

Upcycling used cooking oil into bio-based amines: Precursors for sustainable polymer materials

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ABSTRACT

The increasing demand for sustainable polymer materials has driven research toward renewable and waste-derived feedstocks capable of replacing fossil-based chemicals. Used cooking oil (UCO) is an abundant waste resource with significant potential as a bio-based precursor for functional intermediates in polymer synthesis. UCO is investigated as a feedstock for the preparation of multifunctional bio-based amines. Despite extensive research on the functionalization of vegetable oils, the synthesis and detailed structural characterization of UCO-derived amidoamines remain insufficiently explored. Here, we present a synthesis route for the preparation of bio-based amines under solvent-free conditions using commonly employed zinc(II) perchlorate hexahydrate as a Lewis acid catalyst. UCO was first epoxidized using the Prilezhaev reaction and afterwards reacted with aliphatic and aromatic amines, including 1,6-hexanediamine (HMDA), m-xylylenediamine (m-XDA), and tris(3-aminoethyl)amine (TREN) to ensure oxirane ring-opening and aminolysis of ester bonds. The synthesized HMDA/EUCO, m-XDA/EUCO and TREN/EUCO bio-based amines had total amine values ranging from 280 mg KOH/g to 468 mg KOH/g and viscosities ranging from 12,000 mPa·s to 90,000 mPa·s. The chemical structures of the obtained products were investigated using nuclear magnetic resonance spectroscopy, and Fourier transform infrared spectroscopy, confirming the formation of β -amino alcohol structures resulting from oxirane ring opening, along with aminolysis of ester bonds yielding amide derivatives. Matrix-assisted laser desorption/ionization time-of-flight mass spectrometry was applied to provide detailed insight into the molecular species present in these systems, revealing that the products are mixtures of monomeric, dimeric, and trimeric species. The combined analytical results establish the chemical structure and molecular composition of the obtained UCO-derived bio-based amines, confirming the formation of multifunctional products containing amide, β -amino alcohol, and amine functionalities. The work demonstrates a viable route for the valorization of waste cooking oil into multifunctional bio-based amines and contributes to the development of sustainable polymer precursors within a circular bioeconomy.

1. Introduction

The transition toward a circular polymer economy demands new bio-based monomers that combine structural versatility, high functionality, and scalable production from renewable waste resources. Bio-based amines synthesized from used cooking oil (UCO) offer a practical implementation of this approach, transforming the waste stream into multifunctional building blocks capable of forming sustainable

thermoset networks, enabling structural adaptability and extended material lifetimes. UCO is a highly attractive, renewable, biogenic waste stream suitable for the development of sustainable materials and energy carriers, offering a viable alternative to fossil-derived resources while supporting principles of the circular economy. UCOs are unavoidable lipid residues generated from food preparation in households, the hospitality sector, and food-processing industries, with global production estimated at approximately 42 million tonnes per year (Ramírez et al.,

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2022). UCOs mainly consist of edible vegetable and animal fats, such as rapeseed, sunflower, olive, palm, soybean oils, fish oil, butter, beef tallow and chicken fat, and their composition is largely dictated by the parent oil predominantly composed (>99 wt%) of triacylglycerols containing C16 and C18 fatty acids with varying degrees of unsaturation (Cárdenas et al., 2021). In addition, UCO composition may vary depending on regional dietary habits, cooking practices, and the extent of thermal degradation during frying. Nevertheless, UCO is generated worldwide and is readily available in most regions, enabling the local utilization of this second-generation feedstock for the production of value-added bio-based chemicals and materials. Consequently, the degree of unsaturation and functionalization potential can differ considerably between UCO streams, influencing their suitability for oleochemical conversion pathways such as epoxidation and subsequent synthesis of bio-based amines (Sikdar and Princiotta, 2021). This variability in fatty acid chain length and unsaturation dictates the chemical reactivity and functionalization potential of UCO-derived feedstocks. Its valorization not only mitigates environmental burdens associated with improper disposal but also provides access to low-cost, bio-based raw materials (Awogbemi et al., 2021; Foo et al., 2022; Onn et al., 2024).

Traditionally, UCO is extensively utilized for biofuel production, particularly biodiesel (Awogbemi et al., 2021; Rincón et al., 2019). Beyond fuels, increasing attention has been directed toward the synthesis of biolubricants (Attia et al., 2020; Bashiri et al., 2021; Hussein et al., 2021; Joshi et al., 2023; Sarno et al., 2019; Zhang et al., 2020). UCO is also a valuable precursor for polyol production (Kurańska et al., 2019; Kurańska and Malewska, 2021), enabling the fabrication of bio-based polyurethanes foams (Dang et al., 2016; Kurańska et al., 2020b, 2020a; Malewska et al., 2022; Polaczek et al., 2023, 2022; Werlinger et al., 2024) as well as plasticizers (Suzuki et al., 2018), surfactants (Permadani et al., 2018), and coatings (Su et al., 2020). Furthermore, UCO can serve as a bio-based carbon source for the production of bioplastics (Onn et al., 2024) and biopolymers (Orjuela and Clark, 2020), as well as saponification of UCO allows its conversion into soaps and detergents (Onn et al., 2024), replacing palm oil and animal fats. Also, UCO-derived materials have been explored as binders and modifiers in asphalt, while reducing reliance on petroleum bitumen (Zhao et al., 2023).

