



Preparation and electrical properties of polypyrrole containing photocured thiol–ene based composites

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ABSTRACT

In this study, UV curable polypyrrole containing thiol–ene based composite films were prepared by the reaction between Propoxylated (2) neopentyl glycol diacrylate (SR 9003), Trimethylolpropane tris(3-mercaptopropionate), (3-mercaptopropyl)trimethoxysilane, 1-vinyl-2-pyrrolidinone and polypyrrole. FT-IR was used to confirm the formation of the composites. Thermal stability of the samples was evaluated by thermogravimetric analysis (TGA). The samples were characterized with the following analysis; gel content, water absorption capacity. Hydrophobicity of the samples was determined by the contact angle measurements. Direct current (dc) conductivity measurements of samples were performed depending on polypyrrole content in the composites at room temperature. Capacitance measurements (40 Hz–100 kHz) of the samples were performed in the temperature range of 295–378 K. Dc conductivity and capacitance values of the samples were measured in vacuum in dark. Dc conductivity measurements revealed that dc conductivity values increased with increasing polypyrrole content. Dielectric measurements revealed that dielectric constant of the samples increased with increasing temperature and decreased with increasing frequency. It is also observed that dielectric constant of the samples decreased with increasing polypyrrole content.

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1. Introduction

UV curing method has the characteristics such as fast speed in curing, low power consumption and low curing temperature. The UV cured film has excellent performance, and the curing process has small environmental pollution [1]. The UV-curable materials were mainly used in the fields such as coatings, inks and adhesives. In UV-curable systems, mostly acrylate based resins are used owing to their great reactivity [2].

Thiol-ene photopolymerization reaction has many advantages such as insensitivity to oxygen inhibition, rapid reaction rates, homogeneous networks and high yields [3,4]. In addition, as a result of step-growth mechanism, high viscosities are observed only at high conversions [5].

The photocured thiol–ene networks exhibit narrow glass transition regions and excellent mechanical and thermal properties. One of the attractive features of thiol–ene photopolymerization is that

many types of enes and thiols can be incorporated into thiol–ene networks [6].

PPy is one of the most extensively studied conducting polymer for commercial applications because of its excellent properties, such as good electrical conductivity, excellent environmental stability and ease of polymerization [7,8]. PPy has many application areas such as light emitting diodes [9], lithium ion batteries [10], gas sensors [11] and supercapacitors [12].

In one study PPy/MWCNT (polypyrrole/multiwall carbon nanotubes) composites were prepared. The thickness of PPy film had a great effect on the electrical properties of polymer/CNT composites [13]. In another study polypyrrole/polypyrrole coated short nylon fiber/natural rubber composites prepared by in situ polymerization in latex. The thermal stability of the composites increases on loading with PPy and PPy coated fiber. The conducting composites prepared show substantial improvement in other microwave properties like dielectric heating coefficient, skin depth and absorption coefficient [14]. In our knowledge, preparation of photocured polypyrrole containing sol–gel hybrid composites and DC conductivity characterization have not been reported yet. The aim of this study was to develop novel photocured thiol–ene system and investigate electrical, thermal and hydrophobic properties of

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Table 1
The formulations of the composites.

Sample	SR9003 (g)	TMPMP (g)	MPTMS (g)	NVP (g)	PI ^a (g)	PPy (wt%)
F0	1.48	1.20	0.27	0.37	0.10	–
F1	1.48	1.20	0.27	0.37	0.10	0.5
F2	1.48	1.20	0.27	0.37	0.10	1.0
F3	1.48	1.20	0.27	0.37	0.10	1.5
F4	1.48	1.20	0.27	0.37	0.10	2.0
F5	1.48	1.20	0.27	0.37	0.10	2.5

^a (mixture of 50% camphorquinone and 50% irgacure 184).

the system by adding polypyrrole. UV curable polypyrrole containing thiol–ene based composite films were prepared by the reaction between propoxylated neopentyl glycol diacrylate (SR 9003), trimethylolpropane tris (3-mercaptopropionate) (TMPMP), 3-(Mercaptopropyl) trimethoxy silane (MPDMS) and N-vinyl pyrrolidone (NVP). FT-IR was used to confirm the formation of the composites. Thermal stability of the samples was evaluated by thermogravimetric analysis (TGA). The samples were characterized with the following analysis; gel content, water absorption capacity. Hydrophobicity of the samples was determined by the contact angle measurements. Dc conductivity and dielectric response of the samples were also studied depending on content of polypyrrole in the composites and frequency and temperature in the temperature range of 295–378 K, in vacuum.

