion resistance and adhesion properties.

Keywords: *Geopolymer coating, Mild steel pipelines, Primers, Curing conditions, Adhesion strength*

1. INTRODUCTION

Concrete holds an esteemed status due to its remarkable adaptability and impressive durability, making it an integral component in shaping the modern urban environment. Its widespread implementation in global infrastructure systems significantly enhances numerous individuals' well-being and overall quality of life [1]. However, the production of concrete, particularly cement manufacturing, has significant environmental implications. Cement manufacturing is one of the main contributors to CO2 emissions worldwide, releasing approximately 900 kg of CO2 per ton. Researchers have explored alternative materials and technologies to address the environmental concerns associated with traditional cement production. Geopolymer technology has emerged as a sustainable and cost-effective solution with numerous advantages over traditional cement-based materials [2]. Geopolymer coatings have gained attention for their superior performance and environmental benefits in various applications, such as petrochemical facilities, industrial settings, improved resistance to chemical attack and higher thermal stability [3].

Mild steel pipelines, used for transporting liquids and gases, are susceptible to corrosion when exposed to the surrounding environment [4]. Corrosion compromises the pipelines' structural integrity, wall thickness and overall strength [5]. Proper corrosion protection measures are crucial to ensure the longevity and reliability of these pipelines [6]. Primer coats play a vital role in the corrosion protection system for mild steel pipelines [3]. Two commonly used primer coats are self-etch primer and epoxy metal primer. Self-etch primers can etch the metal surface and form a strong bond, enhancing adhesion between the geopolymer coating and the mild steel substrate [7]. They are beneficial for priming various types of metal that require a short turnaround time. On the other hand, epoxy metal primers are widely used for their corrosion resistance properties and ability to promote adhesion [8]. They act as a sealer on various surfaces, protecting against external elements and removing contamination [9].

It is crucial to comprehend how various primer coats affect the effectiveness and durability of geopolymer coatings in providing corrosion protection [10]. The choice of primer coat can influence the adhesion strength, corrosion resistance and overall performance of the geopolymer coating on mild steel pipelines. Therefore, this study will evaluate self-etch primer and epoxy metal primer in combination with geopolymer paste coatings. The evaluation of geopolymer paste properties will include various testing methods, such as X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM), to examine the crystalline phases and microstructure of the geopolymer matrix. An adhesion test will also be conducted to measure the strength of the bond between the geopolymer coating and the mild steel substrate, indicating the coating's adhesion properties and ability to withstand external stresses.

This study explores the impact of primer coats on the properties of geopolymer coatings used on mild steel pipelines. The goal is to find the most practical combination of primer coats and geopolymer paste to enhance corrosion protection. The findings will contribute to developing effective corrosion-resistant solutions, prolonging the service life of pipelines and reducing maintenance costs. Moreover, this research aligns with advancing sustainable infrastructure development by minimizing the environmental impact of corrosion prevention strategies.

2. METHODOLOGY

2.1 Sample Preparation

Geopolymer paste was prepared using fly ash class C as the base material and mixed with alkaline activators, namely sodium hydroxide (NaOH) solution and sodium silicate (Na_2SiO_3) solution. Mild steel pipelines were divided into three sets: one without primer, one with epoxy metal primer and one with self-etch primer. The primers were applied to the surface of the pipelines using a brush technique. Following primer coating, the geopolymer paste was applied on top. The samples were then divided into two sets, with one set cured at ambient temperature and the other set cured at 60°C in an oven for two hours. After the curing process, the samples were placed under ambient conditions for 28 days. At the end of the 28-day period, the samples were subjected to phase analysis, morphology analysis and adhesion strength testing to assess their properties.

2.2 Elemental Composition Analysis

In the OES analysis conducted for elemental composition of the mild steel pipelines, a sparking method was utilized to vaporize a small amount of material and discharge an electrical charge to the sample. The resulting discharge plasma generated a distinct chemical signature, allowing for determination of the elemental breakdown of the sample. The analysis results are presented as a percentage distribution of the component parts of the mild steel pipeline.

2.3 Materials Characterization

Comprehensive characterization of the raw material and geopolymer paste was conducted through phase analysis, morphology analysis and adhesion strength testing. These analyses provided valuable insights into the composition, structure and performance of the raw material and geopolymer paste.

