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To cite this article: Aistis Lapinskas *et al* 2026 *Smart Mater. Struct.* **35** 105034

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Smart Materials and Structures



PAPER

OPEN ACCESS

RECEIVED

3 April 2026

REVISED

22 September 2026

ACCEPTED FOR PUBLICATION

2 October 2026

PUBLISHED

14 October 2026

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Antimicrobial acrylated epoxidized soybean oil and cinnamyl methacrylate-based thermoresponsive shape-memory photopolymers for microimprint lithography

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Keywords: biobased vitrimer, antimicrobial, shape-memory, microimprint lithography, life cycle assessment

Supplementary material for this article is available [online](#)

Abstract

A series of novel photocurable biobased vitrimers, composed of acrylated epoxidized soybean oil (AESO) and cinnamyl methacrylate was developed in this study. Photocuring kinetics, thermal, mechanical and antimicrobial properties were investigated to select the most suitable one for the production of highly antimicrobial and flexible vitrimer for the application in microimprint lithography. The addition of cinnamyl methacrylate in the composition had increased the flexibility and antimicrobial activity of vitrimers, but reduced the photocuring rate, tensile strength and thermal stability. Vitrimer composed of 1 mol of AESO and 5 mol of cinnamyl methacrylate was selected as the most suitable for this technology due to the highest flexibility, thermoresponsive shape-memory properties, and antibacterial activity. The cell reduction rate of this vitrimer against the bacteria *E. coli* and *S. aureus* was 100 and 91.3%, respectively, and against the fungi *A. niger* and *A. flavus* was 93.1 and 62.0%, respectively. A Life Cycle Assessment, comparing biobased cinnamyl methacrylate vitrimer with petroleum-based vitrimer, showed a 21% reduction in CO₂ emission. The novelty of this study combines the synthesis of cinnamyl methacrylate-based vitrimer with antimicrobial properties and low CO₂ emissions evaluated by LCA study.

1. Introduction

As the world overcomes plastic pollution crisis, biobased polymers are getting more and more popular [1]. Vegetable oils are one of the cheapest and most abundant renewable feedstocks for the production of polymers [2]. Hempseed, castor, canola, linseed, soybean and other vegetable oils were already used in the synthesis of biobased polymers and vitrimers [3]. Vitrimers are a novel class of polymers which combine the strength of thermosets with the reprocessability of thermoplastics [4]. The use of biobased materials in the synthesis of vitrimers creates sustainable materials responding to the 'European Strategy for Plastics in a Circular Economy' initiated by the European Commission. Epoxidized linseed oil was used for the production of biobased vitrimer with excellent recyclability and shape memory properties [5]. Recently biobased epoxy vitrimers were prepared using epoxidized soybean oil with tyramine and exhibited excellent self-repair, solvent resistance and shape memory properties [6]. Epoxidized soybean oil was also used with vanillin derivatives for the synthesis of epoxy vitrimers with high mechanical strength and self-healing properties [7]. However, most of the publications were focused on biobased

epoxide monomers, while the number of publications that are focused on plant oil-based acrylate vitrimers is significantly lower [8].

Acrylated epoxidized soybean oil (AESO) is one of the few commercially available oil-based acrylates, which was already used in synthesis of adhesives, foams, polymers and even used in optical 3D printing technology [9]. However, pure AESO polymers are brittle, have low thermal stability and poor mechanical properties, which limit their application [10]. This issue of AESO polymers can be solved by the addition of other monomers with rigid structure in the composition [11]. For example, the addition of vanillin dimethacrylate resulted in a biobased polymer with high thermal stability and mechanical strength [12]. Previous studies of AESO-based vitrimers focused on self-healing, shape-memory properties, and digital light processing 3D printing. These materials showed thermal stability and high shape-recovery efficiency [13]. However, antimicrobial activity of AESO-based vitrimers has been achieved only by incorporating additional antimicrobial components, such as harmful sulfur-containing monomers [14]. The combined investigation of thermal performance, shape-memory behavior, antimicrobial activity, and application potential within a single AESO-based vitrimer system remains insufficiently investigated. Different comonomers not only reduce the brittleness of polymers, but can also add other valuable properties such as antimicrobial activity. It was determined that addition of cinnamyl methacrylate into composition of 1,6-hexanediol diacrylate-based thermosets highly increases antimicrobial properties of resulted polymers [15]. The presence of a rigid phenyl moiety combined with a flexible aliphatic chain in cinnamyl methacrylate makes it a promising comonomer for incorporation into AESO, enabling enhanced flexibility in the resulting polymers. Cinnamyl methacrylate is a monofunctional acrylate which increases the segment mobility of polymer network thus increasing the flexibility of resulting polymers [16]. Also, the aromatic groups in its structure also contribute to the antimicrobial activity of polymers due to interactions between microorganisms and cinnamyl methacrylate aromatic groups [15]. Furthermore, incorporation of cinnamyl methacrylate into the vitrimer system addresses an existing research gap, as vitrimer systems containing this monomer were not previously reported in the literature.

Flexibility is a crucial characteristic for various applications such as coatings and flexible electronics [17]. Advanced manufacturing techniques provide opportunities to use biobased shape-memory polymers for the production of complex structures. Biomass-derived materials have been explored for 4D-printed biomedical and functional devices, while cellulose-nanofiber-reinforced shape-memory composites have been applied in self-deforming soft grippers [18, 19]. Recently, microimprint lithography has gained popularity due to simple and fast way for creating patterns on surfaces for optical and electronic components. Microimprint lithography uses transparent molds, usually made of polydimethylsiloxane, to create micropatterns from liquid resins that are polymerized by irradiating UV light through the mold [20]. Furthermore, microimprint lithography is considered as a low-waste technology compared to other fabrication techniques such as photolithography [21]. The molds and stamps are reusable and photocuring is considered energy efficient process with minimal waste generation and low environmental footprint [22]. Even though this process is environmentally friendly, it is important to evaluate its environmental impact throughout its entire life cycle by using life cycle assessment (LCA) method [23]. LCA provides an opportunity to improve products sustainability by identifying and reducing environmental issues by selecting sustainable product design and reducing carbon footprint [22]. It is an important tool which provides science-based calculations for product design [24].

