



## Regular article

# The self-regulated infrared emissivity properties of BCG/MgCl<sub>2</sub>/PEG-g-CDA powders for tunable IR signals



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## ABSTRACT

The green BCG/MgCl<sub>2</sub>/PEG-g-CDA powders were synthesized at different temperatures via liquid-phase reaction method. The microstructures of the synthesized powders were analyzed and characterized by FT-IR, XRD and ChemDraw software. The effects of synthesized temperatures on infrared emissivity properties of the powders in the wavelength range 3–5 μm were investigated systematically. It is found that the powders heated at 100 °C exhibit the optimum crystallinity at room temperature and thus show the largest variation (0.4) in infrared emissivity with the test temperature varying from 50 °C to 65 °C. Furthermore, the mechanisms for the self-regulated infrared emissivity properties are also explored thoroughly. The variation of infrared emissivity with temperature is ascribed to the changes in resistivity, which is induced by the transformation of PEG-g-CDA between crystalline and amorphous as well as the molecular structure transition of BCG between quinone and lactone. Our results suggest that BCG/MgCl<sub>2</sub>/PEG-g-CDA powders are potential candidates for intelligent infrared camouflage.

## 1. Introduction

With the rapid development of modern detection technology, military targets are facing great challenges to survive in the battlefield. It is reported that MAN Portable Air Defense Systems (MANPADS), especially the infrared (IR) guided missiles, have caused severe casualties to aerial vehicles in recent wars such as Gulf war and Afghanistan war [1]. Therefore, improving infrared stealth capability has become the key to enhance survivability of the military targets in modern battlefield.

Over the past years, a great number of researches and investigations have focused on characterizing and suppressing the IR signals of aerospace vehicles. Rao [2] modeled IR signal spectral from exhaust plume of a military aircraft and evaluated the effect of engine bypass ratio on IR signature. Wang and Li [3] did computational research on exhaust system of a helicopter with heat shelter nozzle. Rao and Mahulikar [4] assessed the effect of atmosphere on IR signatures of aircraft. Mahulikar et al. [5] investigated the influences of sun, sky and earth on IR signatures of aircraft and indicated that fuselage skin temperature has a tremendous impact on IR radiation of aircraft. Xia et al. [6] analyzed the impact of transient temperature fields of fuselage skin on total IR signatures of aircraft. Lu and Wang [7,8] pointed out the contributions of different fuselage parts to the whole IR signatures. These researches demonstrate that the IR signatures of the aerial targets are intrinsically

related to the environment, especially the temperature. Therefore, it is meaningful to control skin temperatures of the military targets to regulate the IR signatures.

As is known to all, infrared emissivity is an important physical parameter of materials. The materials with high emissivity values can dissipate heat and have an advantage in reducing the local temperature [9–11]. While materials with low emissivity values are advantageous to store heat and hence increase the local temperature [12–15]. Obviously, if the materials coated on the military targets could self-regulate the infrared emissivity to be high when the skin temperature is high and be low when the skin temperature is low, the whole skin temperatures of the targets would be adjusted to get closer to environment temperature and hence prevent the military equipment or vehicles to be detected by infrared detectors.

For decades, the ternary systems based on a leuco dye, a developer and their co-solvent were concentrated upon the properties of reversible thermochromism. Zhu [16], MacLaren [17,18] and Tang [19] reported the thermochromic properties of the ternary systems, which consist of crystal violet lactone, phenolic compounds and long chain alcohols. The results show that the composites can change colors between blue and colorless reversibly. Azizian F [20] studied thermochromic properties of fluorans and the results indicate that the compounds change colors from colorless to red reversibly. Li [21] and Du

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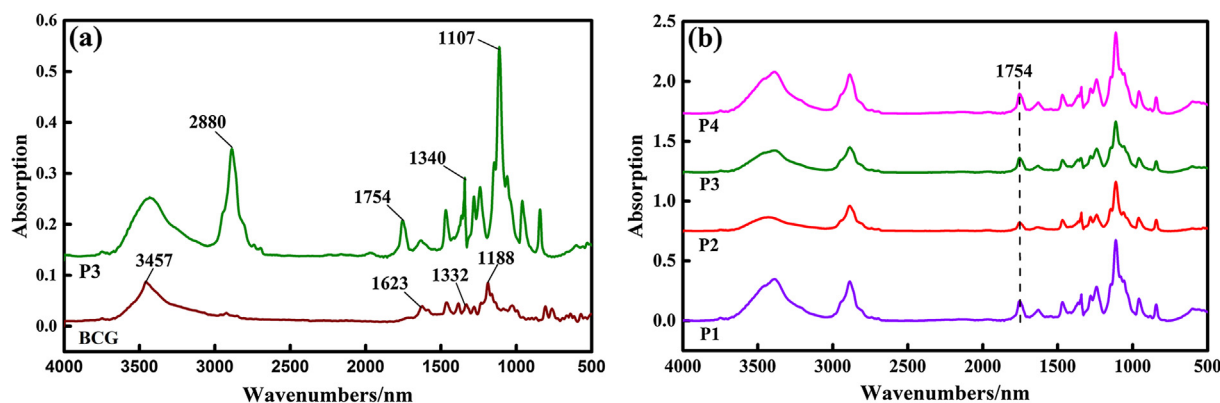


Fig. 1. (a) XRD patterns of P1, P2, P3, P4 powders at room temperature; (b) XRD patterns of P3 powder with measuring temperature raising from 30 °C to 70 °C.

