



Short communication

Green fabrication of ultraviolet curable epoxy acrylate-silica hybrid coatings

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ABSTRACT

Ultraviolet (UV) curable organic-inorganic hybrid materials have attracted increasing researchers' attention due to the environmental advantages and extensive application potential. In this work, a novel green fabrication of UV curable epoxy acrylate (EA)-silica hybrid coating via a sol-gel process induced by photochemical reaction was presented. The inorganic phase, silica was formed at present of an iodonium salt and tetraethyl orthosilicate, avoiding the application of acid or base in traditional synthesis method. The hybrid coatings were characterized by Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM) and thermogravimetric analysis (TGA). Three kinds of silane coupling agents including KH-550, KH-560 and KH-570 were investigated to modify the silica sol. The distribution of SiO₂ in the hybrid coating with KH-570 modification is most homogeneous. TGA results indicate that the hybrid coating with 10% of silica exhibits the most excellent thermal stability. The effects of adding content of silica on the hybrid film's tack free time and hardness were also investigated for the preliminary study.

1. Introduction

UV curing technology is an approach of obtaining the cured coatings by the polymerization of the reactive monomers induced by UV irradiation. Because of its advantages of low energy consumption, zero VOC emission, and fast cure rate at room temperature, UV curing technology has extensively attracted increasing researchers' attention in recent decade [1,2]. In general, UV curable coatings consist of four basic compositions including the photoinitiator, monomer, reactive diluent and assistant additives [3]. Under UV irradiation, the photoinitiator firstly decomposed into the free radicals or cations with chemical activity. Subsequently, the free radicals or reactive cations could initiate the polymerization between the monomer and reactive diluent with the unsaturation double bonds or epoxy bonds [4]. Another crucial function of the reactive diluent is viscosity regulation of the coating system for an applicative value [5]. The assistant additives, such as the pigment, filler, defoamer and flattening agent, play a supplementary role of improving the performance for utilization [6].

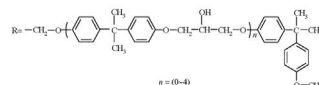
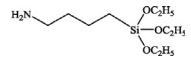
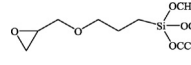
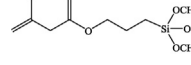
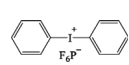
In order to expand the application range of UV curable coatings, it is a promising concept to develop inorganic/organic hybrid coatings by introducing the inorganic phase into UV curable organic system, which can improve the various properties of coatings by means of synergistic effect between organic and inorganic phase. So far, previous studies have reported some preparation methods of UV curable inorganic/organic hybrid coatings including blending [7], sol-gel method [8],

intercalation [9], in-situ synthesis [10], and etc. The same core idea of these methods is to make the inorganic phase in nanoscale at least one-dimensional be dispersed in organic coatings, namely below 100 nm. In this way, the hybrid coatings would probably exhibit more excellent dispersity, more ordered structure, and stronger interaction between organic and inorganic phases. In general, the formation process of inorganic phase needs to be carried out in the presence of acid or basic catalyst. For instance, Lai et al. [11] prepared thermoplastic polyurethane-silica hybrid materials by an acid-catalyzed sol-gel process. Didier et al. [12] prepared the hybrid polyimide-silica materials using ammonium hydroxide as a basic catalyst for inorganic phase formation. Yeh et al. [8] reported the preparation of waterborne polyurethane-silica by a sol-gel process without an external catalyst. The amino-terminated anionic waterborne polyurethane was first synthesized as the monomer and at the same time it played the role of an internal catalyst.

In this work, we presented a novel green fabrication of UV curable EA-SiO₂ hybrid coatings via a sol-gel process induced by the photochemical reaction. Firstly, silica sol was prepared under UV irradiation using diphenyliodonium hexafluorophosphate (C₁₂H₁₀F₆IP) as the cation photoinitiator, benzophenone and triethanolamine as the assistance photoinitiators, tetraethyl orthosilicate as the silicon source. Subsequently, silica sol was applied in UV curable EA coatings after modification by silane coupling agent. The innovation of this route is that the inorganic phase was formed by photochemical method,

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Table 1
Molecule structures of main materials in the experiments.