The aim of this work was to develop a novel biobased vitrimer with antimicrobial properties suitable for microimprint technology. AESO was selected for this study due to its reactivity and high biorenewable carbon content (approximately 82%) [25]. Cinnamyl methacrylate was added to the composition to increase antimicrobial activity of resulting vitrimers, as well as reduce the brittleness and increase flexibility of the resulting polymers [16]. Photoinitiator ethyl(2,4,6-trimethylbenzoyl) phenylphosphinate was chosen for this study due to ability to cure deep resin layers and its high reactivity [26]. Thermal, mechanical and antimicrobial characteristics of vitrimers proved that addition of cinnamyl methacrylate increases the flexibility and antimicrobial activity of vitrimers. Self-welding test was performed to prove the reparability of the vitrimer. Selected resin was applied in microimprint lithography technology, which enables it to be applied in various fields including medicine, electronics, optics, and others. Furthermore, a LCA was conducted for the first time, comparing the life cycle environmental impact of biobased vitrimer formed using microimprint lithography with petroleum-based vitrimer, resulting in 21% lower CO₂ emission.

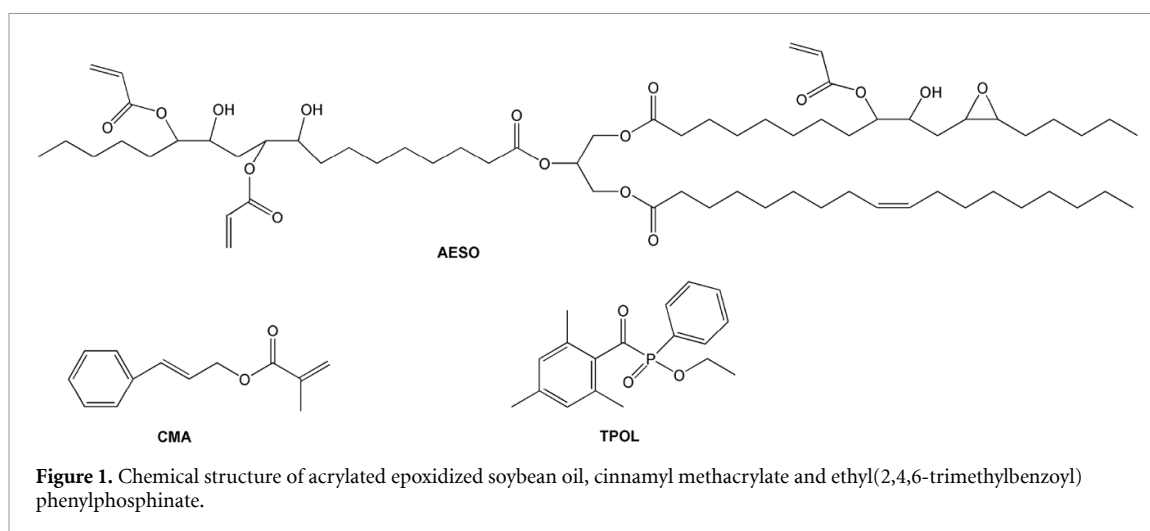


Table 1. Composition of resins A1–A5.

Resin	Amount of AESO, mol	Amount of CMA, mol	Amount of TPOL, mol%
A1	1	0	3
A2	5	1	3
A3	1	1	3
A4	1	5	3
A5	0	1	3

2. Materials and methods

2.1. Materials

AESO (Merck), cinnamyl methacrylate (CMA, Specific Polymers), and ethyl(2,4,6-trimethylbenzoyl) phenylphosphinate (TPOL, Fluorochem) were used as received (figure 1).

2.2. Composition of photocurable resins

The initial mixtures containing 0–5 mol of AESO, 0–5 mol of CMA, and 3 mol% of photoinitiator TPOL were stirred at 25 °C for 5 min until they reached the homogeneous consistency (table 1). The homogenous mixtures of resins were then poured into Teflon mold (70 × 10 × 0.5 mm) and cured for 3–5 min in the UV/Vis irradiation chamber BS-02 (Opsytec Dr Grobel) with an intensity of 30 mW cm⁻² and a wavelength range of 280–400 nm.

2.3. Methods

2.3.1. Real-time photorheometry

UV/V curing tests were performed on a MCR302 rheometer (Anton Paar) equipped with the plate/plate measuring system. The measuring gap was set to 0.1 mm and the samples were irradiated by UV/Vis light through the glass plate using UV/Vis spot curing system OmniCure S2000 (Lumen Dynamics Group Inc.). The temperature was 25 °C. A shear mode with the frequency of 10 Hz and a shear strain of 1% was used in all cases.

2.3.2. Fourier transform infrared (FT-IR) spectroscopy

FT-IR spectroscopy spectra were recorded using a BX II FT-IR spectrometer (Perkin Elmer, Llantrisant, UK). The reflection was measured during the test. The range of wavenumbers was (650–4000) cm⁻¹.

2.3.3. Soxhlet extraction

The Soxhlet extraction was used to determine the yield of insoluble (or gel? As mentioned in the manuscript) fraction. Polymer samples of 0.5 g were extracted with acetone for 24 h. Then the insoluble fractions were dried under vacuum until no weight changes were noticed. The yield of insoluble fraction was calculated as the difference of the weight before and after the extraction and drying.

2.3.4. Swelling test

The swelling values of the cross-linked polymer specimens were obtained by measuring the mass of specimens swollen in solvent at 25 °C. The polymer film specimens of 20 (± 0.1) mm length, 10 (± 0.05) mm width, and 1 (± 0.05) mm thickness were used. The change of mass was measured every 60 min and the percentage change in mass was calculated. Acetone and toluene were selected as solvents.

2.3.5. Dynamical mechanical thermal analysis (DMTA)

DMTA was performed on a DMA 850 apparatus (TA Instrument) in tensile mode. For each formulation, three specimens were analyzed, and the reported values correspond to the average. Measurements were performed over a temperature range from -50 °C to 80 °C at a heating rate of 3 °C.min $^{-1}$, with an applied oscillation frequency of 1 Hz, an amplitude of 15 μ m. Polymer samples of $50 \times 5 \times 0.2$ mm were used.

