International Journal of Biological Macromolecules 89 (2016) 190–197

Contents lists available at ScienceDirect

International Journal of Biological Macromolecules

journal homepage: www.elsevier.com/locate/ijbiomac

Synthesis of cuprous oxide epoxy nanocomposite as an environmentally antimicrobial coating

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ARTICLE INFO

Article history:
Received 24 February 2016
Received in revised form 13 April 2016
Accepted 15 April 2016
Available online 19 April 2016

Keywords:
Cu_2O nanoparticles
Antimicrobial protection
Thermal stability

ABSTRACT

Cuprous oxide is commonly used as a pigment; paint manufacturers begin to employ cuprous oxide as booster biocides in their formulations, to replace the banned organotins as the principal antifouling compounds. Epoxy coating was reinforced with cuprous oxide nanoparticles (Cu_2O NPs). The antibacterial as well as antifungal activity of Cu_2O epoxy nanocomposite (Cu_2O EN) coating films was investigated. Cu_2O NPs were also experimented for antibiofilm and time—kill assay. The thermal stability and the mechanical properties of Cu_2O EN coating films were also investigated. The antimicrobial activity results showed slowdown, the growth of organisms on the Cu_2O EN coating surface. TGA results showed that incorporating Cu_2O NPs into epoxy coating considerably enhanced the thermal stability and increased the char residue. The addition of Cu_2O NPs at lower concentration into epoxy coating also led to an improvement in the mechanical resistance such as scratch and abrasion. Cu_2O NPs purity was confirmed by XRD. The TEM photograph demonstrated that the synthesized Cu_2O NPs were of cubic shape and the average diameter of the crystals was around 25 nm. The resulting perfect dispersion of Cu_2O NPs in epoxy coating revealed by SEM ensured white particles embedded in the epoxy matrix.

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1. Introduction

The addition of hard particles into polymers to improve their physical and mechanical properties is very common. The most common method for improving the coating performance is reducing the filler size from submicron particles to nanoscale [1,2]. Nanoparticles (NPs) have the potential to overcome the drawbacks associated with the incorporation of submicron particles, because of their inherent small size and particle morphology [3,4]. The incorporation of inorganic nanoparticles in surface coatings has been found to improve the thermal, mechanical, electrical, corrosion resistance and antimicrobial resistance of the coatings without disturbing their other properties [5,6]. The improvement of the inorganic nanocomposite properties depends on filler type, size, shape, degree of dispersion of NPs in the polymer matrix and degree of adhesion of NPs with polymer chains [7,8]. Metal oxide nanoparticles have shown their great interest in the coating field due to their unique physical and chemical properties. The most commonly used inorganic NPs in coatings are nano- silica SiO_2 and nano- alumina Al_2O_3 [9,10] which are used to improve scratch and abrasion resistance of the coating; whereas nano-titania TiO_2 [1] and nano-zinc oxide ZnO [11] are most commonly used as UV blocking agents; iron oxide Fe_2O_3 [12] and calcium carbonate $CaCO_3$ nanoparticles [13] are commonly used to improve corrosion, mechanical and the heat resistance of the coating, respectively; and cuprous oxide Cu_2O nanoparticles has been widely exploited for use in antifouling coatings [14]. Generally, there are two methods by which inorganic NPs are dispersed/incorporated in polymeric materials, either as additives by direct mixing with polymer, or as reactive materials by in situ polymerization. Additive- type is widely used; it's generally incorporated into the polymer matrix by physical means. It's an economical and expeditious way of promoting nanocomposite coatings [15]. Epoxy coating is one of the most well-known protective coatings that are widely used in industrial fields. Its level of corrosion protection and chemical resistance are high. However, in spite of a successful application, pure epoxy coating is often susceptible to moisture attack and mechanical scratch [16,17]. It also shows poor resistance to the initiation and propagation of cracks

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http://dx.doi.org/10.1016/j.ijbiomac.2016.04.043
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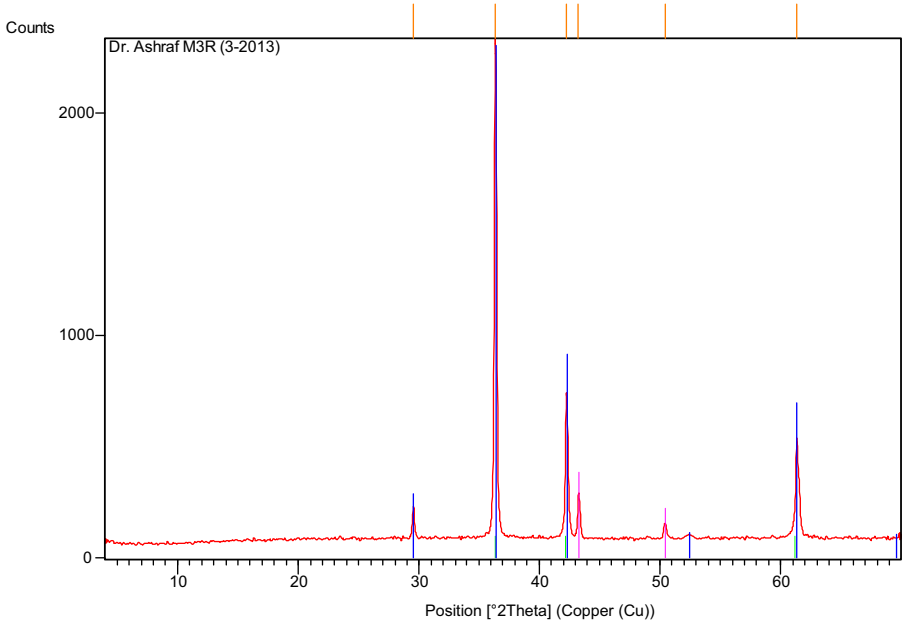