2.4 Phase Analysis

Phase composition and crystalline structure of the raw material and geopolymer paste were determined using X-Ray diffraction (XRD) analysis. The samples were subjected to X-Ray radiation and the resulting diffraction patterns were analyzed. A Bruker D2 Phaser instrument was employed for this analysis, with scanning performed in the 2θ range of 10° to 90° at a scanning rate of 0.2 $deg \cdot s^{-1}$. The obtained diffraction patterns were analyzed using the High Score Plus software to identify the different phases present in the materials.

2.5 Morphology Analysis

The physical structure and microstructural properties of the raw material and geopolymer paste were examined using a Scanning Electron Microscope (SEM). The samples were carefully prepared by coating them with a thin layer of platinum to enhance imaging quality. The SEM analysis was conducted at X100 magnification using a JEOL JSM-6010LV SEM instrument.

2.6 Adhesion Strength Test

The bond strength between different materials or layers was evaluated through adhesion strength testing using standard ASTM D4541. The pull-off adhesion test was employed using an Elcometer 108 apparatus. The samples were securely attached to the apparatus via a dolly and controlled force was applied until detachment occurred. The required force for detachment was measured and used as a quantitative measure of adhesion strength. The results were reported in MPa.

3. RESULTS AND DISCUSSION

3.1 Elemental Composition Analysis

The analysis of the elemental composition of the mild steel pipelines revealed that iron (Fe) is the dominant element, comprising 99.61% of the steel's weight, as depicted in Figure 1(a). In addition to iron, trace amounts of manganese (Mn), copper (Cu) and carbon (C) were identified, weighing 0.178%, 0.0098% and 0.066%, respectively. The lowest percentage was found for boron (B) at 0.00012%. Based on the analysis conducted using the ASTM A36/A36M standard, it can be inferred that the sample corresponds to low carbon steel. The chemical composition of the mild steel pipes used in this study adheres to the requirements outlined in the ASTM A36/A36M standard, as shown in Figure 1(b). All elemental compositions fall within the specified ratio ranges specified by the standard, providing evidence of compliance.

(a)

Element	Weight (%)	Element	Weight (%)	Element	Weight (%)
Fe	99.61	Al	0.044	Nb	0.0015
Mn	0.178	P	0.0064	Ti	0.0012
Cu	0.0098	Mo	0.0013	Sb	0.0015
C	0.066	O	0.010	Zr	0.0010
Si	0.011	S	0.0067	V	0.0011
Cr	0.012	Co	0.0078	Ca	0.0014
Ni	0.012	W	0.0061	B	0.00012
Sn	0.0013	N	0.0069	**Total**	**100**

(b)

Elements	ASTM A36/A36M (%)	Mild steel pipelines (%)
Carbon, C	Max 0.25	0.066
Manganese, Mn	Max 1.20	0.178
Sulfur, S	Max 0.04	0.0067
Phosphorus, P	Max 0.04	0.0064

Figure 1. (a) Elemental composition of mild steel pipelines and (b) chemical composition of ASTM A36/A36M steel and mild steel pipelines.

3.2 Chemical Composition Analysis

The chemical composition of the fly ash was determined using X-Ray fluorescence spectrometry (XRF) and is presented in Figure 2. The XRF analysis results indicate that the fly ash used in this study can be classified as class C fly ash, primarily due to its calcium oxide content, which exceeds 10% (19.1%). Previous studies have also reported on the chemical composition of class C fly ash, highlighting its suitability for geopolymerization processes and the production of geopolymer paste, mainly due to its higher calcium oxide content. These findings support the appropriateness of the fly ash utilized in this study as a suitable material for geopolymer coatings. Furthermore, when the fly ash was combined with a 10M sodium hydroxide (NaOH) solution, it resulted in the formation of a geopolymer paste with desirable properties. The chemical composition of the fly ash, in conjunction with the alkaline activator (NaOH), facilitated the geopolymerization process, leading to the formation of a stable and cohesive geopolymer material. The high concentration of NaOH played a crucial role in activating the fly ash and promoting the desired chemical reactions, ultimately contributing to the successful geopolymerization process. These findings underscore the significance of the fly ash-to-NaOH ratio in achieving optimal geopolymer properties for coating applications on mild steel pipelines.

Component	Weight Percentage (wt.%)
Silica Oxide (SiO_2)	36.7
Alumina Oxide (Al_2O_3)	18.7
Iron Oxide (Fe_2O_3)	17.2
Titanium Dioxide (TiO_2)	1.68
Calcium Oxide (CaO)	19.1
Potassium Oxide (K_2O)	1.78
Sulfur Trioxide (SO_3)	3.04

Figure 2. XRF analysis data for fly ash composition.

