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Bio-based Photopolymer Synthesis: Epoxidized Soybean Oil (ESO) Modified with Itaconic Acid and Vinyl Acetate

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Abstract This study aimed to synthesize a bio-based UV-curable polymer, itaconated epoxidized soybean oil (IESO), using epoxidized soybean oil (ESO) and itaconic acid (IA) as replacements for acrylic polymers. Vinyl acetate (VAc) was added to improve the polymerization rate, adhesion, and water resistance of IESO. The itaconated epoxidized soybean oil/Vinyl acetate (IESO/VAc) content was adjusted to ratios of 9:1, 7:3, and 5:5, then IESO/VAc coated paper and stand-alone films were produced using a UV curing system. Fourier transform infrared spectroscopy analysis confirmed a decrease in C=C bond intensity after UV curing, indicating successful radical polymerization. The adhesion test demonstrated stable adhesive performance, and the water contact angle confirmed that the coating exhibits significant hydrophobicity. In the recycling test, all samples dissolved, indicating that IESO/VAc is a bio-based material that can be chemically recycled. Consequently, IESO/VAc blends have the potential to replace fossil-based curable polymer and may be used as coatings for paper packaging.

Keywords Photopolymer, Bio-based, IESO/VAc, UV-coated paper, Chemical recycling

1. Introduction

Global greenhouse gas (GHG) emissions are projected to reach 1.34 gigatons per year by 2030 and 2.80 gigatons per year by 2050[1]. In this context, UV curable coatings have gained global attention due to their "5E" advantages: efficient, enabling, economical, energy-saving, and environmentally friendly nature[2,3]. Compared to thermosetting solvent-based adhesives and waterborne systems, UV curable resins can reduce carbon dioxide (CO_2) emissions by up to 87%[4]. According to a recent report, the global industrial coatings market is projected to grow from approximately $ 157.2 billion in 2024 to $ 210.2 billion by 2032, with a CAGR of 3.7%[5]. Given this rapid market growth, UV curable coatings are expected to gain widespread adoption in the paper packaging industry, where enhanced barrier properties are increasingly needed.

UV-curable resins, consisting of oligomers, monomers, photoinitiators, and additives, undergo photopolymerization, which can be classified into cationic and radical polymerization [6,7]. Upon UV irradiation, photoinitiators generate free radicals or strong acids, which initiate the polymerization of oligomers and monomers. Cationic polymerization proceeds slowly and continues even after UV irradiation ends, whereas radical polymerization cures rapidly and stops immediately once UV exposure ceases.

Acrylate-based polymers derived from fossil resources raise concerns due to their high volatility, skin irritation, and corrosiveness[8]. Therefore, the demand for renewable and non-toxic alternatives has been increasing. To mitigate the toxicity concerns of conventional acrylic acids, acrylated epoxidized soybean oil (AESO) has been synthesized by esterifying epoxidized soybean oil (ESO) with acrylic acid [9,10]. The resulting AESO enables UV radical cross-linking due to terminal C=C bonds, while the long ESO chains improve the intrinsic brittleness of acrylates[9, 11]. The introduction of functional monomers such as methacrylated vanillin, myrcene, and methacrylated eugenol has enhanced mechanical and thermal properties[12–14]. However, residual acrylic acid in AESO production leads to undesirable odor and severe irritation to the skin and respiratory system[8]. For this reason, to replace the potential toxicity of AESO, research on more eco-friendly and non-toxic itaconated epoxidized soybean oil (IESO) has also been conducted [8,11,15].

ESO is a renewable, non-toxic, and hydrophobic material, making it a potential solution to overcome the limitations of bio-based plastics. Itaconic acid (IA), which contains both carboxyl and C=C functional groups, has structural similarities with acrylic acid and is considered a promising bio-based alternative. The U.S. Department of Energy has recognized it

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as one of the top 12 value-added chemicals derived from biomass[8]. Vinyl acetate (VAc) is an inexpensive material known for its adhesive properties, hardness, and ability to improve curing speed when used as a monofunctional monomer[16,17].

