Study on the effect of surface modification of aluminum powder used in epoxy coating systems for corrosion protection of steel substrates

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Received 13 Aug. 2025; Revised 5 Oct. 2025; Accepted 16 Oct. 2025; Published 18 Nov. 2025.

DOI: https://doi.org/10.54939/1859-1043.j.mst.IMBE.2025.56-62

ABSTRACT

This study investigates the corrosion resistance of epoxy coatings containing aluminum (Al) powder surface-modified with silane coupling agents, namely 3-aminopropyltriethoxysilane (APTES), N-[3-(trimethoxysilyl)propyl]ethylenediamine, and 3-(2-aminoethylamino)propyl-dimethoxymethylsilane (KH-792). The modification process was carried out in isopropanol with sodium metasilicate to enhance the dispersion and compatibility of aluminum flakes with the waterborne epoxy matrix. The coatings were applied onto mild steel (CT3) substrates by spraying. Silane treatment improved filler distribution, increased adhesion, and reduced micro-defects, with the epoxy–Al–APTES system showing the best protective performance, as indicated by a positive shift of the corrosion potential (E_{corr}) and a significant decrease in corrosion current density (i_{corr}). The protection mechanism was attributed to the combination of physical barrier effects, sacrificial anodic protection of aluminum, and enhanced cross-linking through chemical interactions between silane and epoxy. The modified aluminum powders were characterized using SEM, EDX, FTIR, and XRD, while the corrosion resistance was evaluated by potentiodynamic polarization (PD) and electrochemical impedance spectroscopy (EIS) in 3.5 wt.% NaCl solution.

Keywords: Epoxy coating; Aluminum pigment; APTES; KH-792; Corrosion resistance; PDP.

1. INTRODUCTION

Epoxy-based coatings are widely recognized for their high adhesion, superior mechanical strength, and excellent chemical resistance, making them a preferred choice for corrosion protection of metals, especially steel [1]. These coatings function as physical barriers, isolating the metal surface from corrosive environments such as salt or acid solutions, thereby significantly reducing the corrosion rate. However, the incorporation of metallic pigments, particularly aluminum powder, into epoxy matrices has emerged as a promising strategy to further enhance the corrosion resistance of coatings [2, 3]. Aluminum powder serves as an effective metallic pigment in epoxy systems by improving barrier properties and providing cathodic protection through its role as a sacrificial anode [2, 4]. Studies have shown that the addition of aluminum powder, particularly in nano- or micro-scale, can increase coating density and reduce the permeability of corrosive agents, thus improving corrosion resistance [5]. Recently, research trends have focused on chemical surface modification of aluminum powder to address limitations such as poor dispersion, weak interfacial bonding, and incompatibility with epoxy matrices, which may lead to particle agglomeration and reduced protective performance [6, 7]. In this study, aluminum powder was surface-modified with the silane agents APTES and KH-792 to enhance compatibility and corrosion protection when incorporated into epoxy coatings on steel substrates. Comprehensive material characterization techniques, including XRD, FTIR, and PDP, were employed to investigate the crystalline structure, chemical characteristics, and electrochemical properties of the modified aluminum powders and the corresponding coatings. The objective of the modification process was to improve interfacial bonding and particle dispersion within the epoxy matrix, thereby enhancing the barrier effect against corrosive agents. The novelty of this work lies in the simultaneous investigation of two silane agents, APTES and KH-792, for aluminum powder modification - a research direction not previously reported. Notably, the use of an environmentally friendly waterborne epoxy not only meets the requirements for protective performance but also aligns with the trend toward the development of green, health-safe, and sustainable coating materials.

2. EXPERIMENT

2.1. Materials

Aluminum powder (≥ 99%, Xilong, China); N-(2-aminoethyl)-3 aminopropylmethyl-dimethoxysilane (KH-792, ≥ 99.5%); 3-aminopropyltriethoxysilane (APTES, ≥ 99%); ethanol (EtOH, Xilong); and sodium silicate (Na_2SiO_3, Xilong, China) were used. A commercial waterborne epoxy resin was employed as the coating matrix. Mild steel plates (φ = 90 mm) were used as substrates.