3.3 Phase Analysis

In Figure 3(a), the XRD analysis of the fly ash exhibited a broad hump from 20° to 45° 2θ, indicating the presence of amorphous components, consistent with previous studies [11]. The spectrum analysis confirmed the highest concentration of quartz (SiO2) phase in the fly ash. Figure 3(b) revealed the amorphous nature of the geopolymer matrix for samples cured at ambient temperature, with the presence of quartz, mullite and hematite minerals in line with prior research [4]. The optimized fly ash geopolymers showed improved matrix formation and decreased quartz content. Figure 3(c) demonstrated the presence of hematite (Fe2O3) and Fe3O4 phases on the surface of mild steel when treated with a self-etch primer and cured at 60°C for 2 hours, corroborating previous findings on the formation of a protective iron oxide layer [11]. These results validate the successful geopolymerization process and the role of the self-etch primer in enhancing the corrosion resistance properties of the coated substance.

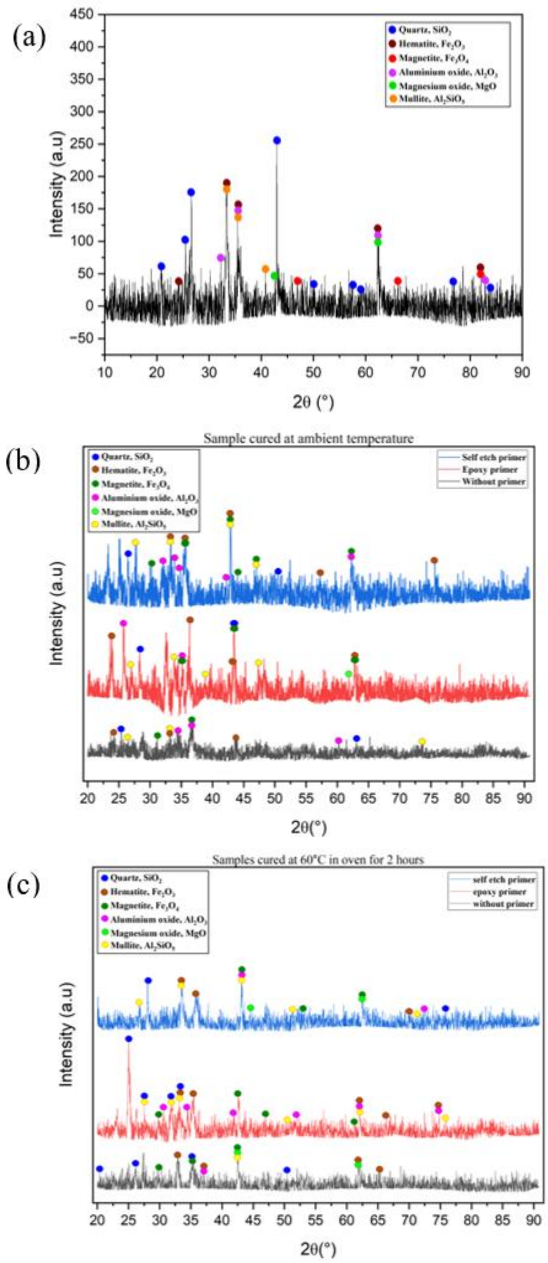

Figure 3 XRD analysis of (a) fly ash, (b) samples cured at ambient temperature and (c) samples cured at 60°C in oven for 2 hours.

3.4 Morphology Analysis

Figure 4(a) depicts some uneven fly ash particles. During combustion, gaseous components are ejected from the inside of the particle because of a rise in internal pressure or a decrease in external pressure [12]. The SEM micrographs in Figure 4 (b) to (f) and Figure 5 provide valuable insights into the microstructure of geopolymer coatings on mild steel pipelines. The sample cured at ambient temperature without primer (Figure 4(b)) exhibits the presence of unreacted fly ash particles and cracks, indicating incomplete geopolymerization. However, the use of a self-etch primer (Figure 4(c)) improves the microstructure with fewer pores and a higher degree of geopolymer binder, suggesting enhanced adhesion and corrosion protection. Similar improvements are observed with the sample using an epoxy metal primer (Figure 4(d)). Moving to samples cured at 60°C for 2 hours, the sample without primer (Figure 4(e)) still shows unreacted fly ash and cracks, while the sample with self-etch primer cured at 60°C (Figure 5) exhibits fully reacted particles, needle-shaped crystals and a dense structure, indicating a well-developed geopolymer matrix. The successful binding of quartz powder further strengthens the coating's properties. These findings support the influence of self-etch primer and elevated temperature curing on the microstructure of geopolymer coatings, reducing unreacted fly ash, promoting dense gel matrices and enhancing adhesion and durability [10]. Overall, the SEM analysis demonstrates that the use of self-etch primer and elevated temperature curing greatly influences the microstructure of geopolymer coatings, potentially leading to superior adhesion, durability and corrosion resistance compared to coatings cured at ambient temperature.