The nucleophilic ring-opening of epoxide groups by amines represents a key strategy for the functionalization of oil-derived materials, enabling the formation of β -amino alcohol functionalities that serve as precursors for crosslinked bio-based polymer networks. The ring-opening of epoxides with amines is the most practical and widely applied method for the preparation of β -amino alcohols (Moser et al., 2022). However, direct nucleophilic ring-opening of epoxide groups with amines often suffers from reduced reaction rates and lower yields due to the inherently limited nucleophilicity of amines, particularly aromatic or sterically hindered species. Consequently, excess amine, prolonged reaction times, elevated temperatures, or hazardous solvents are frequently required to achieve satisfactory conversion (Li et al., 2020; Moser et al., 2022).

To overcome these limitations, different catalytic systems have been explored for epoxide-amine ring-opening reactions from which Lewis acids (various metal salts) are mainly used, such as ZnCl_2 , $\text{Zn}(\text{ClO}_4)_2$ or LiBr , since stronger Brønsted acids tend to form ammonium salts that suppress amine nucleophilicity (Moser et al., 2022). These Lewis acid-catalyzed systems can provide β -amino alcohols in good to excellent yields in the presence or absence of a solvent. In the absence of catalysts, internal fatty epoxides exhibit negligible reactivity even at elevated temperatures (Radojčić et al., 2016). Moreover, reaction efficiency strongly depends on amine structure, with aliphatic amines generally being more nucleophilic than aromatic analogues (Li et al., 2020).

In epoxidized triglyceride or fatty acid-derived systems, competing pathways may arise, as aminolysis of ester groups can proceed alongside or even dominate over oxirane ring-opening (Radojčić et al., 2016).

Previous studies demonstrated that reactions of epoxidized soybean or linseed oils with amines frequently result in direct crosslinking and thermoset formation (Lee et al., 2008; López Téllez et al., 2008), while methyl epoxy stearate reaction with monoamines confirms efficient β -amino alcohol formation under Lewis acid catalysis (Singh and Kamboj, 2010).

However, in most reported cases, reactions between oil-derived epoxides and amines directly yield crosslinked thermosetting materials that represent the final product. In contrast, the present approach aims to synthesise multifunctional bio-based amines from epoxidized UCO as reactive intermediates containing β -amino alcohol, amide, and amine functionalities. Rather than inducing direct network formation during synthesis, the objective is to obtain and characterize these multifunctional intermediates prior to subsequent polymer synthesis. Thus, the bio-based amines obtained in this work are not intended as final thermosetting materials but as intermediates for subsequent polymer synthesis, including dynamic polymer networks (Sharma et al., 2025; Taplan et al., 2021), non-isocyanate polyurethanes (Destaso et al., 2024), and curing agents for epoxy resins, elastomers, and reinforced composites (Abduh et al., 2015).

To address the existing knowledge gap, this study investigates the valorization of UCO as a renewable feedstock for the synthesis of multifunctional bio-based amines through nucleophilic ring-opening of epoxides and triglyceride ester aminolysis. While related reactions have been reported for vegetable oil-derived systems, including direct thermoset formation from epoxidized oils and amines as well as β -amino alcohol synthesis from model fatty epoxides, detailed structural characterization of multifunctional UCO-derived amine products remains limited. Therefore, the obtained products were comprehensively characterized using NMR, MALDI-TOF, FTIR, and GPC/SEC analyses to establish their chemical structure, molecular composition, and the presence of monomeric and oligomeric species. In addition, key properties relevant to their future utilization as polymer precursors, including amine value and viscosity, were determined.

2. Experimental

2.1. Materials

Reagents used for synthesis: Used cooking oil (UCO) was obtained from a local waste management company in Latvia. After filtration and prior to epoxidation, the feedstock was characterized by an iodine value of $104 \pm 2 \text{ g I}_2/100 \text{ g}$, a saponification value of $198 \pm 3 \text{ mg KOH/g}$, an acid value below 3 mg KOH/g , and a viscosity of $114 \pm 5 \text{ mPa}\cdot\text{s}$ at a shear rate of 50 s^{-1} (25°C). Epoxidized used cooking oil (EUCO) prepared in the Latvian State Institute of Wood Chemistry exhibited an iodine value of $4 \pm 2 \text{ g I}_2/100 \text{ g}$, an oxirane value of $0.34 \pm 0.01 \text{ mol/100 g}$, a saponification value of $200 \pm 3 \text{ mg KOH/g}$, an acid value below 3 mg KOH/g , and a viscosity of $456 \pm 11 \text{ mPa}\cdot\text{s}$ at a shear rate of 50 s^{-1} (25°C). Amberlite™ IRC-120H, strongly acidic, hydrogen form, hydrogen peroxide, purum p.a., 35%; glacial acetic acid, puriss, 99.8%, 1,6-hexanediamine (HMDA), > 99.0%; m-xylylendiamine (m-XDA), 99%; tris(2-aminoethyl)amine (TREN), 96% and zinc(II) perchlorate hexahydrate were purchased from Sigma-Aldrich (Schnelldorf, Germany).