2.3.6. Thermogravimetric analysis (TGA)

TGA was performed on a TGA Q500 apparatus (TA Instrument) with a heating rate of 20 °C min $^{-1}$ under nitrogen atmosphere (100 ml min $^{-1}$). The temperature range of 10 °C– 800 °C was used.

2.3.7. Mechanical properties

Mechanical properties of the synthesized polymers were investigated by the tensile test performed on a Testometric M500-50CT testing machine (Testometric Co Ltd) at room temperature (25 °C). The dimensions of the test specimens were $70 (\pm 0.00) \times 10 (\pm 0.00) \times 1 (\pm 0.15)$ mm. Test was performed at the speed of 5 mm min $^{-1}$ until the break of the specimen.

2.3.8. Antimicrobial activity

The study of the antimicrobial activity of polymers was performed using two strains of bacteria: Gram-negative bacteria *Escherichia coli* ATCC 25 922 (*E. coli*) and Gram-positive bacteria *Staphylococcus aureus* ATCC 29 213 (*S. aureus*), as well as two fungal strains: *Aspergillus niger* MUCL 19 001 (*A. niger*), and *Aspergillus flavus* CBS 120 264 (*A. flavus*). The final inoculum concentrations were as follows: 3.5×10^6 colony forming units/ml (CFU ml $^{-1}$) for *E. coli*, 8.0×10^6 CFU ml $^{-1}$ for *S. aureus*, 1.5×10^5 CFU ml $^{-1}$ for *A. niger*, 1.4×10^5 CFU ml $^{-1}$ for *A. flavus*. The test films of $10 (\pm 0.00) \times 10 (\pm 0.00) \times 0.5 (\pm 0.15)$ mm were placed in empty sterile Petri dishes and inoculated with microbial spore suspension (10 μ l). Plates with infected films were incubated for 1 h in a humid chamber (90% relative humidity) at 37 °C infected with bacteria and at 26 °C infected with the fungi. After 1 h of incubation, 2 ml of saline was applied to each film to wash away the spores. The resulting suspensions were plated on nutrient media (bacteria on Mueller Hinton Agar and fungi on Malt Extract Agar) and incubated at 37 °C with bacteria and at 26 °C with fungi. After 1 – 2 d of incubation, the grown bacterial colonies were counted, and after 5 – 6 d, the fungal colonies were counted, and the percent reduction of spores was calculated. The percent reduction was calculated according to the formula: $(a - b)/a \times 100\%$, where a is the concentration of the colony forming units (CFU ml $^{-1}$) in inoculum suspension; b is a mean of the viable spores (CFU ml $^{-1}$) on specimens from triplicate experiments after incubation.

2.3.9. Shape-memory properties

The shape-memory properties of the polymer samples were investigated using the bending technique. Specimens were mechanically bent and cooled below their glass transition temperature T_g . Then mechanical fixation was released and the angle of shape fixity (θ_f) was measured. The bent samples were heated above their glass transition temperature T_g to induce return to permanent shape and the angle of shape recovery (θ_r) was measured. The shape fixity ratio (R_f) and shape recovery ratio (R_r) can be estimated according to equations (1) and (2),

$$R_f = \frac{180^\circ - \theta_f}{180^\circ} \times 100 \quad (1)$$

$$R_r = \frac{\theta_r - \theta_f}{180^\circ - \theta_f} \times 100 \quad (2)$$

θ_f and θ_r are the angles measured in the temporary shape and the permanent shape, respectively.

2.3.10. Stress relaxation measurements

The topology freezing temperature (T_v) was determined by stress relaxation experiments on a MCR302 rheometer. The samples were equilibrated with the selected measurement temperature

(170 °C–230 °C) for 10 min and decreasing stress was recorded over time. In all cases, a shear mode with a frequency of 1 Hz, a shear strain of 5%, and a force of 5 N was used. Topology rearrangements were investigated by measuring the stress relaxation of vitrimer, and the T_v was determined from the Arrhenius curves for relaxation times of 10^6 s. The activation energy (E_a) of the vitrimer was determined by taking the slope ($m = E_a/R$) of the straight line fitted to the data.

2.3.11. Self-welding behavior

The self-welding experiment was carried out by cutting the LCD 3D printed (Elegoo Mars Pro 3D printer) sample ($70 \times 10 \times 1$ mm) into two equal pieces which were placed on top of each other with 1 cm² overlap and heated in the temperature chamber at 180 °C for 30 min. No additional pressure was applied to the samples. After that, samples were cooled down to room temperature, and a tensile test was performed.

2.3.12. LCA

In the LCA study, a batch of 1 kg of the developed biobased vitrimer was compared with the same amount of a petroleum-based vitrimer. The biobased vitrimer was composed of AESO and cinnamyl methacrylate (CMA) in a molar ratio of 1:5 (sample **A4**). The petroleum-based vitrimer was composed of bisphenol A glycerolate dimethacrylate and methyl methacrylate in the same molar ratio. The system boundaries of the analysis followed a cradle-to-gate approach, beginning with raw material extraction and ending with micrometer-scale patterns formed using microimprint lithography, ready for packing. The LCA model was developed using SimaPro version 9.5.0.2 software, applying the cut-off approach. The impact categories considered in the ILCD 2011 Midpoint+ method include climate change, ozone depletion, human toxicity (cancer and non-cancer effects), particulate matter, ionizing radiation (human health and ecosystems), photochemical ozone formation, acidification, eutrophication (terrestrial, freshwater, and marine), freshwater ecotoxicity, land use, water resource depletion, and depletion of mineral, fossil, and renewable resources.

2.3.13. Microimprint lithography

Microimprint lithography was used to test the suitability of the resins for replica production. First, a target structure was manufactured out of PlasGray material with the Asiga Pico2 39 UV table-top 3D printer (Asiga). Next, polydimethylsiloxane (PDMS) was poured over this target structure and thermally cured at 100 °C for 1 h, thus creating a soft mold, which was then used to make replicas of the resins. The 3D printed structure was a 1951 USAF resolution target (25% of the downloaded vector file size), used as a sample in optical engineering for testing imaging systems. The UV irradiation chamber was used to cure the resins through the soft mold and obtain the photocured replicas. The curing time was set to 5 min. Five replicas were produced and a group photograph was taken. The lengths of the replicas were measured using the image measurement software MeasureOnImage.com and compared with a 3D printed target.