In this study, the epoxy rings of ESO were opened by an ester exchange reaction with the hydroxyl groups of IA to synthesize IESO, an eco-friendly UV-curable oligomer capable of radical polymerization. Subsequently, VAc was added and cured. The properties of the itaconated epoxidized soybean oil/Vinyl acetate (IESO/VAc) films and coated paper were characterized using Fourier transform infrared spectroscopy (FT-IR), thermogravimetric analyzer (TGA), surface analysis, and chemical recyclability assessment. This research aims to develop a sustainable alternative to conventional acrylate-based UV-curable resins by utilizing bio-based components and evaluating their potential applicability in environmentally friendly coating systems.

2. Materials and Methods

2.1. Materials

Epoxidized soybean oil (ESO) was purchased from Aladdin Scientific (Riverside, CA, USA). Itaconic acid (IA) was bought from Sigma-Aldrich (St. louis, MO, USA). Vinyl acetate monomer (VAc) was obtained from Daejung Chemicals & Metals (Siheung-si, Korea). An initiator, 2-hydroxy-2-methylpropiophenone (Darocur 1173), was purchased from TCI (Tokyo, Japan). Ethyl Alcohol 99.9% (EtOH) was purchased from Duksan Pure Chemical (Ansan-si, Korea). A 1 mol/L sodium hydroxide solution (1N) was obtained from Samchun Pure Chemical (Seoul, Korea). A 3M® Scotch® "610" Cellophane Film Tape was purchased from SciLab Korea (Seoul, Korea). The 258 μm-thick Kent paper (170 g/m²) was obtained from Hanapaper (Namyangju-si, Korea).

2.2 Synthesis and Film preparation of IESO/VAc

The IESO was synthesized based on previously reported methods[15]. The formulation was prepared with an epoxide/acid group ratio of 1:1.15. To prepare the solution, 20 mL of EtOH was added to a 100 mL beaker and heated to 90°C, and 5.84 g of IA was added to the beaker and maintained for 10 mins. After IA completely dissolved, 16 g of ESO was added and the mixture was stirred at 90°C, 300 rpm for 1 hour. At this stage, the uncured solution was called IESO-S. The solu-