Reagents for analysis: chloroform, puriss p.a., 99.0–99.4%; dichloromethane, puriss p.a., ACS reagent, $\geq 99.9\%$; methanol, puriss p.a., ACS reagent, $\geq 99.8\%$; perchloric acid, ACS reagent, 70%; potassium hydroxide, ACS reagent, pellets, $\geq 85\%$; potassium iodide, ACS reagent, $\geq 99\%$; tetraethylammonium bromide, reagent grade, 98% were purchased from Sigma-Aldrich, (Schnelldorf, Germany). Hanus solution, volumetric 0.1 M IBr; hydrochloric acid, puriss. p.a., fuming $\geq 37\%$ were purchased from Fluka (Seelze, Germany).

2.2. Characterization methods

The feedstock and synthesized bio-based amines were analyzed following standardized methods. The acid value was evaluated according to DIN EN ISO 2114:2002-06, utilizing a TitroLine® 7000 automatic titrator (Xylem Analytics, Weilheim, Germany). Epoxy and iodine values were measured in accordance with ASTM D 1652-04:2004 and ISO 3961:2013, respectively, while the saponification value was determined following ASTM D 5558-95:1995. The total amine value was determined using ASTM D 2073-92:1992.

The proposed method for primary amine determination thus quantified secondary and tertiary amine values using a method developed in-house employing salicylaldehyde as a selective blocking reagent for primary amine groups. In this procedure, approximately 0.25 g of sample was accurately weighed into a 100 mL conical flask and dissolved in 50 mL of glacial acetic acid containing 2–5% acetic anhydride. The solution was briefly warmed to 40–50 °C, cooled to 25 °C, and subsequently treated with salicylaldehyde (0.50 M in acetic acid, 15.0 mL). The mixture was stirred at room temperature for 16 h to ensure complete blocking of the primary amine groups. The remaining secondary and tertiary amine groups were then titrated potentiometrically to equivalence with 0.100 N HClO₄ in acetic acid. The content of primary amine groups was calculated as the difference between the total amine value and the sum of secondary and tertiary amine values.

$$AV_{\text{prim}} = AV_{\text{total}} - AV_{\text{sec+tert}} \quad (1)$$

Where AV_{total} and $AV_{\text{sec+tert}}$ is titrated total amine value and sum of secondary and tertiary amine values in mg KOH/g, respectively, and AV_{prim} is primary amine value. The relative contributions of primary, secondary, and tertiary amines were calculated proportionally based on the number of nitrogen atoms of each type present in the proposed monomer, dimer, and trimer structures, assuming that each nitrogen atom corresponds to one titratable site.

$$AV_i = AV_{\text{total}} \times \frac{N_i}{N_{\text{total}}} \quad (2)$$

Where N_i is the number of nitrogen atoms of primary, secondary or tertiary amine groups in corresponding monomeric, dimeric or trimer structures and N_{total} is the sum of all nitrogens in the corresponding structure.

Apparent viscosity was investigated using an Anton Paar MCR 92 rheometer (Anton Paar, Graz, Austria) equipped with a cone-plate geometry (gap: 48 µm). Shear rate sweeps were performed over the range of 1–500 s⁻¹ at 25 °C, with temperature control provided by a hood.

All ¹H, ¹³C, 2D ¹H-¹³C HSQC and HMBC nuclear magnetic resonance spectra were acquired on a Bruker 400 AVANCE NEO NMR (Bruker BioSpin GmbH, Rheinstetten, Germany) spectrometer equipped with a 5 mm Z149000_0043 (CPP BBO 400S1 BB-H&F-D-05 Z) probe at 25 °C. Samples were dissolved in CDCl₃ and pipetted into 5 mL NMR tubes. The NMR spectra were processed and analyzed using Topspin 4.5.0 software.

MALDI-TOF mass spectra were acquired using an UltrafleXtreme TOF-TOF mass spectrometer (Bruker Daltonics, Bremen, Germany) equipped with a 2000 Hz SmartBeam-II laser (355 nm) in positive ion reflectron mode. Panoramic pulsed ion extraction and external calibration were applied for molecular weight determination. Samples were prepared using the dried-droplet method. Solutions of the sample (10 mg/mL), matrix 2,5-dihydroxybenzoic acid (20 mg/mL), and the ionizing agent sodium trifluoroacetate (CF₃COONa, 10 mg/mL) in N,N-dimethylformamide were combined in a volumetric ratio of 4:20:1. A 1 µL aliquot of the resulting mixture was deposited onto a ground steel MALDI target for analysis.