2.3.14. Statistical data analysis

The collected data were statistically analyzed using ANOVA for the Microsoft Excel program. All the experiments were performed in triplicate, except for the tensile test and antimicrobial properties test in which five replicates were used. The results were presented as the average values $\pm$ standard deviation. The estimated p -value was below 0.05 within the groups.

3. Results and discussion

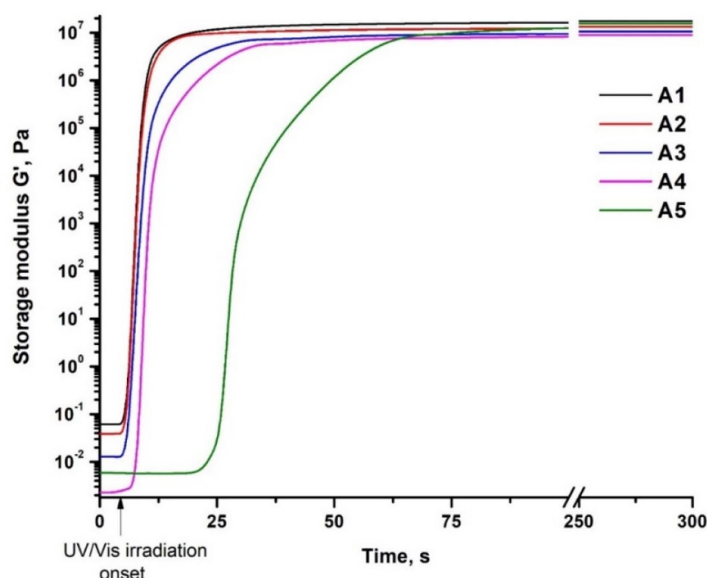
3.1. Photocuring kinetics

Real-time photorheometry was used to investigate the photocuring kinetics of resins. The results are summarized in table 2. The dependence of the storage modulus of the resins on irradiation time is shown in figure 2.

Polymer **A1**, prepared from pure AESO, had the highest rigidity (described by G' value), fastest photocuring (lowest value of t_{gel}), and the highest shrinkage, which can be explained by high amount of reactive acrylate groups with a tendency to shrink during photocuring [27]. The addition of CMA in the composition reduced the rigidity from 17.8 (**A1**) to 9.0 MPa (**A4**), photocuring rate from 1.7 to 4.5 s, but also reduced the shrinkage from 9 to 7.5%. This is due to the formation of long linear polymer chains between the cross-linking points formed by adding CMA as it has only one acrylic group. CMA is monofunctional acrylate and because of that increasing amount of CMA in the composition

Table 2. Rheological characteristics of resins A1–A5.

Resin	Storage modulus G'^a , MPa	Loss modulus G''^a , MPa	Complex viscosity ^a , mPa·s	Induction period, s	Gel point t_{gel} , s	Shrinkage, %
A1	17.8 ± 0.5	5.1 ± 0.0	311 ± 9	0.0 ± 0.0	1.7 ± 0.0	9.0 ± 0.0
A2	13.6 ± 0.5	4.8 ± 0.1	243 ± 7	0.0 ± 0.0	1.9 ± 0.0	8.5 ± 0.0
A3	10.7 ± 0.3	3.6 ± 0.1	178 ± 4	0.0 ± 0.0	3.0 ± 0.0	8.0 ± 0.2
A4	9.0 ± 0.3	3.1 ± 0.1	167 ± 4	4.0 ± 0.0	4.5 ± 0.1	7.5 ± 0.3
A5	16.2 ± 0.4	5.0 ± 0.0	254 ± 6	14.0 ± 0.5	57.0 ± 0.5	3.0 ± 0.0

^a after 300 s of irradiation.**Figure 2.** Dependence of G' of resins A1–A5 on the irradiation time.

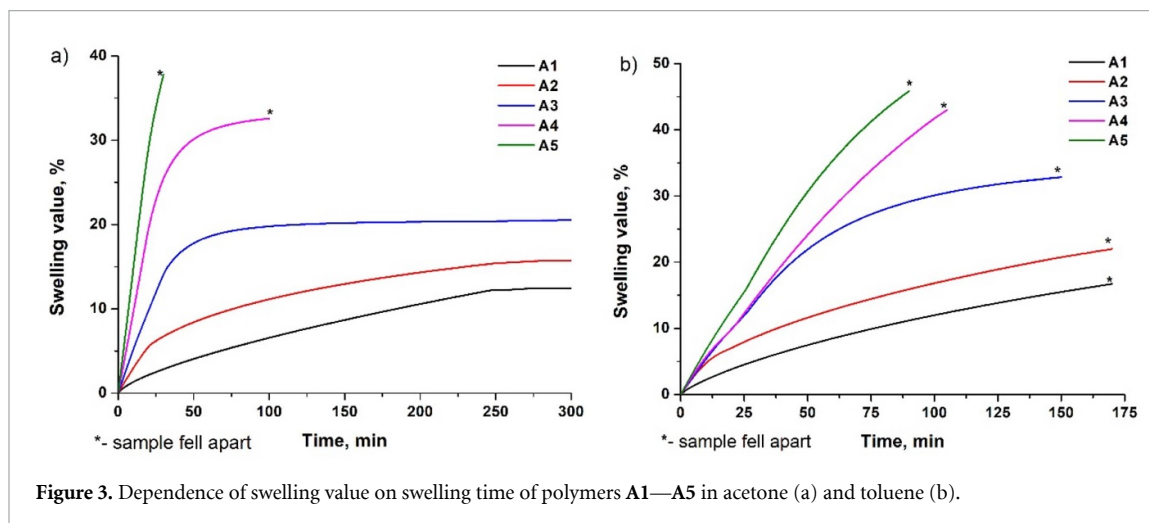
leads to lower crosslinking density and lower value of storage modulus. The lower shrinkage can be also explained by aromatic structure of CMA [16]. Because of that, polymer A5, prepared from pure CMA, had the lowest shrinkage (3%) value and the lowest photocuring rate ($t_{gel} = 57$ s). However, its rigidity was higher compared to polymers prepared using both monomers (A2–A4), and was only slightly lower than that of pure AESO-based polymer A1 (16.2 and 17.8 MPa, respectively). Even though mono-functional monomers form linear polymer chains, they can tangle with each other and form physical network, which can explain high rigidity of polymer A5 [28].