2.2. Experimental

2.2.1. Research methodologies

The morphology and elemental composition of the modified aluminum powder were examined using a scanning electron microscope (SEM), MIRA MLU (TESCAN), coupled with energy-dispersive X-ray spectroscopy (EDX). A Fourier-transform infrared (FTIR) spectrophotometer, Alpha II (Bruker) and X-ray diffractometers, D8 Advance (Bruker), were employed to identify chemical bonds and crystalline phases. The corrosion resistance of the coatings was evaluated by potentiodynamic polarization (PDP) measurements conducted in 3.5% NaCl solution using a three-electrode system (working electrode: epoxy-coated steel, counter electrode: stainless steel, reference electrode: Ag/AgCl). Data were processed with Nova 2.0 software to determine the corrosion potential (E_{corr}) and corrosion current density (i_{corr}).

2.2.2. Surface modification of Al

The synthesis process was carried out in three main steps. First, 3 g of aluminum powder was cleaned by stirring in 150 mL of acetone, followed by filtration, washing, and drying to obtain aluminum powder with a purified surface. Next, surface modification was performed by dispersing 2 g of the pretreated Al powder in 200 mL of isopropanol, then adding Na_2SiO_3, followed by the dropwise introduction of the silane coupling agent (KH-792 or APTES) at an amount corresponding to 1 wt% of the Al powder. Deionized water was added to promote hydrolysis, and the suspension was stirred at 60 °C for 24 h. The final product was filtered, washed with deionized water and ethanol, and vacuum-dried at 60 °C.

2.2.3. Method of manufacturing epoxy paint/modified aluminum powder system

The coatings were prepared from a commercial waterborne epoxy system (Water-based Metal Coat) containing 1 wt% silane-modified aluminum powder (Al–APTES, Al–KH792). Mild steel substrates (φ = 90 mm) were mechanically polished, cleaned with acetone, and dried at room temperature. The epoxy paint was diluted with deionized water to a viscosity of ~20 s (Ford Cup), after which the modified Al powder was dispersed by stirring. The coatings were applied by pneumatic spray, yielding a dry film thickness of approximately 20 µm.

3. RESULTS AND DISCUSSION

3.1. Structure and morphology of silane-treated aluminum powder

The XRD patterns (figure 1-a) show that all three samples-acetone-washed Al, Al–APTES, and Al–KH792—exhibit characteristic diffraction peaks of metallic aluminum at 2θ values around

38.54°, 44.76°, 65.13°, and 78.22°, corresponding to the (111), (200), (220), and (311) crystal planes, respectively. This indicates that the crystal structure of aluminum remains unchanged after surface modification. These peaks are clearly observed in all samples, and no new peaks appear, confirming that no impurity phases formed after modification.