2. Experimental

2.1. Materials

Propoxylated neopentyl glycol diacrylate (SR 9003) was supplied by Sartomer. Trimethylolpropane tris (3-mercaptopropionate) (TMPMP) was purchased from Aldrich, 3-(Mercaptopropyl) trimethoxy silane (MPDMS), N-vinyl pyrrolidone (NVP) and polypyrrole were purchased from Sigma–Aldrich. 1-Hydroxy cyclohexyl phenyl ketone (Irgacure 184) and Camphorquinone were obtained from Ciba Specialty Chemicals.

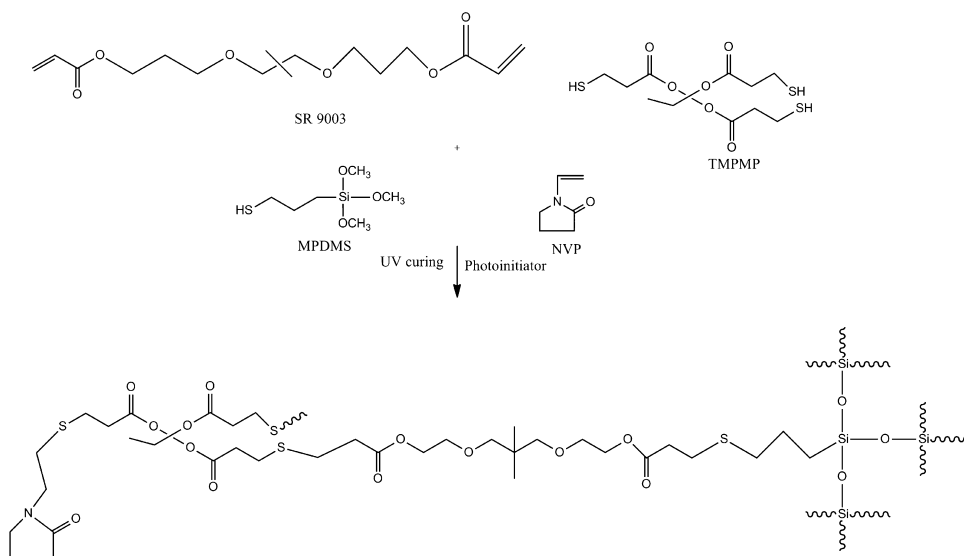
2.2. Preparation of the composites

UV-curable hybrid formulations were prepared by mixing propoxylated neopentyl glycol diacrylate (SR 9003), trimethylolpropane tris (3-mercaptopropionate), 3-(Mercaptopropyl)

trimethoxy silane, N-vinyl pyrrolidone (NVP) and various amounts of undoped polypyrrole used as filler. SR 9003 used as a crosslinker and NVP used to homogeneously disperse to PPy in the formulation. 3-(Mercaptopropyl) trimethoxy silane used for making organic/inorganic hybrid coating, 50% Camphorquinone and 50% Irgacure 184 mixtures were added to formulations as photoinitiator. The composition of all formulations is given in Table 1. Each formulation was prepared in a 10 mL beaker with adequate stirring to obtain homogeneous mixtures. The prepared mixtures were poured onto Teflon mold in order to obtain hybrid free films and irradiated with a high pressure UV-lamp (OSRAM 300 W, λ_{\max} at 365 nm; the distance between the lamp and the sample was 25 cm) for 3 min. The composites were annealed at 100 °C for 24 h in order to achieve high degree cross-linking via condensation of remaining silanol groups. Photopolymerized composite films are highly uniform with dense network structures as depicted in Scheme 1.

2.3. Preparation of samples for electrical characterization

Both dc conductivity and capacitance measurements of the samples were carried out in a sandwich structure form (Al/Composite/Al). Circular shaped electrodes were deposited onto both sides of samples using Edwards auto 500 thermal evaporation system in high vacuum ambient ($<2 \times 10^{-6}$ mbar). Aluminum was used as electrode material. Thickness of the metal electrodes was measured by film thickness monitor (FTM7) of the thermal evaporation system. Radius of the metal electrodes was 2.5 mm. In order to investigate effects of the content of polypyrrole in the composites on dc conductivity and dielectric constant properties, six samples were prepared, undoped sample F0, samples of polypyrrole doped with 0.5–2.5% (wt.%) in the composites, F1–F5, respectively.



Scheme 1. Preparation of the composites.

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