FTIR spectra were recorded on a Thermo Scientific Nicolet iS50 spectrometer (Thermo Fisher Scientific, Waltham, MA, USA) at a resolution of 4 cm⁻¹ with 32 scans. Measurements were carried out using the

attenuated total reflectance (ATR) technique with a diamond crystal to characterize the chemical structure of the synthesized monomers.

SEC analyses were carried out in pure HPLC methanol at 25 °C, with a flow rate of 0.5 mL min⁻¹ using Watrex Streamline system equipped with a Streamline P1 Pump, a Streamline AS2 Autosampler, a Streamline CT1 Column Thermostat, and a Streamline RI1 detector (Watrex Praha, Prague, Czech Republic). The separation was performed on two PLgel 5 µm mixed-D columns (Agilent Technologies, Inc., Santa Clara, CA, USA). Calibration was done on PEG standards in acetate buffer.

2.3. Epoxidation of UCO and synthesis of bio-based amines

The pretreatment of the obtained UCO was conducted as follows: oil was heated up to 60 °C while stirring and kept stirring for 30 min at 500 rpm, following filtration through the sieve with 0.1 mm diameter openings to remove solid impurities. Subsequently, UCO epoxidation was performed using the same methodology as reported in our previous work (Abolins et al., 2023). These steps are expected to mitigate the influence of degradation products and other feedstock-derived impurities prior to the subsequent bio-based amine synthesis. The scheme of the epoxidation reaction and bio-based amine synthesis is depicted in Fig. 1.

Bio-based amines were synthesized from EUCO by aminolysis of the triglyceride ester bond and nucleophilic oxirane ring-opening with amines. Three different amines were used: HMDA, m-XDA, and TREN. For simplicity, the notation R_iNH₂ is used throughout the manuscript and figures to denote the corresponding amine employed in the synthesis, where R_i represents HMDA, m-XDA, or TREN. The following acronyms were used for each synthesized bio-based amine: HMDA/EUCO, m-XDA/EUCO, and TREN/EUCO.

The synthesis of bio-based amines was carried out in a 500 mL four-necked round-bottom flask equipped with a mechanical stirrer, a thermocouple, a dropping funnel, and a reflux condenser. The amount of amine was determined based on both the oxirane value and the saponification value of EUCO, corresponding to a 1:1 molar ratio of amine to oxirane groups and a 1:1 molar ratio of amine to ester groups participating in aminolysis. For each synthesis, the required amount of amine was introduced into the flask: HMDA – 40.5 g, m-XDA – 47.5 g or TREN – 56.0 g. The reaction flask was heated using an oil bath, and the amine was brought to 60 °C under continuous stirring (400 rpm). The synthesis was performed under ambient atmospheric conditions. Preheated EUCO (50.0 g, 60 °C) was then added dropwise via the dropping funnel over approximately 10 min. Zinc(II) perchlorate hexahydrate (2 wt% relative to the EUCO mass) was added directly to the reaction mixture as a solid during the addition of EUCO. After all reactants had been added, the reaction temperature was increased to 100 °C and maintained under continuous stirring (400 rpm) for 24 h. Intermediate samples for NMR analysis were collected after 7 h of synthesis. Upon completion, the obtained bio-based amines were cooled to room temperature and further subjected to testing and analysis.

3. Results and discussion

3.1. Synthesis of bio-based amines from EUCO

The epoxidation reaction of UCO and synthesis of EUCO-derived bio-based amines are depicted in Fig. 1.

As stated before, the EUCO was used as a renewable triglyceride-based feedstock for the synthesis of multifunctional bio-based amines using HMDA, m-XDA, and TREN. The reactions were performed under solvent-free conditions in the presence of zinc (II) perchlorate hexahydrate as a Lewis acid catalyst, selected to promote oxirane activation while avoiding the rapid crosslinking typically observed with strong Brønsted acids. This adjusted approach was adopted based on the methodology reported by Singh and Kamboj (2010), who demonstrated efficient β-amino alcohol formation from methyl epoxy stearate under