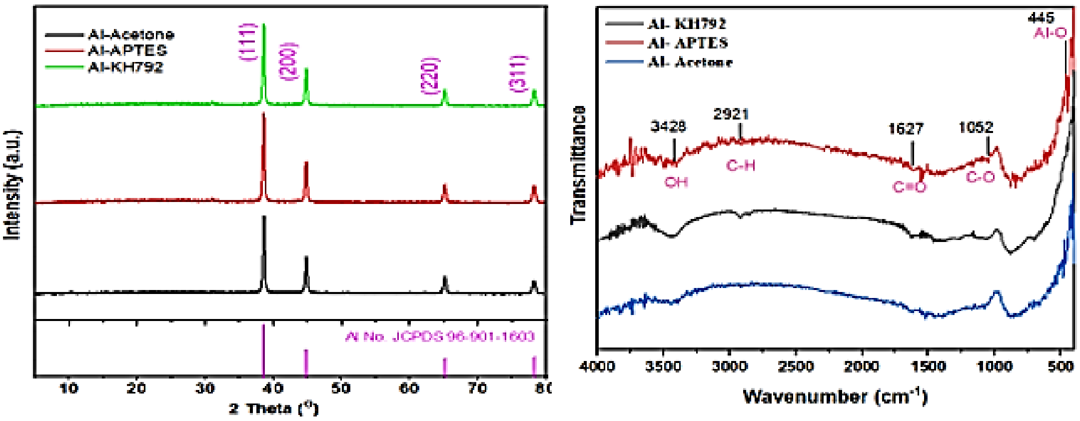
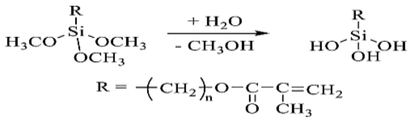
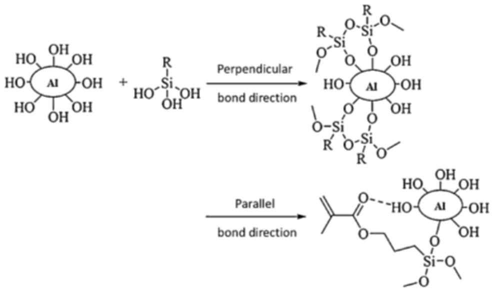


Figure 1. *(a) XRD patterns and (b) FTIR spectra of Al- Acetone, Al-APTES and Al-KH792.*

The modifier was successfully grafted onto the surface of aluminum flakes through chemical bonding, forming Al-O-Si bonds:



(1)



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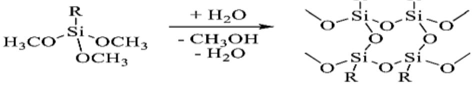
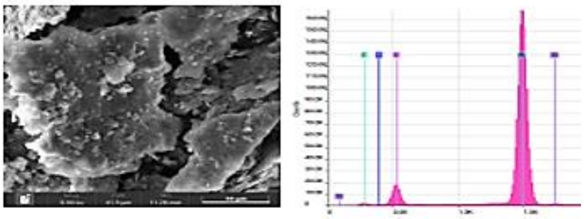


(3)

Figure 1b shows the FTIR spectra of Al-APTES, Al-KH792, and acetone-washed Al. The broad peak at 3428 cm^{-1} corresponds to -OH stretching, prominently observed in Al-APTES, indicating hydrolysis of alkoxysilane to silanol. The band at 2921 cm^{-1} is assigned to C-H stretching of hydrocarbon chains, typical of silane structures. The peak at 1627 cm^{-1} relates to C=O vibrations, possibly from surface oxidation, while 1052 cm^{-1} corresponds to C-O bonds, likely from Si-O-Si or C-O-C linkages. A strong absorption around 446 cm^{-1} confirms the presence of Al-O bonds, characteristic of the alumina structure. Among the samples, Al-APTES shows the most intense and numerous peaks, suggesting effective surface functionalization due to its $–NH_2$ and -OH-rich structure. Al-KH792 exhibits similar but weaker signals, and the acetone-washed Al shows mainly Al-O, indicating minimal surface modification [9].

Figure 2 presents the scanning electron microscopy (SEM) images combined with energy-dispersive X-ray spectroscopy (EDX) spectra of Al surfaces modified with two different silane agents: APTES (a) and KH792 (b). The Al-APTES sample shows a rough surface with agglomerated and uneven coating layers, whereas the Al-KH792 sample exhibits a more dispersed morphology with finer and more uniform particles. The EDX spectra of both samples reveal the presence of Si, O, C, and N elements, confirming the successful attachment of silane agents onto the aluminum surface. Notably, the higher Si signal intensity in the Al-APTES sample suggests a thicker silane layer compared to that of Al-KH792.