[22] reported the thermochromic properties of Bromocresol green (BCG) and alkaline earth metals ion or transition metals ion ( $\text{Co}^{2+}$ ) in the solvents. However, among their studies, the infrared emissivity properties of the materials were ignored. Actually, the infrared emissivity properties of such ternary system materials have been rarely reported so far. Nevertheless, the changing crystallinity of solvents under temperature fluctuation would change resistivity of the ternary system materials simultaneously. Therefore, the ternary systems could also be promising candidates to achieve self-regulated infrared emissivity properties.

In this paper, BCG/ $\text{MgCl}_2$ /PEG-g-CDA powders, in which BCG,  $\text{MgCl}_2$ , PEG-g-CDA are typical leuco dye, developer and solvent respectively, were prepared with different synthesis temperatures. The impact of synthesized temperature on infrared emissivity properties of the materials are investigated in detail. The mechanisms for the self-regulated infrared emissivity properties are also analyzed deeply in this work. Ultimately, the powders synthesized at 100 °C show the largest infrared emissivity variation ( $\sim 0.4$ ) when the test temperature varies from 50 °C to 65 °C. The results indicate that they are potential candidates to regulate IR signatures of the aerial vehicles.

## 2. Experimental details

### 2.1. Materials

Bromocresol green (BCG), polyethylene glycol (PEG-4000), and cellulose diacetate (CDA) were purchased from Chengdu Aike Reagent Co., Ltd (Chengdu, China). Magnesium chloride ( $\text{MgCl}_2$ ) and Acetone were purchased from Aladdin Reagent Co., Ltd (Shanghai, China). All the chemicals were used as received without any further purification.

### 2.2. Experiment content

#### 2.2.1. Synthesis of BCG/ $\text{MgCl}_2$ /PEG-g-CDA

BCG/ $\text{MgCl}_2$ /PEG-g-CDA powders were synthesized by liquid phase reaction method. The weight ratio of BCG,  $\text{MgCl}_2$ , PEG and CDA was 1:1:17:6. Firstly, PEG-4000 was placed into a three-neck flask and heated at 60 °C in oil bath. After PEG-4000 being melted completely, BCG and  $\text{MgCl}_2$  were added into the three-neck flask. The mixtures were heated at 70 °C, 85 °C, 100 °C, 115 °C in oil bath for 2 h and then dried at room temperature for 12 h in a vacuum oven.

Afterwards, 6 g CDA and 250 mL acetone were added into the resultant mixtures and stirred for 6 h at room temperature. Finally, the BCG/ $\text{MgCl}_2$ /PEG-g-CDA powders were obtained by centrifuging with a rate of 3500 rpm for 15 mins and then air-drying at room temperature. The obtained powders prepared at 70 °C, 85 °C, 100 °C, 115 °C were marked with P1, P2, P3 and P4, respectively.

#### 2.2.2. Characterization

The X-ray diffraction patterns of the BCG/ $\text{MgCl}_2$ /PEG-g-CDA powders were monitored by the Philips X'Pert Pro 3040/60 diffractometer at  $2\theta$  range of  $10^\circ \sim 40^\circ$  and temperature range of 30–70 °C in the air, which is equipped with graphite, filtered  $\text{Cu K}\alpha$  ( $\lambda = 1.5406 \text{ \AA}$ ) radiation. The infrared spectra were recorded in a wavenumber range of  $4000\text{--}500 \text{ cm}^{-1}$  using the FT-IR 6300 spectrometer at room temperature. The  $E_{\text{HOMO}}$  and  $E_{\text{LUMO}}$  of the quinone and lactone structured BCG were calculated by ChemDraw software. The DC electrical conductivity was carried out by the two-probe direct current method from room temperature to 200 °C, the test method is based on the reference [23]. The temperature-dependent infrared emissivity values in  $3\text{--}5 \mu\text{m}$  of the powders were measured from room temperature to 200 °C with an interval of 10 °C by using the IR-2 Infrared Emissometer (Shanghai Institute of Technological Physics, China) and BC-1 temperature control instrument.

## 3. Results and discussion

### 3.1. XRD analysis

To investigate the impact of different synthesized temperatures on the crystallinity of P1, P2, P3 and P4 powders, XRD analysis was performed at room temperature and the patterns are shown in Fig. 1a. Clearly, strong and sharp diffraction peaks around  $2\theta$  degrees of  $19.2^\circ$  and  $23.4^\circ$  were observed, which is corresponding well with PEG-4000 [24] and indicates that crystallinity of the powders is mainly controlled by PEG-4000 [25]. However, it can be seen that the diffraction intensity of the powders enhances firstly from P1 to P3 and then diminishes from P3 to P4 under identical test conditions. Moreover, the full width at half maximum (FWHM) narrows from P1 to P3 but broadens from P3 to P4 gradually. This implies that crystallinity of the powders increases firstly from P1 to P3 and then decreases from P3 to P4 [26].

Fig. 1b shows the high temperature X-ray diffraction (HTXRD) pattern of P3 at temperature range of 30–70 °C. It is obvious that the diffraction peaks referring to PEG-4000 are still remained below 50 °C, though they are weakened and broadened a little with the measuring temperature increasing. It means that P3 still possesses a large amount of crystalline phase in this temperature range. However, only a broad and dispersion peak is detected with temperature further rising from 50 °C to 70 °C, which indicates that the powders are converted to amorphous phase in this temperature range.

### 3.2. Infrared spectroscopy

Fig. 2a depicts the FTIR spectra of pure BCG and P3 powders. The bands at  $3457 \text{ cm}^{-1}$  and  $1623 \text{ cm}^{-1}$  are associated with stretching vibration and bending vibration of  $-\text{OH}$  group of BCG, correspondingly. For pure BCG powders, the bands at  $1188 \text{ cm}^{-1}$  and  $1332 \text{ cm}^{-1}$  are

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