3.2. Characterization of polymers structure

The chemical structure of the polymer was analyzed by FTIR spectroscopy, the results obtained are described in supplementary section S3.2 and figure S1. The cross-linked structure of the polymer was confirmed by calculating the gel fraction and swelling values of the polymers. (table 3, figure 3). Polymer A1 had the highest amount of the gel fraction and the lowest shrinkage values in polar and non-polar solvents confirming that dense polymer network with short chains between the crosslinking points formed during photopolymerization and photodimerization. It is well known that cinnamyl fragments can undergo photodimerization which create polymer network after light irradiation at wavelength higher than 290 nm [29]. However, FTIR cannot fully distinguish radical polymerization from dimerization, because the peaks in their spectra are partially overlapping. The addition of CMA in the polymer structure reduced the gel fraction and increased the swelling values of polymers. Accordingly, pure CMA-based polymer A5 had the lowest value of the gel fraction and the highest value of swelling in polar and non-polar solvents because of its linear structure. Furthermore, polymers prepared with higher amount of AESO (A1–A3) were more resistant to solvent acetone and maintain their physical shape after 300 min of swelling while polymers A4 and A5 fell apart after 20–50 min of swelling. Polymers A4 and A5 were prepared using CMA as only or main component and its linear structure resulted in less stable polymer network. Furthermore, all polymers swell more and fell apart after 90–180 min of swelling in

Table 3. Gel fraction and swelling values of polymers.

Polymer	Gel fraction, %	Swelling value in acetone after 30 min of swelling, %	Swelling value in toluene after 90 min of swelling, %
A1	96.6 ± 0.4	2.9 ± 0.0	11.3 ± 0.1
A2	95.7 ± 0.1	6.8 ± 0.0	16.0 ± 0.4
A3	92.2 ± 0.2	14.3 ± 0.5	29.2 ± 1.0
A4	89.8 ± 0.1	25.6 ± 1.4	39.1 ± 1.3
A5	89.6 ± 0.1	37.8 ± 2.1	45.8 ± 1.2



toluene indicating that their structure has higher affinity to a nonpolar solvent toluene, and its molecules can penetrate deep in the sample causing high swelling and even breakage of polymer sample.

3.3. Thermal characteristics of polymers

Thermal characteristics of polymers were determined by DMTA and TGA, and the results are presented in figure 4. The highest and almost identical temperature of 10% weight loss was reached by polymers A1–A3 prepared with the highest amount of AESO indicating their high thermal stability. This behavior can be explained by higher amount of acrylate groups in AESO structure that leads to dense polymer network formation. The resulted higher crosslinking density can delay thermal degradation and increase the energy required for thermal destruction [30]. Polymers prepared from pure CMA (A5) or with higher amount of CMA compared to AESO (A4) exhibited lower thermal stability. The incorporation of CMA into the polymer composition resulted in a decrease in T_{α} . When the CMA content was increased from 0 mol (A1) to 5 mol (A4), T_{α} determined by dynamic mechanical analysis shifted from 47 to 29 °C. In DMA, T_{α} corresponds to the α -relaxation and is defined as the temperature at the maximum of the $\tan \delta$ peak, which is associated with the glass transition of the amorphous phase. The T_{α} of polymer A5 could not be determined due to its extreme brittleness.

3.4. Mechanical characteristics of polymers

Tensile test was selected for the investigation of mechanical properties of polymers (table 4) and tensile stress–strain curves were obtained (supplementary S3.4, figure S2). Polymer A5 was not tested because of its brittleness. Pure AESO-based polymer A1 had the highest values of Young's modulus (E) and tensile strength (δ), indicating that it is a rigid polymer with low flexibility due to extremely low elongation at break (ϵ) value. ϵ shows how much material can stretch before breakage and directly indicates its flexibility [31]. As it was expected, the addition of CMA in the polymer increased the ϵ from 0.83 to 2.59% (polymers A1 and A4) while also increasing the flexibility. However, addition of CMA also reduced the mechanical strength of polymers as δ was reduced from 3.29 to 1.69 MPa while E was reduced from 47.03 to 14.05 MPa. Although incorporation of CMA in polymer structure increased the flexibility of polymers, the tensile strength remained low. This limits the application areas of these polymers to films, coatings, low-load components or flexible structures produced by microimprint lithography

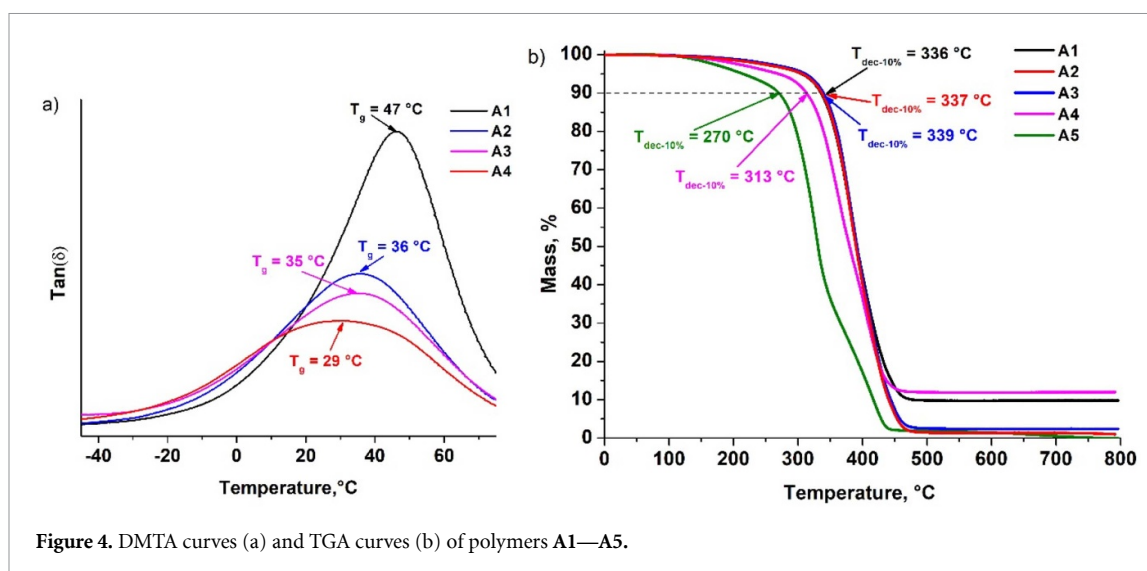


Figure 4. DMTA curves (a) and TGA curves (b) of polymers A1–A5.