(a), Al-APTES



(b), Al-KH792

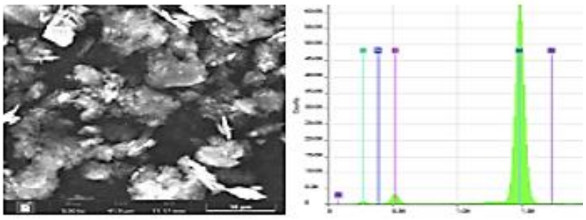


Figure 2. *SEM/EDX analysis of (a) Al- APTES, (b) Al-KH792.*

3.2. Corrosion properties

The potentiodynamic polarization results presented in figure 3 and table 1 indicate that the pure epoxy coating exhibited the poorest corrosion resistance, with a high corrosion current density (i_{corr}) and corrosion rate (CR), particularly after 21 days ($i_{corr} = 7.24\times10^{-4}$ mA/cm², CR = 8.425 mm/year). The incorporation of aluminum powder significantly improved the corrosion protection by reducing both i_{corr} and CR. Notably, the silane-modified aluminum samples (APTES, KH-792) showed superior performance, with a positive shift in E_{corr} and much lower i_{corr} and CR values. Among them, the epoxy–Al–APTES coating maintained the lowest i_{corr} (1.86×10^{-5} mA/cm²) and CR (0.217 mm/year) after 21 days, demonstrating the most stable and effective long-term corrosion protection [10]. The Bode plots presented in figure 4 reveal distinct differences in corrosion protection performance. The pure epoxy exhibited the weakest protective performance, with low-frequency impedance decreasing significantly from $\log|Z| = 2.95$ (day 1) to 2.05 (day 21), while the phase angle dropped from –43.8° to –31.5°, indicating a rapid deterioration of dielectric properties and barrier performance against electrolyte penetration. When unmodified aluminum was incorporated (epoxy–Al), the corrosion resistance improved compared to pure epoxy ($\log|Z| = 3.15 \rightarrow 2.74$), but the values still declined over time, suggesting that aluminum particle agglomeration led to a less homogeneous coating and facilitated corrosive ion ingress. In contrast, the epoxy–Al–KH792 coating maintained higher impedance values (3.21, 2.95, and 2.68 from day 1 to day 21) and relatively stable phase angles (–49.7° → –43.2°), demonstrating that KH792 functionalization improved interfacial bonding, reduced coating defects, and enhanced dielectric properties. However, the epoxy–Al–APTES system exhibited the most outstanding performance: its low-frequency impedance remained at the highest level (3.45, 3.27, and 3.12) with only a slight reduction in phase angle (–52.2° → –46.7°), consistently outperforming both KH792-modified and unmodified aluminum. These results confirm that APTES provided superior nanoparticle dispersion and stronger interfacial cross-linking with the epoxy network, leading to a denser coating structure and more effective resistance against electrolyte penetration, thereby ensuring the best long-term corrosion protection.