Abbreviation	Chemical names	Molecule structures
EA	epoxy acrylate	$\text{H}_2\text{C}=\text{CH}-\text{C}(=\text{O})-\text{O}-\text{CH}_2-\text{CH}(\text{OH})-\text{R}-\text{CH}(\text{OH})-\text{CH}_2-\text{O}-\text{C}(=\text{O})-\text{CH}=\text{CH}_2$ 
TPGDA	tripropylene glycol diacrylate	$\text{H}_2\text{C}=\text{CH}-\text{C}(=\text{O})-\text{O}-\left(\text{CH}_2-\text{CH}(\text{CH}_3)-\text{O}\right)_3-\text{C}(=\text{O})-\text{CH}=\text{CH}_2$
KH-550	γ -aminopropyltriethoxysilane	
KH-560	3-glycidyloxypropyltrimethoxysilane	
KH-570	methacryloxypropyltrimethoxysilane	
DPIHFP	diphenyliodonium hexafluorophosphate	

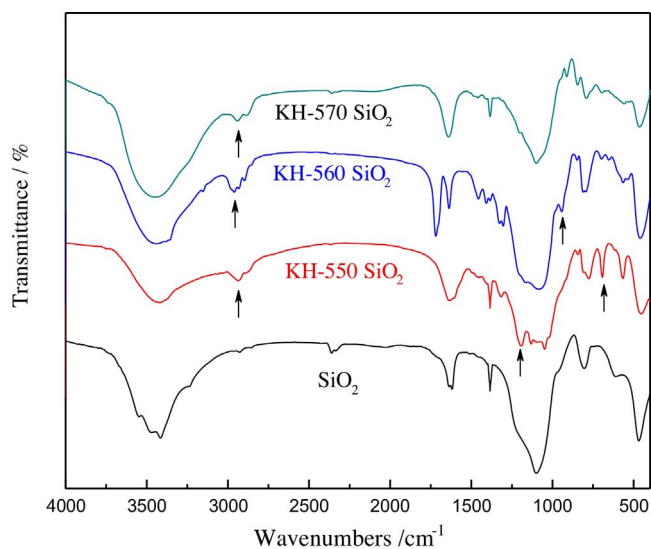


Fig. 1. FT-IR spectra of silica sol with modification.

avoiding the application of acid or base in traditional synthesis method.

2. Experimental

2.1. Materials

Benzophenone (BP), triethanolamine (TEA), anhydrous ethanol, tetraethyl orthosilicate (TEOS) were purchased from Sinopharm Chemical Reagent Co. Ltd. Bisphenol A epoxy acrylate (EA) was

purchased from Jiangmen ever-ray Co. Ltd. Tripropylene glycol diacrylate (TPGDA) was purchased from Tianjin Institute of Chemical Reagent. Diphenyliodonium hexafluorophosphate (DPIHFP) was purchased from TCI Co. Ltd. The silane coupling reagents, γ -aminopropyltriethoxysilane (KH-550), 3-glycidyloxypropyltrimethoxysilane (KH-560) and methacryloxypropyltrimethoxysilane (KH-570) were obtained from Shanghai Yaohua Chemical Company. All reagents were used as received without further purification. The molecule structures of main materials in this work are listed in Table 1.

2.2. Preparation of silica sol

In our previous work [13], we successfully synthesized silica nanoparticles by a photochemical method. The preparation of silica sol was in accordance with the previous work. Typically, 0.2 g of BP and 0.2 g of DPIHFP were dissolved in 20 mL of 50% aqueous ethanol solution. Subsequently, 20 g of TEOS, and 1 mL of TEA were added. The mixture was kept for 8 min under UV irradiation (the intensity is 50 mW/cm²) after ultrasonication for 5 min. After UV irradiation, the flavescent colloid was present. The mixture was sealed with a parafilm and kept for 2 h during magnetic stirring. Afterwards, 5% of silane coupling agent hydrolysate was added to the equal weight of colloid in order to modify the surface and enhance the hydrophobicity. Three kinds of silane coupling agent including KH-550, KH-560, and KH-570 were used, respectively. The final modified sol was removed into a vacuum oven to evaporate the redundant water and ethanol.

2.3. Curing process of hybrid coatings

The main coating compositions were EA and TPGDA (4:5 of weight ratio). The photoinitiators were 3% of BP and 1% of TEA. The various

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