Table 4. Mechanical characteristics of polymers.

Polymer	E , MPa	δ , MPa	ϵ , %
A1	47.03 ± 0.23	3.29 ± 0.05	0.83 ± 0.01
A2	25.74 ± 0.51	3.02 ± 0.04	1.17 ± 0.02
A3	21.86 ± 0.47	2.21 ± 0.02	1.32 ± 0.04
A4	14.05 ± 0.34	1.69 ± 0.02	2.59 ± 0.07

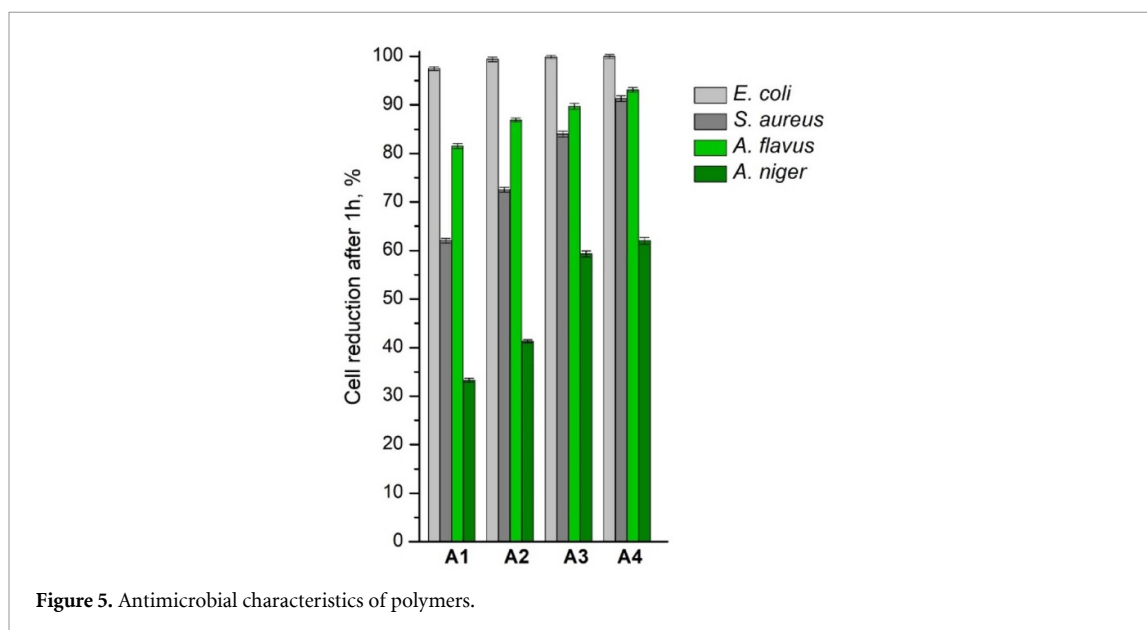


Figure 5. Antimicrobial characteristics of polymers.

3.5. Antimicrobial activity of polymers

The antimicrobial activity of polymers was tested against Gram-positive bacteria *S. aureus*, and Gram-negative bacteria *E. coli*, and against microscopic fungi, *A. flavus* and *A. niger*, after 1 h of contact time (figure 5).

All polymers exhibited higher antimicrobial activity against *E. coli* (97.5%–100%) compared to *S. aureus* (62.0%–91.3%). *S. aureus* has thick peptidoglycan cell wall which makes it more resistant to biocides than *E. coli* [32]. Antibacterial activity against both bacteria increased with the increased amount of CMA in polymer. For example, antimicrobial activity against *S. aureus* increased from 62.0% (polymer A1) to 91.3% (polymer A4) when the amount of CMA increased from 0 to 5 mol. That is because of strong antimicrobial activity of cinnamon compounds. Cinnamyl methacrylate and cinnamond-derived compounds possess aromatic and hydrophobic moieties that can interact with microbial cells, alter membrane organization and permeability, and interfere with essential cellular processes [33, 34].