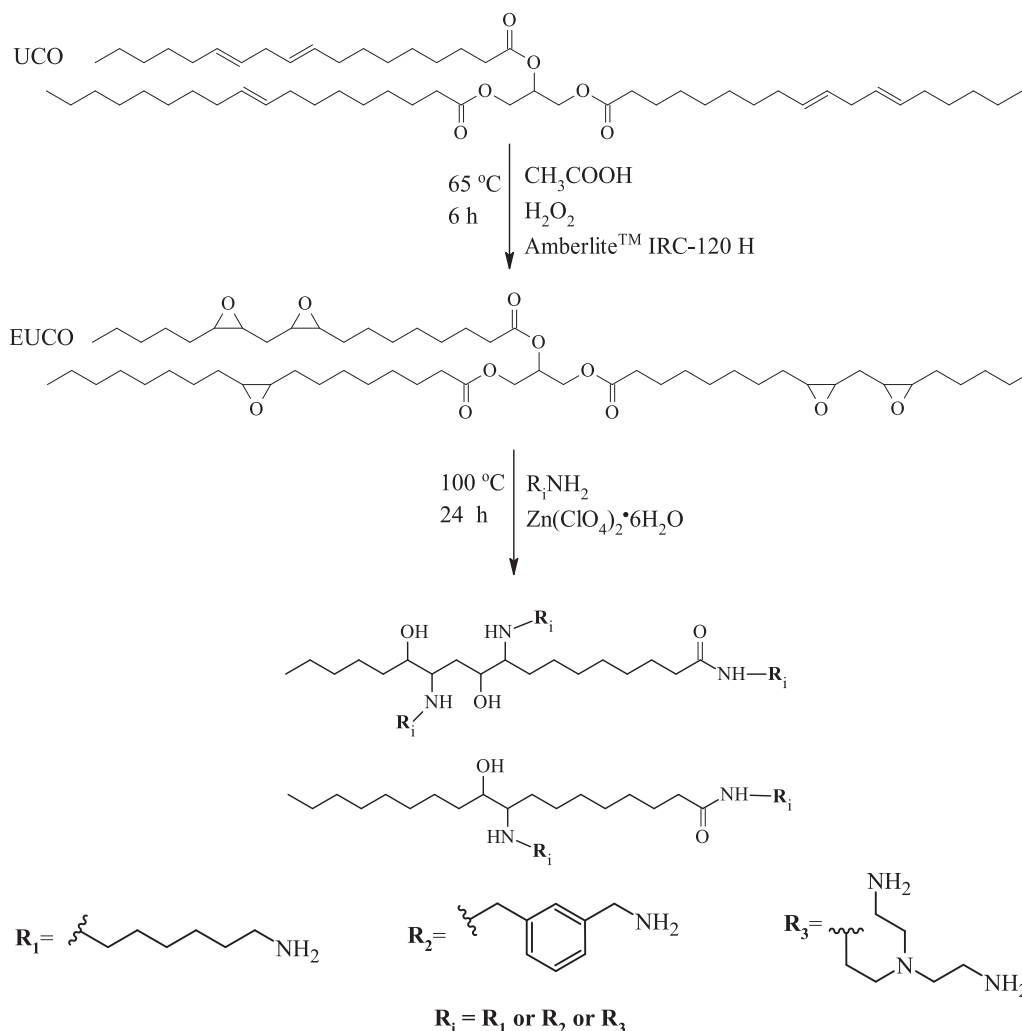


Fig. 1. Synthesis scheme of the bio-based amines using EUCO and different multifunctional amines. R_i represents the corresponding amine employed in the synthesis (HMDA, m-XDA, or TREN).

solvent-free conditions using zinc (II) perchlorate. However, their use of monoamines inherently prevented the formation of multifunctional amine intermediates. In contrast, the proposed approach intentionally benefits from employing di- and tri-functional amines to introduce multiple reactive sites. Compared to the strongly acidic conditions reported by Lee et al. (2008), which promoted extensive crosslinking in soybean oil systems, the milder Lewis acid catalysis applied here enabled the formation of well-defined intermediates rather than insoluble networks. Similarly, while López Téllez et al. (2008a) utilized ZnCl₂ for oxirane ring-opening in solvent systems, the present solvent-free methodology avoids organic solvents and reduces purification requirements. The amount of amine was determined based on both the oxirane value and the saponification value of EUCO, corresponding to a 1:1 molar ratio of amine to oxirane groups and a 1:1 molar ratio of amine to ester groups participating in aminolysis. Excess amine was deliberately avoided, which is in line with observations by Radojčić et al. (2016), who reported that excess amine does not significantly enhance internal epoxide conversion. To gain insight into the reaction progression, intermediate samples collected after 7 h of synthesis were compared with the final products obtained after 24 h using NMR analysis. The comparison showed that glycerol signals, indicative of ester aminolysis, were already present after 7 h, whereas signals corresponding to the oxirane ring remained detectable. In contrast, the spectra obtained after 24 h showed that the oxirane ring signals were no longer detectable, while the glycerol signals remained largely

unchanged. These observations suggest that ester aminolysis proceeds faster than oxirane ring-opening under the selected reaction conditions (Fig. S1–S3). Furthermore, this is also supported by Radojčić et al., who reported that hydroxyl groups generated during ester aminolysis may contribute to epoxide activation through hydrogen-bond-assisted mechanisms, acting synergistically with Zn(ClO₄)₂·6H₂O (Radojčić et al., 2016). No crosslinking was observed under selected conditions, indicating that stoichiometric balance and moderated catalytic activation effectively suppressed uncontrolled crosslinking. Therefore, the main distinction of the present work is not the formation of amide or β-amino alcohol groups alone, which has been reported previously, but the controlled synthesis and comprehensive structural characterization of multifunctional UCO-derived amine intermediates containing both amide and β-amino alcohol functionalities, without direct formation of insoluble thermoset networks.

The results demonstrate that EUCO can be transformed under controlled, solvent-free conditions into structurally complex, multifunctional bio-based amines containing β-amino alcohol and amide functionalities, providing primary, secondary, and tertiary amine groups, thereby establishing synthesized bio-based amines as versatile intermediates suitable for subsequent polymer synthesis.