Fig. 1. XRD patterns for Cu_2O NPs.

due to a high cross- linked structure. Therefore, many efforts have been made by researchers to overcome these drawbacks of epoxy, by adding various nanofillers to epoxy coating [18]. In recent years, epoxy antimicrobial coatings are of great interest for protection of surfaces. Therefore, the development of epoxy nanocomposite coatings with antimicrobial properties is essential. In this study, we reported the synthesis and characterization of Cu_2O NPs. We focus on studying the influence of its direct dispersion as reinforcing nano filler, into a commercial two component epoxy coating, at different loading levels, to optimize antimicrobial activity and to enhance thermal stability and mechanical properties of the epoxy coating.

2. Experimental

2.1. Materials

All the materials used in this work were sourced internationally, or from local companies, were of analytical grade and were used without any further purification. Commercial two component, epoxy coating was used as the polymer matrix. The typical characteristics of the epoxy coating are as follows: epoxy equivalent weight (184–190 g/eq), viscosity at 25 °C (12.0–14.0 Pa.s), density at 25 °C (1.16 kg/l) and mixing ratio by volume of base to hardener is 4:1.

2.2. Methods and techniques

2.2.1. Synthesis of cuprous oxide nanoparticles (Cu_2O NPs)

Cu_2O NPs were prepared as reported in the previous work by L. Huang et al. [19], using liquid phase chemical synthesis method; method no. 2. The crystalline structure of the obtained yellow Cu_2O NPs powder was characterized by XRD and its average diameter was calculated by TEM.

2.3. Characterization study of the prepared Cu_2O NPs

2.3.1. X-ray diffraction (XRD) analysis

Crystal structure of the prepared Cu_2O NPs was identified by X-ray diffraction (XRD) patterns using a PANalytical X'pert PRO (The Netherlands), employing Cu-Kα radiation at 50 kV and 200 mA. Scattering reflections were recorded for 2Θ angle between 4 and 70 corresponding to *d*-spacing between 1.47 and 3.26 A°. To confirm the resolution of the diffraction peaks with standard reproducibility in 2-theta (±0.005), the sample measurement was recorded by using a monochromator and detector, which were used to generate focusing beam geometry and parallel primary beam. The standard diffraction data was identified according to the International Centre for Diffraction Data (ICDD) software with PDF-4 release 2011 database.

2.3.2. Transmission electron microscope (TEM) analysis

High resolution transmission electron microscopy (HRTEM) of Cu_2O NPs was conducted at an accelerated voltage of 200 KV electron microscopes (JEM2100 LaB6, Japan) which has a point-point resolution of 0.14 nm. In the HRTEM the solid sample was dispersed in ethanol solution using an ultrasonicator and then dropped on a copper grid coated with carbon film prior to inserting the samples in the HRTEM column, the grid was vacuum dried for 15 min.

2.4. Preparation of Cu_2O epoxy nanocomposite (Cu_2O EN) coating films

Cu_2O EN coating films were prepared by dispersing different loadings of Cu_2O NPs in the epoxy coating. The first step was to disperse Cu_2O NPs at different loading levels 0.1–1.0 wt.% by continuously sonication in acetone solvent, using a sonicator model Sonics & Materials, VCX-750, USA. The sonicator utilized a frequency of 20 kHz, equipped with a 13 mm diameter titanium probe, for 30 min. The second step involved the mixing of the dispersed Cu_2O NPs with epoxy base component for 30 min, by means of stirring. The prepared samples were mixed with the correct ratio of the epoxy hardener component under continuous stirring. The resulting solution was degassed for 15 min, to remove any dissolved gases and any air bubbles formed during mixing of the solution. The degassed solution was allowed to stabilize for 10 min and directly applied to steel panels and filter paper using conventional spraying. All efforts were made to maintain a uniform dry film thickness (DFT) of 75 ± 5 μm for evaluating the antimicrobial, thermal stability and mechanical properties.

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