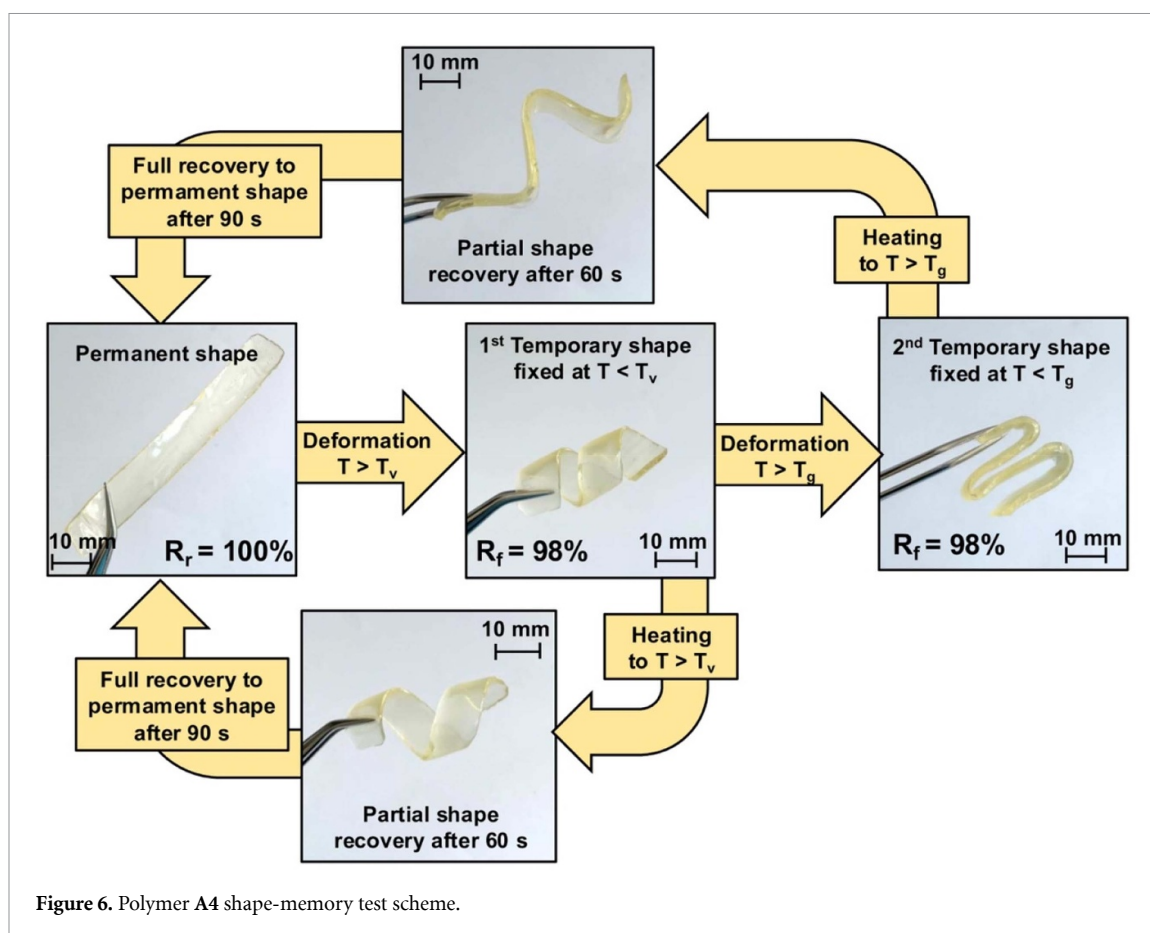


Figure 6. Polymer A4 shape-memory test scheme.

Similar trends were also observed in antifungal activity of polymers. Antifungal activity against *A. flavus* increased from 81.5% (polymer A1) to 93.1% (polymer A4) when the amount of CMA increased from 0 to 5 mol. Lower antifungal activity was observed against *A. niger* than *A. flavus*. For example, polymer A4, prepared with 5 mol of CMA, reached 93.1% cell reduction rate against *A. flavus*, but only 62.0% against *A. niger*. *A. niger* is more resistant to antimicrobial materials because of higher protein content in its walls, which makes it more robust [35]. Furthermore, *A. niger* is more resistant to environmental influence and can thrive under extreme conditions such as low or high temperatures [36]. That shows that even though antifungal activity against *A. niger* is not very high, but it is still highly important due to extreme resistance of this fungus. Polymer A4 was selected for investigation of vitrimer properties due to its highest antimicrobial activity determined by antimicrobial testing and its highest flexibility determined by tensile test.

3.6. Shape-memory properties

Vitrimer A4 exhibited thermoresponsive shape-memory behavior allowing it to change its permanent shape and retain 2 temporary shapes regulated by its T_α and T_v . This behavior is illustrated in figure 6. To acquire the 1st temporary shape, the specimen has to be heated to a temperature above 68 °C, mechanically transformed, and cooled to a temperature below 68 °C to fix the temporary shape. To acquire the 2nd temporary shape, the specimen has to be heated to a temperature above 29 °C, mechanically transformed, and cooled to a temperature below 29 °C.

The shape-memory properties of the polymer samples were evaluated by bending of the sample, to determine the shape fixity ratio (R_f) and the shape recovery ratio (R_r). It was calculated that the shape fixity ratio (R_f) in the temporary shape and the shape recovery ratio (R_r) in the permanent shape to which the sample returned were 98% and 100%, respectively. These results show that vitrimer has high recovery efficiency. The testing was performed five times and the results were identical, indicating cyclic stability.

3.7. Stress relaxation of vitrimers

Vitrimers have an unique structure with dynamic covalent bonds that allows them to rearrange their internal network structure and thus reduce the relaxation of internal stress caused by deformation due

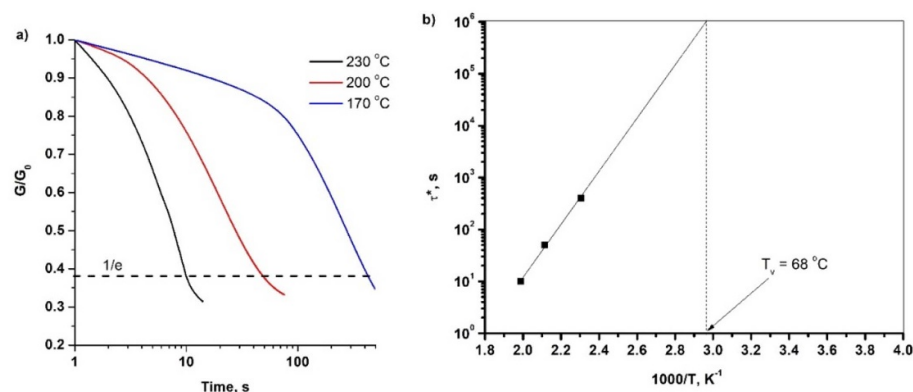


Figure 7. Stress relaxation curves versus time (a) and the Arrhenius plot of relaxation times of polymer A4.

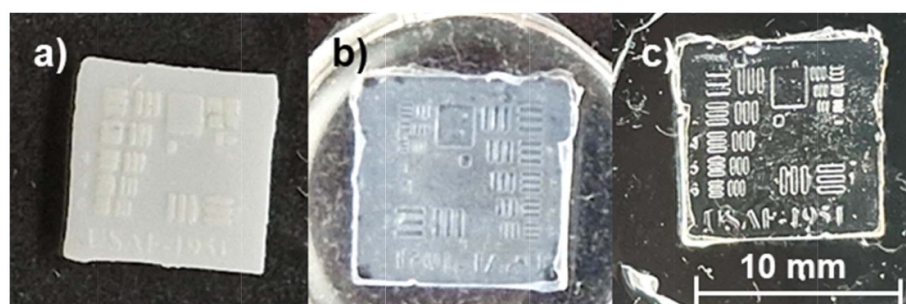


Figure 8. Photos made during the microimprint lithography test: 3D printed target (a), PDMS mold (b), and replica made from resin A4.

to the thermally activated transesterification reaction of ester and hydroxyl groups. Topology rearrangements were investigated by measuring the stress relaxation of vitrimer A4, and the topology freezing transition temperature (T_v) was determined from the Arrhenius curves for relaxation times of 10^6 s (figure 7). As a result, the topology freezing transition temperature of vitrimer A4 was 68 °C and the calculated activation energy was $72.3 \text{ kJ} \cdot \text{mol}^{-1}$.