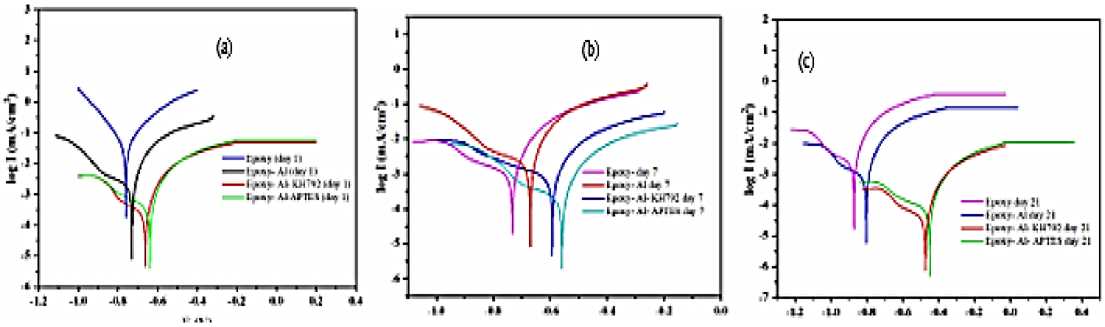
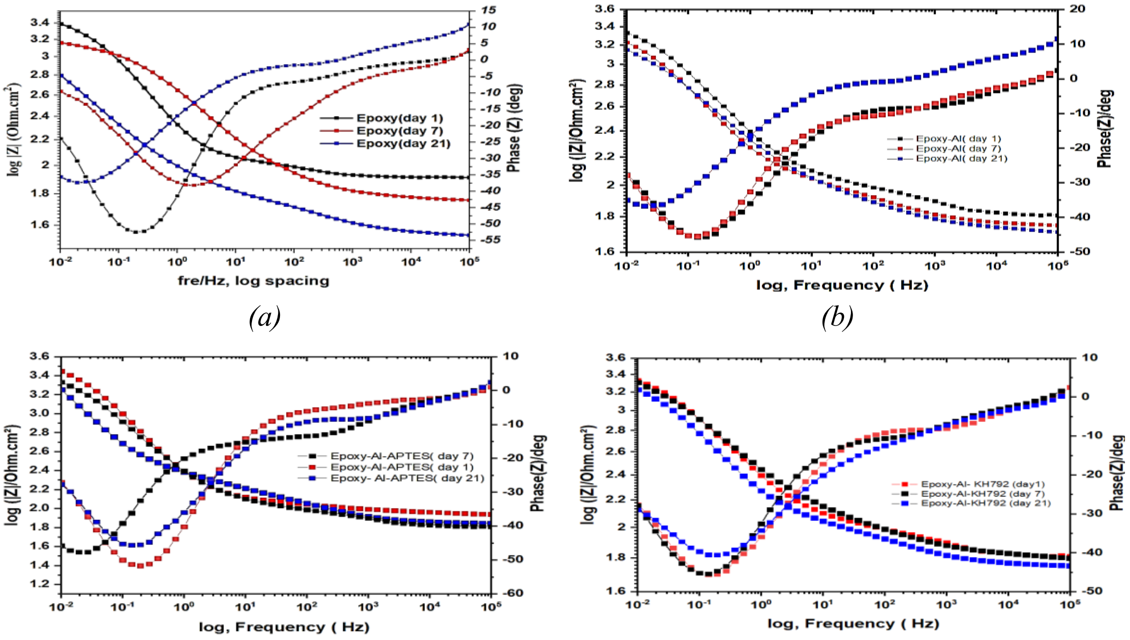


Figure 3. *Potentiodynamic polarization curves of nanocomposite coatings in 3.5% NaCl solution after (a) 1 day, (b) 7 days and (c) 21 days of immersion.*



(a) *(b)*

(c) *(d)*

Figure 4. *Bode plots of the pure epoxy (a), epoxy–Al- APTES (b), epoxy– Al- KH792 (c) after 1, 7 and 21 days immersion in 3.5 wt.% NaCl solutions.*

Table 1. *Electrochemical parameters of epoxy-Al coating samples.*

Time	Samples	E_{corr} (V)	i_{corr} (mA/cm^2)	CR (mm/year)
Day 1	Epoxy- Al-APTES	-0.63	3.90×10^{-5}	0.078
	Epoxy- Al-KH792	-0.66	3.20×10^{-5}	0.014
	Epoxy- Al	-0.72	1.20×10^{-4}	0.0036
	Epoxy	-0.75	6.76×10^{-4}	0.0045
Day 7	Epoxy- Al-APTES	-0.55	4.33×10^{-6}	0.05
	Epoxy- Al-KH792	-0.59	1.005×10^{-5}	0.116
	Epoxy- Al	-0.66	1.685×10^{-5}	0.195
	Epoxy	-0.73	9.73×10^{-4}	11.316
Day 21	Epoxy- Al-APTES	-0.44	1.86×10^{-5}	0.217
	Epoxy- Al-KH792	-0.47	2.40×10^{-5}	0.279
	Epoxy- Al	-0.80	2.82×10^{-5}	0.328
	Epoxy	-0.86	7.24×10^{-4}	8.425

