



Modification of Al pigment with graphene for infrared/visual stealth compatible fabric coating



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ABSTRACT

Al pigment was modified with graphene to reduce the lightness and visible reflectance via a three-step preparation method, including pretreatment of Al pigment with silane coupling agent, absorption of graphene oxide(GO), and reduction of GO. Al pigment was pretreated with silane coupling agent to introduce $-NH_2$ groups so that it will be able to combine with GO by ionic bands, which were confirmed by Zeta potential tests and FTIR spectra. The SEM images of Al pigment modified with reduced graphene oxide (Al-RGO) showed that RGO was absorbed onto the surface of Al pigment, which resulted in the reduce of lightness and visual reflection. Raman spectra conformed the multi-layer structure of RGO and indicated that a great number of defects remained in RGO sheets. After modification, the metallic lightness of Al pigment was significantly suppressed, and the color hue of Al pigment changed to dimgray from silver white. Al-RGO was used in low emissivity fabric coating with optimized visual property. In comparison with blank Al pigment fabric coating, the lightness L^* of Al-RGO coating was decreased by 49%, the visual light reflectivity was reduced by 23%, and the gloss was suppressed upto 58%, while the infrared emissivity in the range of 8–14 μm waveband only exhibited a slight increase of 0.12. Al-RGO works as a novel low infrared emissivity pigment with improved visual spectral stealthy compatible property.

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1. Introduction

Low infrared emissivity coating is a kind of functional coating with low infrared emittance, and this coating has been widely employed in vast applications, such as energy conservation buildings, solar collector systems, and military camouflage targets [1–3]. The controllability of infrared emissivity is of great significance for military targets, for the survive chance of them would drop rapidly once they were distinguishable from background [4]. Typically, in non-sunshine time, the infrared emissivity of green plant, withered grass or rock, and shrub is about 0.45–0.60, 0.65–0.75, and above 0.90, respectively [5]. To achieve low infrared emissivity coating, many efforts have been made over the past decades, such as preparing multilayer coatings [6], transparent conductive oxide compounds [7], polyurethane/titania nanocomposites [8], silver particles, and Cu and Al pigment coatings [9–11], among which aluminum flake pigment has received considerable attention due to its low price and excellent performance in reducing infrared

emissivity [12]. However, high visible lightness and glossiness of Al pigment lead to compatibility problems with visible and near-infrared light [13], which badly constrained the application range of Al pigment.

In order to control the lightness and glossiness of Al pigment, surface modification has been widely researched in recent years, and superficial treatment of Al pigment is considered to be an effective way to reduce the gloss and reflectivity of Al pigment [14]. High concentration of colored organic pigments were originally employed to modify the lightness and gloss of Al pigment, while most colored pigments were of high infrared emissivity due to their strong absorption in infrared waveband, leading to a distinct increment in infrared emissivity [15]. Surface pretreatment of Al pigment using polyethylene wax was able to suppress the gloss of Al pigment, but it resulted in the rapid increase of infrared emissivity. Fe_3O_4 and Prussian blue modification of Al pigment had been proved to be effective in reducing visible lightness of low infrared coating, and the lightness and visual light reflectivity could be decreased by 45% and 56% compared with uncoated flake Al pigment, but the infrared emissivity was also increased [16–18]. Optimizing the visual stealth property of Al pigment without

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damaging its infrared emissivity is a big challenge, but few research was devoted to investigating low infrared emission materials for Al pigment modification in the past decades.

Graphene, two-dimensional planar sheets composed by sp^2 -bonded carbon atoms, has gained immense recognition in many areas as a new material with vast merits in the past decades for its distinctive behavior in electrical, mechanical, and optical applications [19,20]. The graphene exhibits a level splitting energy spectrum due to free movement of electrons and the restraint from boundary effect, and during the electron transition process radiation in far, mid, and near infrared waveband would be absorbed, which makes graphene an excellent radiation absorber. The optical absorption of graphene arises from intraband and interband optical transitions, and in the mid-to near-infrared region, the absorbance is attributable primarily to interband transitions [21].

Due to the poor solubility of pure graphene sheets in aqueous solution, GO is usually chosen for reactions, following by a reduction process to obtain RGO. RGO exhibits low reflection in infrared waveband, and most infrared emissions can be absorbed due to enhanced infrared scattering from the pore-wall structure and high defects density of multi-layer RGO, especially multi-layer RGO [22]. The dark color of multi-layer RGO enhances its covering ability in visual waveband, making it a promising material for low emissivity Al pigment modification. However, no researches about modifying Al pigment with graphene for visual-infrared compatible stealth application has been reported.

In this work, a three-step method was employed to prepare Al-RGO to reduce the visual reflection and lightness. Because of the poor dispersity of graphene, GO was chosen to be absorbed onto the surface of cationic pretreated Al pigment through electrostatic attraction. Due to the excellent high infrared absorption and low visual reflection properties of graphene, the color hue, visual reflectance and lightness of Al pigment would be suppressed, without reducing the infrared emissivity property.

