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Compatibility and biodegradability studies of linseed oil epoxy and PVC blends

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ABSTRACT

With the aim to utilize linseed oil epoxy – a product from sustainable resource for the processing of hard polymers, blends of linseed oil epoxy (LOE) with polyvinylchloride (PVC) were prepared for the first time by solution blending of the requisite amounts of the two components in the weight ratios LOE/PVC- 85/15, 65/35, 55/45, 45/55, 35/65, and 15/85 through solution method by mixing in tetrahydrofuran (THF). The miscibility of the two components in solution phase was investigated by relative viscosity, reduced viscosity and density measurements. The blend with 85 wt% of LOE yielded stiff and flexible films with maximum stress of 33 MPa and 216% elongation at break. Biodegradability of these blends has been studied by soil burial test, enzymatic degradation, and hydrolytic degradation in phosphate buffer. The blend LOE/PVC 85/15 showed the highest biodegradation of 68 wt% in 6 months in the soil burial test. The degradability was found to increase with the increase in LOE content in the blend compositions. The potential applications for such sustainable resource based blend films include packaging materials and biodegradable plastic sheets which can be formed into products such as bio-bags.

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1. Introduction

Vegetable oils of non-edible seeds constitute a major resource for several polymeric resins such as alkyd, polyesters, polyurethanes, epoxies and others [1–4]. In addition to their other applications, vegetable oil based products like epoxies and esters are also used as plasticizer and additives to commercial polymers to improve upon their toughness, tensile strength and biodegradability [1,2]. Linseed oil is a yellowish drying oil derived from the dried ripe seeds of the flax plant (*Linum usitatissimum*). Its uses include – animal feeds, putty, sealants, caulking compounds, brake linings, linoleum, textiles, foundry products, leather treatment, polishes, varnishes and oil paints, animal care products, wood preservation and synthetic resins. Medium-chain-length polymers derived

from linseed oil have a relatively small molar mass and contain a high concentration of unsaturated side-chains. As such, these polymers take on the consistency of a viscous liquid at room temperature. In order to increase the application potential of this material, the side-chain olefinic groups are converted to epoxy. Epoxidation helps in the formation of crosslinked brittle product with higher durability than the original materials.

The formulation of polymer blends has been an active area of research for the past three decades owing to the enhancement in the physical and mechanical properties of the blends achieved via synergism [5–7]. The physical as well as chemical properties of the blend depend on the degree of miscibility of the blend components. Although even immiscible or partially miscible blends have found commercial applications, the

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complete miscibility of the components in the blend is most desirable because mixing on molecular scale results into superior physical as well as mechanical properties with change in composition [8–10]. Polyvinylchloride is a well-known commercially viable and non-biodegradable polymer, which makes rigid transparent films and sheets. However, its major drawback is its brittleness and non-biodegradability [11].

Several attempts have been made to improve upon its fracture resistance by blending it with elastomers like natural rubber [12], polyurethane [PU] [13], ethylene polypropylene rubber [14] polybutadiene [PB] [15,16], high-density polyethylene [17], poly (acrylic acid) [18], polypropylene [19], ethylene vinyl acetate [20], polycarbonate [21], poly (amide) [22]. The morphology, miscibility and mechanical properties of these blends are well documented. In these blends, the problem of immiscibility and phase separation is overwhelmingly encountered that ultimately hampers the synergistic properties of the blend [23]. Alternatively, the improvement in the toughness, flexibility and tensile strength of a polymer of high molar mass can also be achieved by blending it with a substance of low molar mass. Low molar mass products from vegetable resource like lactose, starch cinnamate and dehydrated castor oil epoxy (DOCE) have also been used to modify the properties of poly (acrylic acid), poly (methylmethacrylate) and other polymers [1,23–25].

In the first instance, we have attempted to study the miscibility of linseed oil epoxy (LOE) and polyvinylchloride (PVC) blends by viscosity, and density measurements. The mechanical properties of the blends were analyzed by tensile strength measurements. Vegetable oil epoxy does not make freestanding films in ambient condition. The blend of a vegetable oil epoxy with PVC will expectedly yield freestanding films and sheets even with higher amount of former in the blend. Moreover, the synergism in the properties of the two components will be commercially profitable particularly when they are biodegradable in nature besides being compatible with each other.

2. Experimental

2.1. Materials

Linseed oil epoxy (LOE) and PVC were purchased from M/s Atul Chemicals Pvt. Ltd. Delhi (India). The oil was dewaxed by keeping it in a refrigerator at 15 °C and filtering before use. LOE was prepared after a reported method, which yielded epoxy equivalent of 175 [2]. PVC was purified by solvent non-solvent method. The molar mass of PVC was determined by viscosity and was found to be 1,40,000 Da. Tetrahydrofuran (THF) (Merck, India), glacial acetic acid (Merck, India), benzene (Merck, India), hydrogen peroxide (Merck, India), diethyl ether (Merck India), acetic anhydride (Merck, India), sulphuric acid (Merck, India) were used without further purification.

2.2. Preparation of LOE

Linseed oil (40 ml), benzene (40 ml), and glacial acetic acid (8 ml) were taken in a 3-necked conical flask equipped with a mechanical stirrer, burette and thermometer. Conc.

sulphuric acid (1 ml) was added to the reaction mixture immersed in a cold water bath maintained at 0 °C. Hydrogen peroxide (50%) (50 ml) was added drop wise through the burette with continued stirring. The temperature of the reaction was maintained at 0 °C during the addition of hydrogen peroxide. Then the temperature was raised to 50 °C and was maintained for 1–2 h. The progress of the reaction was monitored by determining epoxy equivalent and TLC at regular intervals. The epoxy equivalent reached a final value of 175. The product obtained was then washed with water and ether using separating funnel. The ethereal layer containing oil epoxy was separated with the help of rotary vacuum evaporator at reduced pressure.

2.3. Preparation of LOE/PVC blends

The blends of LOE with PVC were prepared by mixing the epoxy and the polymer in weight ratios LOE/PVC- 85/15, 65/35, 55/45, 45/55, 35/65 and 15/85 by taking the requisite amounts of the two components to obtain 100 ml of 4 wt%, 6 wt% and 10 wt% solutions of the blend in THF. All solutions were thoroughly mixed by agitation on a magnetic stirrer for 2 h. Selected samples were kept for over a week. Appearance of turbidity or precipitation was not noticed.

2.4. Preparation of LOE/PVC films

Solutions (6 wt%) of selected composition of the blend in THF were cast on transparency sheet and were allowed to dry under ambient conditions. In 10 days freestanding films were obtained. The films were further dried in vacuum oven kept at 60 °C for 10 h.

3. Characterization

Viscosity of the blend solutions was measured at temperatures 20 °C, 30 °C and 40 °C (accuracy = ± 0.05 °C) in a thermostatic bath using an Ubbelohde suspended level viscometer. The densities of the solutions were measured at the above temperatures by a pycnometer.

3.1. Morphological studies

The morphology of across-section of the cryogenically fractured blend films were examined by the scanning electron microscope (SEM, Jeol, JSM-840 SEM) at different magnifications. SEM Micrographs were taken after coating the fractured surface with a thin layer (10–20 nm) of gold.

3.2. Tensile strength measurements

Tensile strength studies were performed on Instron digital tensile tester interfaced with a PC. Five samples were tested for each composition. The maximum stress and elongation percentage at that point were taken to be the average of the five tests for each composition.

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