4. CONCLUSIONS

This study successfully demonstrated the surface modification of aluminum powder using silane coupling agents (APTES and KH-792), as confirmed by SEM/EDX, FTIR, and XRD analyses, which revealed the presence of Al–O–Si bonds and characteristic functional groups. Particle size distribution indicated that aluminum was used in flake form with an average size of ~14 µm, and the modification process did not alter its original morphology. Electrochemical tests clearly confirmed the significant role of silane modification in enhancing the corrosion resistance of epoxy coatings. Pure epoxy exhibited the poorest performance, with i_{corr} = 7.24×10^{-4} mA/cm^2 and CR = 8.425 mm/year after 21 days. The addition of unmodified aluminum improved the protection (i_{corr} = 2.82×10^{-5} mA/cm^2; CR = 0.328 mm/year after 21 days), though the effect was limited due to particle agglomeration. The epoxy–Al–KH792 system showed further improvement (i_{corr} = 2.40×10^{-5} mA/cm^2; CR = 0.279 mm/year after 21 days). Most notably, the epoxy–Al–APTES coating demonstrated the best long-term protection, with i_{corr} as low as 1.86×10^{-5} mA/cm^2 and CR = 0.217 mm/year after 21 days, accompanied by the highest and most stable low-frequency impedance values ($\log|Z|$ = 3.45 on day 1; 3.27 on day 7; 3.12 on day 21). These results demonstrate that APTES-modified aluminum flakes significantly improve the electrochemical performance of waterborne epoxy coatings on CT3 steel substrates in chloride-rich environments.

Acknowledgment: *We acknowledge Institute of Tropical Technology, Academy of Military Science and Technology and the University of Technology Ho Chi Minh City for supporting this study.*

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TÓM TẮT

Nghiên cứu ảnh hưởng của các chất biến tính bề mặt bột nhôm, dùng trong hệ sơn Epoxy để bảo vệ chống ăn mòn cho nền thép

Nghiên cứu này khảo sát khả năng chống ăn mòn của lớp phủ epoxy chứa bột nhôm (Al) được biến tính bề mặt bằng các hợp chất silane, cụ thể là 3 aminopropyltriethoxysilane (APTES), N-[3-(trimethoxysilyl)propyl]ethylenediamine và 3-(2aminoethylamino)propyl-dimethoxymethylsilane (KH-792). Quá trình biến tính được thực hiện trong dung môi isopropanol có bổ sung natri metasilicat, nhằm cải thiện khả năng phân tán và tính tương hợp của vảy nhôm với nền epoxy gốc nước. Lớp phủ được tạo trên nền thép CT3 bằng phương pháp phun. Việc xử lý silane giúp cải thiện sự phân bố chất độn, tăng độ bám dính và giảm khuyết tật vi mô, trong đó hệ epoxy–Al–APTES cho hiệu quả bảo vệ tốt nhất với điện thế ăn mòn (E_{corr}) dịch chuyển về phía dương và mật độ dòng ăn mòn (i_{corr}) giảm đáng kể. Cơ chế bảo vệ được xác định là sự kết hợp giữa hiệu ứng rào cản vật lý, bảo vệ anot hy sinh của nhôm và tăng cường liên kết chéo nhờ tương tác hóa học giữa silane và epoxy. Các đặc trưng của bột nhôm biến tính được phân tích bằng các kỹ thuật SEM, EDX, FTIR và XRD, trong khi khả năng chống ăn mòn được đánh giá thông qua phương pháp phân cực thế động và phổ tổng trở điện hóa (EIS) trong dung dịch NaCl 3,5%.

Từ khoá: Bột nhôm; KH-792; APTES; Lớp phủ epoxy; Khả năng chống ăn mòn; PDP.