2. Materials and methods

2.1. Materials

Ethanol, ammonia solution (NH_4OH), acetone, isopropyl alcohol, (3-Aminopropyl)-trimethoxysilane (APTES), and 85% of hydrazine hydrate, all of AR grade, were provided by Sinpharm Chemical Reagent Co., Ltd. China. The multilayer GO was obtained from Hengqiu Graphene Technology Co., Ltd. China. The Al pigment was purchased from Zuxing New Materials Co., Ltd. China. The thickener PFL and Waterborne polyurethane resin 303C was supplied by Guanzhi Chemical Co., Ltd. China. The cellulosic fabric of 25×25 tex was purchased from Hongdou Co., Ltd. China.

2.2. Preparation of Al-RGO

Before the preparation of Al-RGO, the purchased Al pigment was purified to remove the organic grinding agents and improve its dispersity in aqueous solution. Typically, 1 g Al pigment was dispersed in 100 mL acetone solution under vigorous stirring for 30 min with T18 digital ULTRA-TURRAX disperser supplied by IKA group, Germany, the purified Al pigment was collected for the following treatment.

Al-RGO was prepared by a three-step preparation method, which includes cationic pretreatment of Al pigment, absorption of GO onto Al surface and the reduction of GO. 1 g purified aluminum powder was added into 100 mL isopropyl alcohol and stirred with T18 digital ULTRA-TURRAX disperser for 10 min, 0.02 g sodium silicate was used as disperser to get Al pigment dispersion. After that 0.1 g APTES was dropped into the dispersion, followed by the

addition of 0.2 mL deionized water, then the reaction solution was kept under 60°C to complete the hydrolyzation process of APTES and the reaction with the hydroxyl groups on the surface of Al pigment. A certain amount of 1 mg/mL GO water dispersion (GO: Al ratio 0–0.8) was added into the APTES pretreated Al pigment (Al-APTES) dispersion, and the Al-APTES reacted with GO at room temperature for 4 h to complete the absorption process. Although graphene sheets require certain surfactant to be well dispersed, GO sheets contain plenty carboxyl and the concentration of GO can reach 5 mg/mL without any disperser [23,24]. After the Al-GO was prepared, the Al-GO was collected and washed with deionized water for five times to remove the remained APTES and unabsorbed GO. UV–Visual absorbency curves of washing solutions (Fig. S1) confirmed that the APTES was removed completely. Finally, the Al-GO was dispersed in deionized water with the 1 mol/L of ammonia solution used to adjusting the pH to 10, then a certain amount of hydrazine hydrate was dropped in. After the reaction being kept at 90°C for 10 h with continuous stirring, Al-RGO was prepared.

2.3. Coating of Al-RGO on fabric

A certain amount of Al-RGO (0–10 wt%) was added into deionized water and stirred for 15 min, then the pigment paste was prepared by fully mixing the thickener PFL and binder (the Waterborne polyurethane resin 303C). No.5 coating stick (coating thickness $50\ \mu\text{m}$) was chosen to coat the pigment paste on cellulosic fabric with K202 coater, then the coated fabric was baked under 150°C for 90 s. The thickness of fabric coating was controlled by the coating times of No.5 coating stick.

2.4. Characterizations

The Zeta potential of Al pigment, APTES, Al-APTES, GO, and Al-GO were measured by ZS90 Nano-zeta potential analyzer (Malvern Co. Ltd., UK) to monitor the changes of charges during the preparing process. FTIR spectra of blank Al pigment, Al-APTES, Al-GO, and Al-RGO were measured by TENSOR-2 Fourier Transform interferometer (Bruker Co. Ltd., Germany) in $4000\text{--}500\ \text{cm}^{-1}$ to investigate the change of component of each step. Raman spectrum was recorded on an inVia Raman Microscope (Renishaw Pty Ltd., UK) at room temperature, exciting at 532 nm with Ar^+ radiation. The morphology changes in Al pigment surface were investigated by SEM and TEM taken by Su1510 scanning electron microscope (Jeol Ltd. Japan) and H-7000 transmission electron microscope (Hitachi, Co. Ltd, Japan), respectively.

Infrared emissivity in the range of $8\text{--}14\ \mu\text{m}$ wavelengths was measured by IR-2 dual-band infrared emissivity measuring instrument (Shanghai Wangjia optical instrument Co. Ltd, China). Microscope transmitting images of Al-RGO fabric coatings were taken by VW-6000E Motion Analysis Microscope (Keyence Co. Ltd., Japan). Reflectivity, lightness and K/S values of fabric coatings were determined with Xrite-8400 spectrophotometer (Xrite Co. Ltd, American) under illuminate D65 using the 10° standard observer. The gloss of Al coating and Al-RGO coating was measured by XGT gloss meter (Tianjin Xintian Electronic Technology Co. Ltd, China) with measuring angle of 60° .

3. Result and discussion

3.1. Preparation of Al-RGO

Al pigment has no adhesion to GO because both surfaces of Al pigment and GO were negatively charged. In order to reduce the electrostatic repulsion between Al and GO, Al pigment was

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