3.2. Visual appearance and viscosity analysis of synthesized bio-based amines

The synthesized bio-based amines are shown in Fig. 2. Their viscosity vs. shear rate is summarized in Fig. 3. While the HMDA/EUCO product had the lowest viscosity – 11,946 mPa·s, an ~8-fold increase in apparent viscosity was observed for the m-XDA/EUCO product. The higher viscosity of m-XDA/EUCO can be explained by the aromatic moiety within the product, which creates more rigid regions, thereby increasing viscosity, whereas aliphatic amines as reactants allow more flexible aliphatic regions. A similar observation was made when aromatic systems were incorporated: polyester polyols with higher aromaticity exhibited higher viscosity (Duval et al., 2024). The apparent viscosity with aliphatic amines was 11,946 mPa·s for HMDA/EUCO and about 4-fold higher for TREN/EUCO - 37,844 mPa·s. This can be explained by the amine reactant structure: HMDA is more linear than the more branched TREN molecule. Similarly, in bio-based polyol systems, when a more branched aliphatic reactant rather than a linear one was used for oxirane ring opening, viscosities tended to increase with greater branching (Abolins et al., 2023; Pomilovskis et al., 2022). Finally, the presence of different oligomeric species with $\text{NH}_2/\text{NH}/\text{OH}$ functionalities can explain the high viscosities of the synthesized bio-based amines, due to increased intermolecular hydrogen bonding (Cao et al., 2019).

3.3. NMR analysis of synthesised bio-based amines

The NMR analysis will be provided only for significant protons and carbons. That is proton shifts that appeared or disappeared after functionalization and/or during the reaction. Also, for the sake of clarity, detailed discussion of peak integration and signal multiplicity is omitted due to extensive proton overlap and the compositional complexity of bio-based feedstocks, which results in multiple species in the final product mixture; a similar approach was adopted by López Téllez et al. (2008a) when analyzing amine-modified epoxidized vegetable oils by NMR. For more complex and overlapping signals, the two-dimensional NMR experiments, including heteronuclear single quantum coherence (HSQC) and heteronuclear multiple bond correlation (HMBC), were performed to elucidate the molecular structure of the synthesized bio-based amines. Alongside 2D HSQC and HMBC experiments, ^{13}C DEPT-135 spectroscopy was applied. The 2D spectra in combination with ^{13}C and ^{13}C DEPT -135 for the obtained bio-based amines are provided in the supplementary section (Fig. S4–S6).

The ^1H proton spectra (Fig. S7) are for feedstock - UCO and epoxidized product, EUCO. The most significant changes in the proton spectra after UCO epoxidation are observed in the region from 2.85 ppm to 3.22 ppm, which demonstrate the formation of the epoxide moiety. The complementary signal for epoxide formation is the disappearance at 5.35 ppm, which is the proton signal for UCO double bonds. Also, the signals at 2.02 ppm and 2.78 ppm have disappeared after epoxidation, which correspond to methylene protons adjacent to double bonds. The signal at 2.31 ppm corresponds to the alpha carbon adjacent to the carbonyl group. All above-mentioned proton shifts are typical for vegetable oils and epoxidized vegetable oils and have also previously been reported by Lee et al. (2008) and López Téllez et al. (2008) (see Fig. S1). After epoxidation, the EUCO was reacted with HMDA, m-XDA



Fig. 2. The visual appearance of obtained HMDA/EUCO, m-XDA/EUCO and TREN/EUCO.

and TREN to synthesise functionalized bio-based amines (Fig. 4). The resonances at around 0.88 ppm across all products represent the remote parts of the fatty chains that are the terminal methyl group (signal a). The broad multiplet between 1.16 and 1.75 ppm arises from overlapping $-\text{CH}_2-$ resonances of the fatty acid chain backbone. This region is non-diagnostic for reactions but is a common signal in oil-derived spectra, also reported by Lee et al. (2008).

Moving downfield, a very broad overlapping signal between 1.75–2.09, 1.95–2.23 and 1.88–2.31 ppm for HMDA/EUCO, m-XDA/EUCO and TREN/EUCO, which represents fast exchangeable protons in OH/NH_2 groups. Comparing the signal at 2.31 ppm for unreacted EUCO, the $\alpha\text{-CH}_2$ (adjacent to the ester carbonyl), it can be observed that after reaction, the $\alpha\text{-CH}_2$ (signal b) moves slightly upfield to 2.19 ppm for m-XDA/EUCO, 2.15 ppm for TREN/EUCO and 2.13 ppm for HMDA/EUCO, being more isolated and less overlapping with OH/NH_2 . This shift is consistent with the conversion of an ester ($-\text{CO}-\text{O}-\text{R}$) to an amide ($-\text{CO}-\text{NH}-\text{R}'$): the amide carbonyl is less electron withdrawing to the α -methylene than the ester carbonyl, hence the observed upfield movement. The similar observations was reported by Abduh et al. (2015) and Pramanik et al. (2019) when epoxidized rubber seed oil was reacted with triethylenetetramine, providing amidated moieties. The carbon signal (C_b) adjacent to the newly formed amide group is observed slightly downfield at around 37 ppm, as opposed to the unreacted EUCO, where this signal is 34 ppm (see Supplementary Fig. S8).

