

From fossil to bio-based AESO–TiO₂ microcomposite for engineering applications

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Test S1: Characterization of the acrylated and epoxidized soybean oil (AESO)

The presence of the acrylate group in AESO was demonstrated via the ¹H–NMR technique. Figure S1 depicts the ¹H–NMR spectrum of AESO. The signal peaks at 5.7–6.2 ppm are consistent with acrylate ester entities. The signal peaks from 5.1–5.3 ppm and 4–4.5 ppm denote the –CH₂–CH–CH₂– glycerol methine and glycerol methylene protons, respectively, from the triglyceride backbone [1]. The weak signals in the range 3.2–4 ppm correspond to each hydroxyl group created for every acrylate moiety bonded to a triglyceride. The low intensity peaks in the range 2.8–3.2 ppm describe the protons of the epoxide ring owed to incomplete acrylation. A complete acrylation reaction of epoxidized soybean oil has not yet been reported, due to the bulkiness of the triglyceride molecule [2]. The following peaks are also attributed: –(CH₂)_n– at 1.1–1.4 ppm, terminal –CH₃ at 0.9–1.1 ppm, α–CH₂–(C=O)–O– at 2.2–2.4 ppm and β–CH₂–(C=O)–O– at 1.6–1.8 ppm [2]. In order to calculate the number of acrylate groups per molecule AESO or acrylation degree the peak of terminal –CH₃ groups (0.9–1.1 ppm) was used as internal standard. The acrylation degree was estimated from the ratio of peak areas $A_{5.7-6.2 \text{ ppm}}/A_{0.9-1.1 \text{ ppm}}$ and found to be 2.42. This is in good agreement with literature data, according to which the acrylation degree for the commercial AESO Vikoflex© 7170, for example, is 2.76 [2].

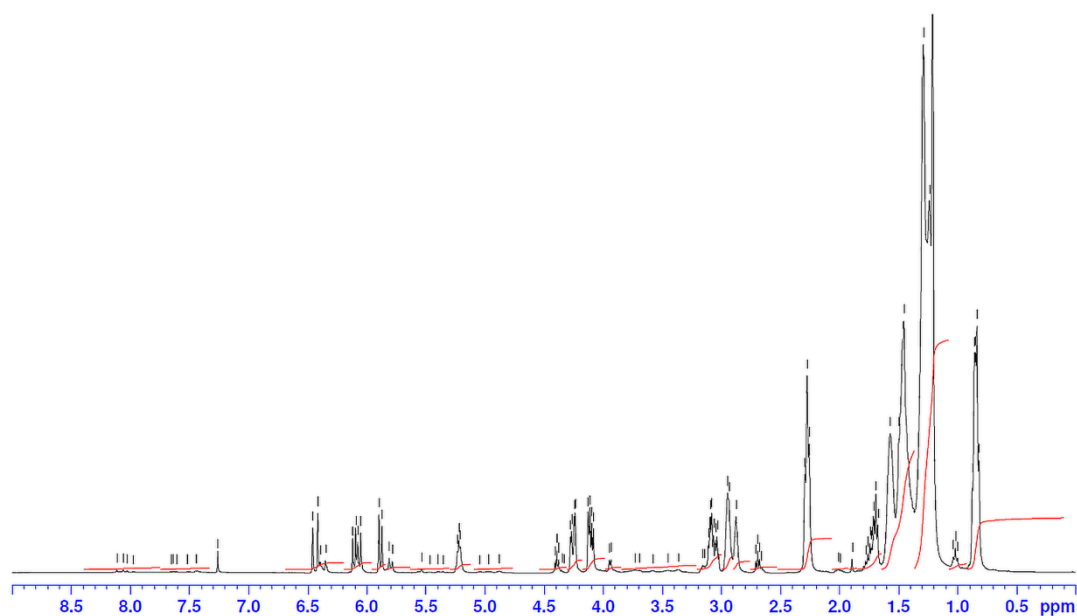


Figure S1. The ^1H -NMR spectrum of AESO

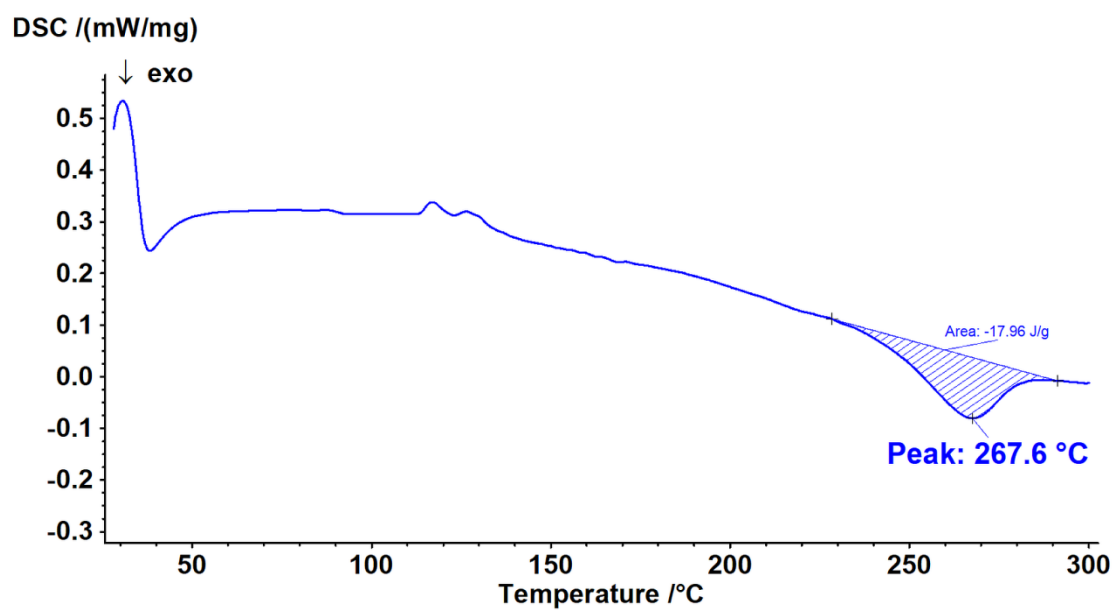


Figure S2. The first DSC heating curve of AESO –TiO₂ formulation.

Table S1. Thermal analyses data.

Sample	Stage	T _s (°C)	T _{5%} (°C)	T _{peak} (°C)	T _{30%} (°C)	M (%)	R (%)	T _g (°C)
AESO	I			390		64.31		
	II	150	197	420	380	17.83	5.46	–
	III			454		12.35		
AESO–m	I			393		57.14		
	II	180	340	426	387	19.85	7.49	–20
	III			460		14.71		
AESO–TiO ₂	I			400		48.89		
	II	183	342	430	395	28.17	5.52	–10
	III			462		16.49		

T_s – static heat resistant index; T_{5%} – temperature at 5% mass loss; T_{peak} – temperature of maximum rate of decomposition; T_{30%} – temperature at 30% mass loss; M – percentage of mass loss for each stage; R – percentage of mass residue remained at 700 °C; T_g – glass transition temperature

References

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2. Saithai P, Lecomte J, Dubreucq E, Vanrattanakul V (2013) Effects of different epoxidation methods of soybean oil on the characteristics of acrylated epoxidized soybean oil–co–poly(methyl methacrylate) copolymer. *Express Polym Lett* 7:910-924. doi: 10.3144/expresspolymlett.2013.89