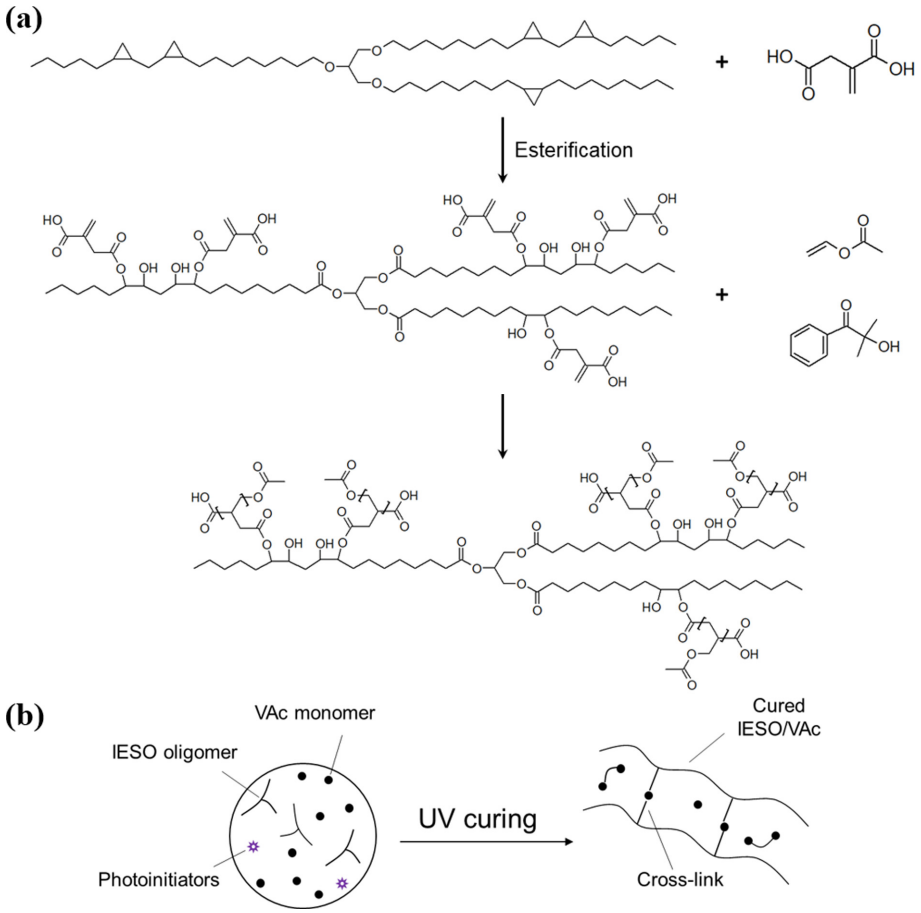

Fig. 1. (a) Schematic diagram of the chemical structure of IESO synthesized by esterification and UV polymerization. (b) UV curing process forming a cross-linked IESO/VAc network.

Table 1. Sample compositions with IESO, VAc, and Darocur 1173

Sample	IESO (wt.%)	VAc (wt.%)	Darocur 1173 (phr)
IESO-S	100	0	0
IESO	100	0	3
IESO/VAc 9:1	90	10	3
IESO/VAc 7:3	70	30	3
IESO/VAc 5:5	50	50	3
PVAc	0	100	3

Table 2. Thermal stability of cured IESO/VAc blends and PVAc

Sample	TGA				
	$T_{5\%}$ (°C)	$T_{10\%}$ (°C)	$T_{30\%}$ (°C)	T_{max1} (°C)	T_{max2} (°C)
IESO	206	280	353	377	-
IESO/VAc 9:1	241	303	357	377	-
IESO/VAc 7:3	229	302	362	379	-
IESO/VAc 5:5	227	293	355	379	-
PVAc	252	307	327	332	433

tion was cooled to room temperature, and VAc was added at concentrations of 0, 10, 30, and 50 wt.%. To prepare the coating solution, 3 phr of Darocur 1173 was added to the IESO/VAc mixture. The schematic of the IESO/VAc blend synthesis and UV curing process is shown in Fig. 1, and the sample compositions for different IESO/VAc ratios are presented in Table 2.

Coating solutions with varying concentrations were applied to white paper (258-260 μm) and cured for 60 mins under a UV curing system (RX-T21, Raynics, Siheung-si, Korea) with a wavelength range of 320-420 nm. The thickness of the cured coated paper ranged from 312-387 μm.

2.3. Characterization

FT-IR was used to compare the chemical bonds of the samples before and after curing. FT-IR analysis was performed using an ATR-equipped Fourier transform infrared spectrometer (Spectrum 65, PerkinElmer, Shelton, CT, USA) over a wavelength range of 400-4000 cm^{-1} with 64 scans.

To evaluate the surface characteristics of the coated paper, a cross-cut test (ASTM D3359-17, Standard Test Methods for Rating Adhesion by Tape Test) and water contact angle measurements (Phoenix 300 contact angle goniometer, SEO, Suwon-si, Korea) were performed. The cross-cut test was conducted to assess the interfacial adhesion between the paper and the coating. Twelve parallel cuts, spaced 10 mm apart, were made on the coating surface, and 3M® Scotch® "610" cellophane film tape was uniformly applied. After peeling the tape off at a 45° angle, adhesion was evaluated on a scale from 0B to 5B.

The thermal stability of the IESO/VAc cured films was analyzed using a TGA (TGA Q500, TA Instruments, New Castle, DE, USA) under nitrogen at a flow rate of 10 mL/min, with a temperature increase of 10°C/min from 30°C to 700°C.

The chemical recyclability of the coated paper was also examined[15,18]. IESO/VAc blends and PVAc coated paper, as well as uncoated paper (10 mm × 10 mm), were immersed in 20 mL of 1 N NaOH solution at 60°C for 4 hours. After immersion, the samples were thoroughly rinsed with deionized water and dried in an oven at 60°C for 24 hours. Chemical recyclability was assessed by measuring and comparing the weight and thickness of each sample before and after immersion to determine the extent of coating removal.