The characteristic oxirane ring proton signals of EUCO at 2.82 – 3.22 ppm after reaction with amines were not detectable; instead, two new resonance signals appeared. The first signal (c) of great importance was $\text{CH}(\text{NH})$ proton. For HMDA/EUCO and m-XDA/EUCO, signals at 2.25 ppm and 2.42 ppm, respectively. The proton (c) after nucleophilic ring-opening for TREN/EUCO is identified at about 2.26 ppm and overlaps with a broad signal, which consists of OH/NH_2 and alpha carbon protons (H_b) to the carbonyl group. The second important signal (d) is assigned to $\text{CH}(\text{OH})$ at 3.15–3.26 ppm for HMDA/EUCO, 3.27–3.37 ppm for m-XDA/EUCO and 3.21–3.36 ppm for TREN/EUCO. Both of these proton signals (c and d) were similarly reported by Singh and Kamboj (2010) and López Téllez et al. (2008) after nucleophilic ring opening of epoxides with amines. 2D HSQC spectra confirmed the disappearance of oxirane proton–carbon correlations and the emergence of new cross-peaks corresponding to β -amino alcohol moieties. These changes of the oxirane signals, combined with the appearance of the β -amino alcohol resonances, indicate that the epoxide rings are opened completely under the applied conditions (see Supplementary Fig. S1–S3). Also, new signals appear and correlate in ^{13}C spectra, where carbon (d) adjacent to $-\text{OH}$ after oxirane ring opening is observed for all obtained products at around 72 ppm and carbon (c) next to NH group is observed around 63 ppm. The carbon shifts (signal c and d) of β -amino alcohol moieties are also reported by Radojčić et al. (2016) and Singh and Kamboj (2010). These signals are clearly visible when ^{13}C DEPT-135 spectroscopy was applied to confirm β -amino alcohol moieties, providing complementary evidence for nucleophilic ring opening of epoxides with amines (see Supplementary Fig. S4–S6).

For HMDA/EUCO and TREN/EUCO, the proton shift (d) of the $\text{CH}(\text{OH})$ group overlaps also with the proton signal (e). The carbon signal (e) for these products is at 39 ppm and 37 ppm, respectively (see Fig. S8). However, when m-XDA is used as a reactant, the proton signal (e) shifts downfield to around 4.42 ppm, due to aromatic and amine moieties that cause electron withdrawing. Carbon (e) adjacent to this proton is observed and shifts downfield to around 43 ppm (see Fig. S8). Also, the proton shift (f) for m-XDA/EUCO is around 4.82 ppm, while the same proton shifts for HMDA/EUCO and TREN/EUCO are observed at 2.48 ppm and 2.55 ppm, respectively. The same carbon signals (f) are observed at 65 ppm for m-XDA/EUCO and at 47 ppm and 45 ppm for HMDA/EUCO and TREN/EUCO, respectively (see Fig. S8).

The signal common to all synthesized bio-based amines is proton shifts in the range from 3.52 to 3.76 ppm, which represents glycerol

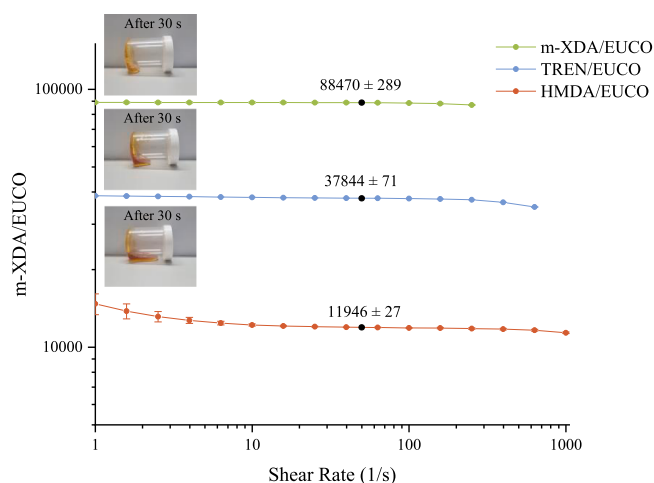


Fig. 3. The viscosity vs. shear rate of the HMDA/EUCO, m-XDA/EUCO, and TREN/EUCO.