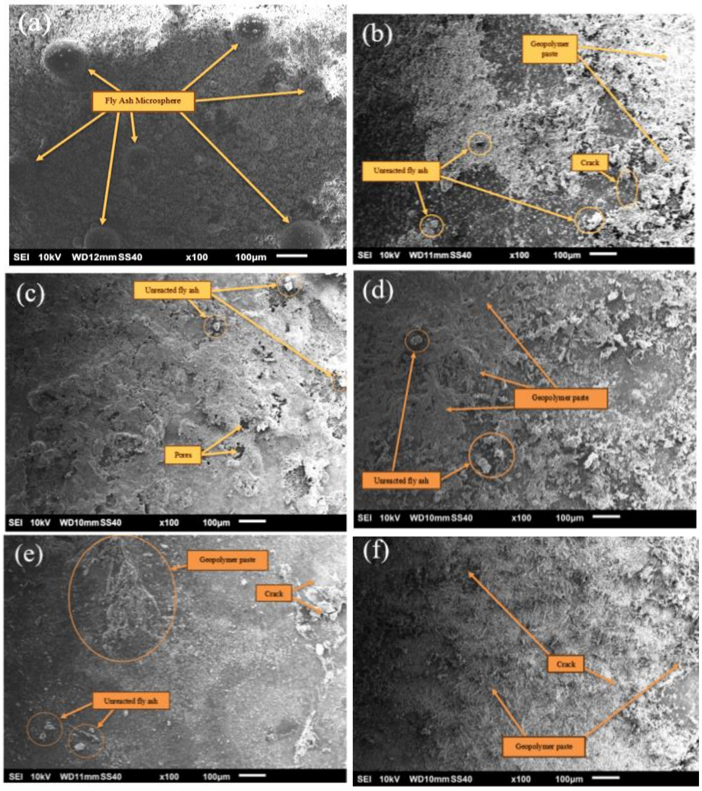

Figure 4. SEM micrograph of (a) fly ash, sample (b)without primer, (c) with self-etch primer, (d) with epoxy metal primer, cured at ambient temperature, sample (e) without primer, (f) with epoxy metal primer, cured at 60°C in oven for 2 hours.

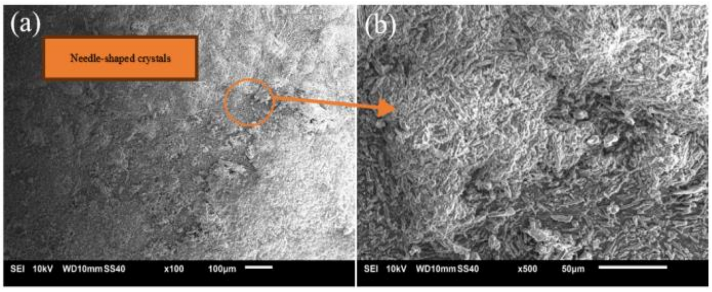

Figure 5. SEM micrograph of sample with self-etch primer cured at 60°C in oven for 2 hours.

3.5 Adhesion Strength Test

Figure 6(b) illustrates that during the adhesion test, the adhesive layer can either completely detach from the coating or experience internal tearing under applied force. To ensure the test's validity, it is essential for the coating to cover at least 50% of the dolly surface. A faulty test occurs when the top layer of the test dolly is either uncoated or has less than 50% coverage [9]. The adhesion strength tests were conducted after 28 days at ambient conditions using the Elcometer 108 instrument, following the ASTM D4541 standard. Among the samples, the highest adhesion strength of 2.2 MPa was achieved by the sample with self-etch primer cured at 60°C for 2 hours. The second highest value of 1.0 MPa was obtained by the sample with self-etch primer cured at ambient temperature. In contrast, the samples without primer exhibited low adhesion strength due to cracking and peeling of the geopolymer layer from the surface of the mild steel pipelines. Similar low adhesion strength was observed in samples with epoxy metal primer. Figure 6(a) displays the graph depicting the adhesion strength results of geopolymer coatings on mild steel pipelines with and without primer.