3. Results and discussion

3.1 Fourier transform infrared spectroscopy (FT-IR)

Fig. 2 presents the FT-IR spectra of the starting monomers and IESO-S, both of which do not contain a photoinitiator, along with IESO and IESO/VAc blends, while also analyzing the chemical bond changes resulting from radical polymerization in the IESO/VAc blends. IA exhibits peaks for O-H stretching (3,100-3,200 cm^{-1}), C-H stretching (2,886 cm^{-1} and 2,834 cm^{-1}), carbonyl (C=O) stretching (1,682 cm^{-1}), and C=C stretching (1,621 cm^{-1})[19,20]. ESO shows peaks for C-H stretching (2,925 cm^{-1} and 2,855 cm^{-1}), ester C=O stretching (1,740 cm^{-1}), C-O stretching (1,239 cm^{-1}), and epoxy group (821 cm^{-1})[21,22].

The FT-IR spectrum of IESO-S shows that the O-H stretching vibration of IA (3,100-3,200 cm^{-1}) and the -C-H stretching peaks of ESO (2,925 cm^{-1} and 2,855 cm^{-1}) merged into a single peak, which formed a broad peak due to the influence

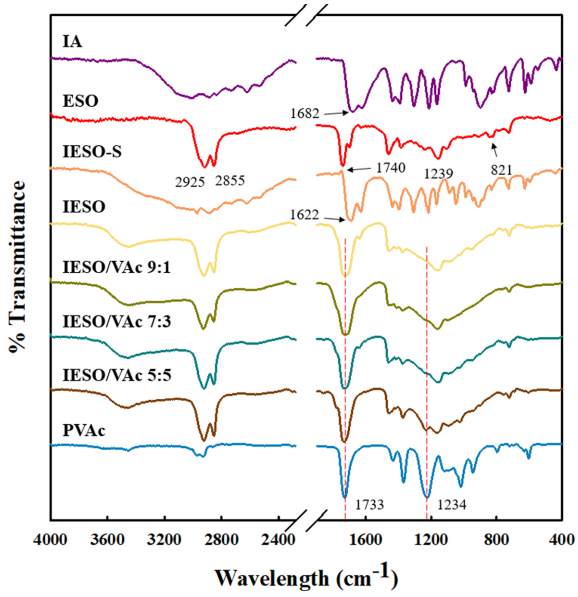

Fig. 2. FT-IR spectra of the starting monomers, IESO-S, IESO, and IESO/VAc blends.

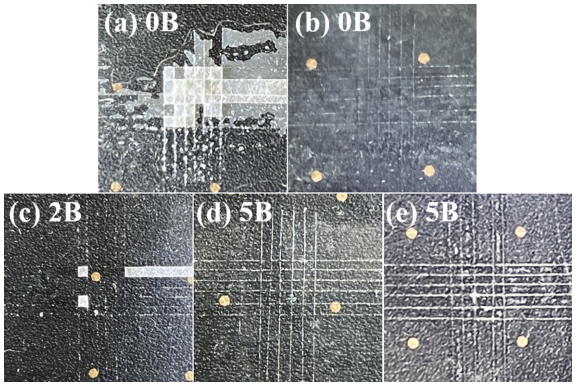

Fig. 3. Cross-cut test result of (a) IESO, (b) IESO/VAc 9:1, (c) IESO/VAc 7:3, (d) IESO/VAc 5:5, (e) PVAc.

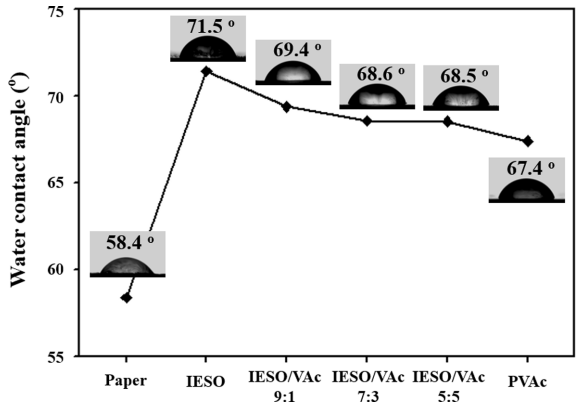

Fig. 4. Comparisons of the water contact angle on coated papers and uncoated paper.