3.8. Self-welding properties

For the investigation of self-welding properties, the tensile test was carried out and mechanical characteristics of vitrimer A4 before and after the self-welding test (A4.SW) were compared. The values of E and δ increased twice comparing vitrimer A4 (14.1 MPa and 1.7 MPa) and vitrimer after self-welding A4.SW (29.6 MPa and 3.2 MPa) while ϵ reduced from 2.6 to 1.1% indicating that transesterification reaction occurred during this process. The calculated tensile strength and Young's modulus self-welding efficiency was 188% and 210%, respectively. These results are higher than other AESO-based vitrimer that exhibit the tensile strength self-welding efficiency of 94% [13]. These values were also higher than bisphenol A diglycidyl ether-based vitrimer which reached 84% [37]. These comparisons confirm the effective self-welding of vitrimer A4. The FT-IR spectra of vitrimers A4 and A4.SW were recorded. Only transesterification reaction occurred and no other chemical changes took place during self-welding as there are no visible changes between vitrimer A4 and A4.SW spectra (figure S2 of the supporting information).

3.9. Microimprint lithography

Vitrimer A4 was applied in microimprint lithography technology, which is commonly used for fast and efficient production of flexible electronics [38]. Figure 8(a) shows 3D printed target. Figure 8(b) represents PDMS mold made using the same target. Figure 8(c) shows a replica made of A4 resin. The mean value of replicas length was 9.96 ± 0.02 mm. All features of the target are clearly visible in the replica, including numbers, letters and lines. This confirms that resin A4 is suitable for the production of flexible devices by microimprint lithography.

3.10. LCA

The LCA study was conducted to compare the life cycle environmental impact of biobased vitrimer with petroleum-based vitrimer formed using microimprint lithography. The selection of bisphenol A glycerolate dimethacrylate and methyl methacrylate for the petroleum-based vitrimer synthesis was based on thermal stability and enhanced mechanical properties of polymers synthesized from these materials. Bisphenol A glycerolate dimethacrylate is widely used in polymer chemistry due to its high rigidity and chemical resistance, which makes it an ideal choice for rigid vitrimer structures [39, 40]. Methyl methacrylate enhances mechanical strength and transparency, further optimizing the use of vitrimer for advanced applications [41]. The ILCD Midpoint+ method was chosen for the LCA study due to its comprehensive impact assessment framework, aligning with European Commission recommendations [42]. It covers a broad range of environmental impact categories, ensuring a holistic comparison of biobased and petroleum-based vitrimers. The processes included in the Ecoinvent database for both biobased and petroleum-based vitrimers with their corresponding inventory data are detailed in table S1 of the supporting information S3.10. The LCA results (table S2 of the supporting information S3.10) indicate that the biobased vitrimer showed reduced impact in 11 out of 16 categories, including climate change, ionizing radiation, human toxicity, photochemical ozone formation, terrestrial eutrophication, acidification, particulate matter, freshwater ecotoxicity, and fossil resource depletion, making it a more sustainable option in these aspects. Overall, the synthesis of the biobased vitrimer results in lower hazardous emissions due to the use of renewable monomers, with CO₂ emissions reduced by 21% compared to the petroleum-based vitrimer. Also, soybean oil is produced using plants that absorb CO₂ through photosynthesis, resulting in a green and low-carbon raw material [43]. However, biobased-vitrimer shows a higher impact in ozone depletion, freshwater eutrophication, and land use due to agricultural practices when cultivating the soybean. It can be concluded that, biobased resin is a better option than petroleum-based resin for vitrimer production, as it has a lower impact on climate change and human health. A similar conclusion was reported for cardanol-based vitrimer. Their preliminary LCA indicated that substitution of fossil-derived materials with renewable feedstocks could reduce the environmental impact of vitrimer production [44]. This is consistent with the present results, in which the A4 vitrimer showed lower environmental impact than the petroleum-based vitrimer in 11 of the 16 categories, including a 21% reduction of CO₂ emissions.

4. Conclusions

A series of novel photocurable biobased vitrimers, composed of AESO and cinnamyl methacrylate was developed successfully in this study and their rheological, thermal, mechanical and antimicrobial characteristics were investigated to find the most suitable one for microimprint lithography. The main conclusions are as follows:

- The addition of cinnamyl methacrylate in the composition reduces the photocuring rate, storage modulus and shrinkage of the polymers leading to slower photopolymerization and less rigid polymers.
- When the cinnamyl methacrylate content is increased from 0 to 5 mol% in the formulation, T_g shifts from 47 to 29 °C, indicating enhanced chain mobility and increased flexibility of the polymer network and the tensile strength is reduced from 3.29 to 1.69 MPa, but the elongation at break increases from 0.83 to 2.59%, indicating that vitrimer becomes more elastic and flexible. Low tensile strength limits the application areas of these polymers to films, coatings, low-load components or flexible structures produced by microimprint lithography. The future work will focus on increasing tensile strength of polymers to overcome this limitation.
- The highest antibacterial activity with cell reduction rate of 100% against *E. coli* and 91.3% against *S. aureus*, and the highest antifungal activity with cell reduction rate of 93.1% against *A. niger* and 62.0% against *A. flavus* was shown by polymer, prepared with the highest amount of cinnamyl methacrylate.
- Selected vitrimer had thermoresponsive shape-memory properties with the ability to maintain two temporary shapes and one permanent shape.
- The LCA of biobased vitrimer formed using microimprint lithography shows 21% lower CO₂ emission compared to petroleum-based vitrimer.

This study presents the development of a novel photocurable vitrimer made using cinnamyl methacrylate. This vitrimer has a potential for manufacturing antimicrobial flexible devices by microimprint lithography with lower environmental impact than petroleum-based vitrimers.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Supplementary Data 1 available at <https://doi.org/10.1088/1361-665X/aeaf95/data1>.

Funding

This research was funded by the Research Council of Lithuania (project No. S-MIP-23-52).

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