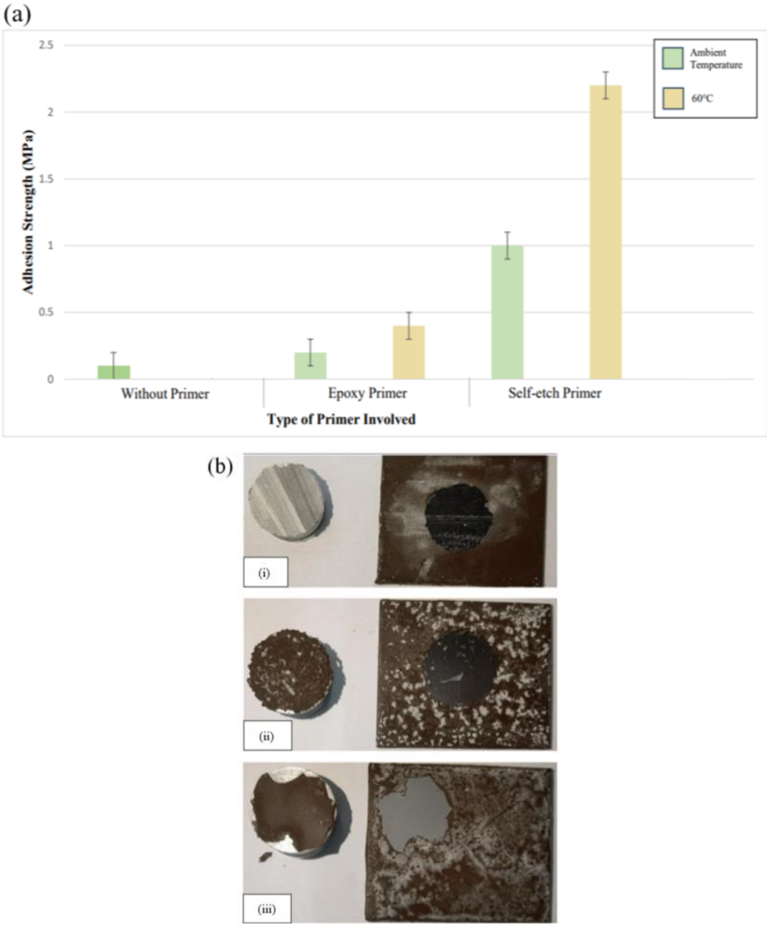

Figure 6. (a) Adhesion strength result of geopolymer coating on mild steel pipelines with and without primer and (b) Surface of dolly and sample after peel off test: (i) sample with self-etch primer, (ii) sample without primer and (iii) sample with epoxy metal primer.

4. CONCLUSION

In this study, the properties of geopolymer paste with and without a primer coat were evaluated, focusing on the curing method for mild steel pipelines. Extensive analysis was performed using X-Ray diffraction (XRD), scanning electron microscopy (SEM) and adhesion strength testing. The application of a primer aimed to enhance the adhesion layer between the geopolymer coating and the mild steel pipelines. Results provided valuable insights into the performance and characteristics of the geopolymer coating system, particularly when combined with a self-etch primer and cured at 60°C for 2 hours. Physical observations revealed surface cracks and efflorescence, attributed to the curing process and excess alkalis. These findings emphasize the importance of carefully considering curing conditions and geopolymer composition to ensure optimal performance and durability. Besides that, several factors can contribute to the development of surface cracks and efflorescence on various surfaces, especially in construction and masonry such as moisture issues, material quality, environmental conditions, structural issues, chemical reactions, design flaws and maintenance issues. Material characterization confirmed the presence of various compounds, including quartz, hematite, magnetite, aluminum oxide and mullite through XRD analysis. The presence of iron oxide particles indicated chemical precipitation in the self-etch primer-coated samples. Based on the comprehensive analysis, the geopolymer coating applied with a self-etch primer and cured under optimized conditions demonstrated the highest adhesion strength to mild steel pipelines, reaching 2.2 MPa. The self-etch primer positively influenced the morphology analysis, promoting a denser and more cohesive microstructure, suggesting improved adhesion and corrosion resistance properties. The cohesive fracture observed during adhesion tests further confirmed strong bonding between the geopolymer coating and the mild steel pipelines.

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