of residual ethanol that was not fully evaporated during synthesis[19-23]. Additionally, the epoxy group peak at 821 cm^{-1} was reduced[21,22]. A comparison between the spectra of IESO-S and IESO revealed that the characteristic C=C stretching peak (1,621 cm^{-1}), a major indicator of photopolymerization, had nearly disappeared after curing[19,20,22]. As the VAc content increased, the C=O stretching (1,733 cm^{-1}) became sharper, while an additional C–O–C stretching peak (1,234 cm^{-1}) emerged[19-21,24]. These spectral changes suggest that the incorporation of VAc influenced the polymer structure, contributing to modifications in chemical bonding during the curing process.

3.2. Cross-cut test

The cross-cut test was conducted to evaluate the adhesion properties between the coating solution and the substrate, and the results are shown in Fig. 3. The IESO coating layer exhibited very low adhesion, with over 65% detachment due to incomplete curing. As the VAc content increased, adhesion improved. In IESO/VAc 9:1, the uncured coating surface transferred to the tape, while IESO/VAc 7:3 and higher formulations showed significant adhesion enhancement, with IESO/VAc 5:5 achieving the highest rating of 5B. This enhancement is due to the reactive VAc monomer, which increases the radical conversion rate during UV exposure[25]. Additionally, studies have shown that the addition of functional monomers improves wear resistance by promoting elastic deformation and forming a uniform network structure[26]. Therefore, increasing the VAc content enhanced the adhesion of UV-cured coatings.

3.3. Water Contact Angle

Fig. 4 presents the changes in contact angles after coating. The initial contact angle of uncoated paper was 58.4°, whereas after coating, the values were 71.5° for IESO, 69.4° for IESO/VAc 9:1, 68.6° for IESO/VAc 7:3, 68.5° for IESO/VAc 5:5, and 67.4° for PVAc. The IESO coating increased the contact angle by approximately 13.1° compared to uncoated paper. This relative increase was influenced by IESO, a fatty acid with long hydrocarbon chains, while UV-induced cross-linking reduced the number of pores available for water penetration, further enhancing hydrophobicity[27]. Although VAc is a nonpolar material, the decrease in contact angle with increasing VAc content can be attributed to its oxidation under UV exposure, which contributed to improved hydrophilicity[28].

3.4 Thermogravimetric Analyzer (TGA)

To evaluate the thermal stability of the polymer, TGA measurements were conducted. The weight loss curves as a function of temperature are presented in Fig. 5, and the corresponding data are summarized in Table 2. The 5 wt.% mass loss temperature ($T_{5\%}$) was 206°C for IESO and 252°C for PVAc. Compared to IESO alone, the IESO/VAc blends proved increased $T_{5\%}$, 10 wt.% mass loss temperature ($T_{10\%}$), and 30 wt.% mass loss temperature ($T_{30\%}$) values. This improvement is attributed to the role of VAc, which acts as a cross-linker within the IESO matrix and enhances crystallinity due to its short-chain structure. In contrast, IESO, with its long-chain

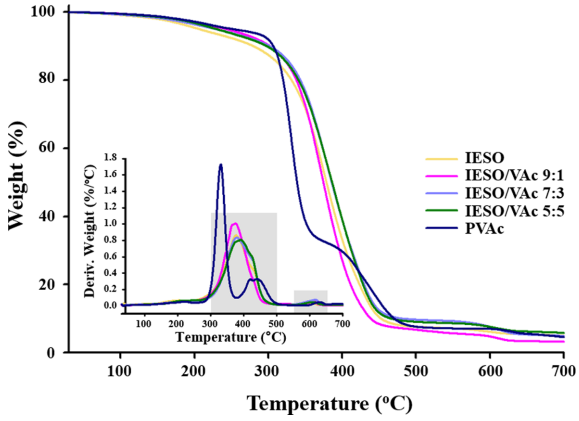

Fig. 5. TGA curves of the cured IESO/VAc blends and PVAc.

structure, increases flexibility and amorphous regions, leading to lower thermal stability.

PVAc exhibited a two-step weight loss pattern, which was due to the removal of acetic acid at 300°C, followed by the degradation of polyene in a subsequent step between 400 and 500°C[29–31]. IESO, IESO/VAc 9:1, IESO/VAc 7:3, and IESO/VAc 5:5 exhibited the most significant decomposition between 377-379°C. However, as the VAc content increased, the influence of polyene chain scission led to the formation of a broad decomposition peak in the range of 300-500°C. Furthermore, the weight loss observed 550-650°C is attributed to the thermal degradation or combustion of residual soot[32].

3.5. Chemical recycling

Recyclability is crucial in the development of sustainable cross-linked polymers. The presence of ester bonds in IESO suggests the potential for chemical recycling[15]. Results showed that all coatings, except PVAc, completely dissolved in NaOH. Table 3 presents the weight and thickness of uncoated paper and paper coated with IESO/PVAc blends before and after chemical recycling. The weight of the paper before immersion was 170 mg, and after NaOH immersion, the paper, IESO, IESO/VAc 9:1, IESO/VAc 7:3, and IESO/VAc 5:5 exhibited similar values. This indicates that all coatings were completely dissolved in NaOH. In contrast, PVAc retained 62% more weight, indicating its resistance to NaOH.

Table 3. Changes in thickness and weight of coated paper before and after NaOH immersion

Sample	Before NaOH immersion		After NaOH immersion	
	Weight (mg)	Thick (μm)	Weight (mg)	Thick (μm)
Paper	170	258	169	338
IESO	374	387	166	289
IESO/VAc 9:1	346	334	178	294
IESO/VAc 7:3	305	344	177	330
IESO/VAc 5:5	294	312	171	283
PVAc	299	316	273	421

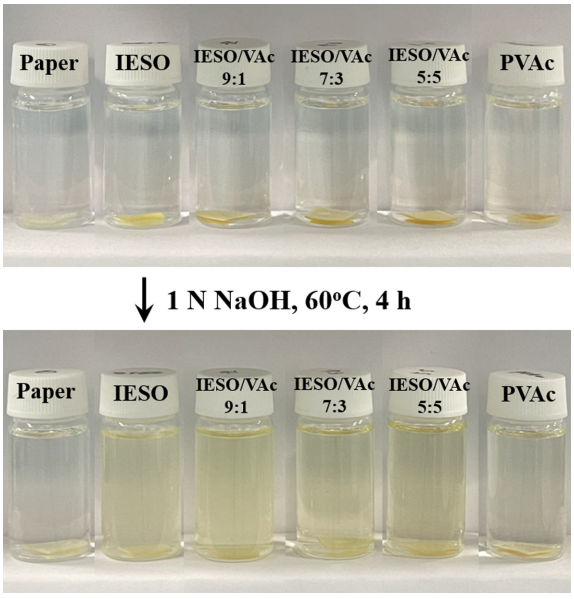

Fig. 6. Chemical recycling in 1N NaOH at 60°C after 4hours.

The visible changes can be observed in Fig. 6. After 4 hours, the fully dissolved solutions showed a gradual decrease in color intensity, with IESO exhibiting the highest coloration, followed by IESO/VAc 9:1, IESO/VAc 7:3, and IESO/VAc 5:5. This trend is attributed to the inherent transparency of VAc.

4. Conclusions

In this study, to manufacture eco-friendly UV-coated paper, IESO/VAc blend solutions were prepared using VAc, a functional monomer, with a small amount of photoinitiator. All IESO/VAc blends exhibited improved hydrophilicity, thermal stability, and chemical recyclability. Among the various formulations tested, the IESO/VAc 5:5 blend was identified as the most effective, demonstrating the highest rating in the cross-cut adhesion test, superior thermal stability compared to other ratios, and a water contact angle of 65.5°, indicating balanced surface properties. Based on these findings, the IESO/VAc 5:5 can be considered a promising candidate for use in the packaging industry, particularly as a sustainable alternative for producing UV-glossy coated paper with improved environmental performance.

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