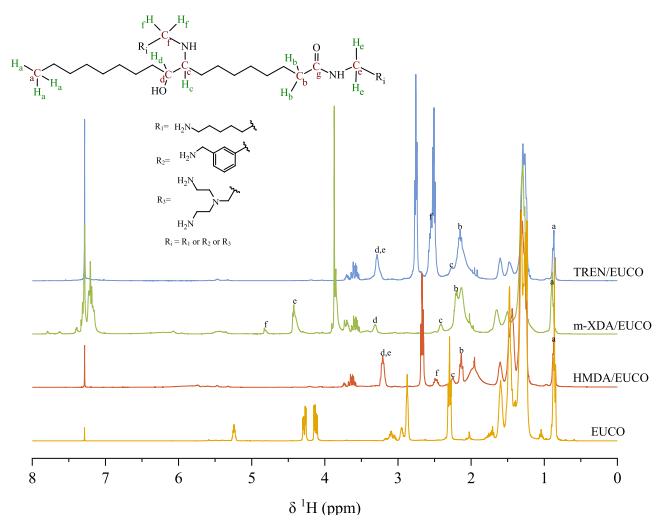


Fig. 4. ^1H proton shifts for newly synthesised EUCO-based bio-based amines.

protons after the aminolysis of triglyceride ester occurs.

3.4. MALDI-TOF analysis of synthesized bio-based amines

The MALDI-TOF mass spectra of the synthesized bio-based amines are shown in Fig. 5.a–c. The spectra reveal that across all synthesised products, three distinct molecular-weight regions are identifiable. These regions correspond to monomeric, dimeric, and trimeric structures of the bio-based amines obtained. In each spectrum, the expected product derivatives can be clearly assigned based on their molecular weights. For HMDA/EUCO, m-XDA/EUCO, and TREN/EUCO, the sodium adduct molecular ion peaks ($M + \text{Na}^+$) are observed at 536 u, 576 u, and 596 u, respectively, which are attributed to the monomeric species. These findings are in agreement with the NMR results, further confirming that β -amino alcohol and aminated derivatives are present in the end product. The monomeric structures are proposed to predominantly originate from fatty acid chains containing a single double bond, which, upon epoxidation, would generate one oxirane ring per fatty acid unit.

The second molecular weight region, located just below 1000 u, corresponds to dimeric structures of the synthesized bio-based amines. In the MALDI-TOF spectra, molecular ions of bio-based amines with sodium adduct molecular ion peaks at 932 u, 992 u, and 1022 u are detected, corresponding to dimeric HMDA/EUCO, m-XDA/EUCO, and TREN/EUCO species, respectively. Representative structures of these

dimers, comprising two coupled fatty acid chains, are depicted in Fig. 5. a–c.

The third and highest molecular weight region clearly corresponds to trimeric structures of the bio-based amines obtained. The molecular weights of the trimers are observed at 1329 u for HMDA/EUCO, 1409 u for m-XDA/EUCO, and 1449 u for TREN/EUCO. Due to the multifunctional nature of the amines and the presence of multiple reactive primary amine groups, a broad distribution of structurally related species is generated, as evidenced by the MALDI-TOF spectra showing distinct monomeric, dimeric, and trimeric entities. Consistent with these observations, the formation of monomeric, dimeric, and trimeric species following oxirane ring-opening and subsequent esterification of epoxidized tall oil fatty acids with multifunctional alcohols was also described by Pomilovskis et al. (2023) and Vanags et al. (2018) where MALDI-TOF analysis served as a reliable tool to elucidate the structural diversity within the resulting product mixtures.

3.5. FTIR analysis of synthesized bio-based amines

FTIR spectral evolution confirms the chemical conversion of EUCO to bio-based amines (see Fig. 6 and Table 1.). The defining feature of this transformation is the disappearance of the ester carbonyl absorption and the appearance of amide-related bands.

The starting EUCO exhibits a typical strong and sharp absorption carbonyl group band at approximately 1740 cm^{-1} (Region 4), which corresponds to the $\text{C}=\text{O}$ stretching vibration of the ester functionality present in the triglyceride backbone. Upon reaction with amines, this ester carbonyl band decreases in intensity and, at the end of synthesis, disappears entirely. Simultaneously in spectra, the formation of new absorption features in the amide-related bands (Region 2, 1) is observed. The amide band, typically centered between 1680 and 1630 cm^{-1} , originates predominantly from $\text{C}=\text{O}$ stretching vibrations of the amide group, while the amide band, observed between 1560 and 1520 cm^{-1} , is attributed to coupled $\text{N}-\text{H}$ bending and $\text{C}-\text{N}$ stretching vibrations. The emergence of these two absorption bands and the disappearance of the ester band represent amide formation, confirming that the aminolysis of the triglyceride ester has proceeded to completion. Also, there are potential concurrent reactions at the epoxide sites. The broad $\text{NH}/\text{NH}_2/\text{OH}$ stretching region ($3000\text{--}3400\text{ cm}^{-1}$) confirms the presence of amine and hydroxyl groups in the reaction products. The typical $\text{C}-\text{H}$ stretching band vibrations of the aliphatic hydrocarbon chains of the triglyceride backbone are visible at 2925 and 2850 cm^{-1} . The disappearance of the epoxide absorption around 840 cm^{-1} (Region 5), corresponding to $\text{C}-\text{O}-\text{C}$ asymmetric stretching of the oxirane ring, provides additional evidence of β -amino alcohol formation.

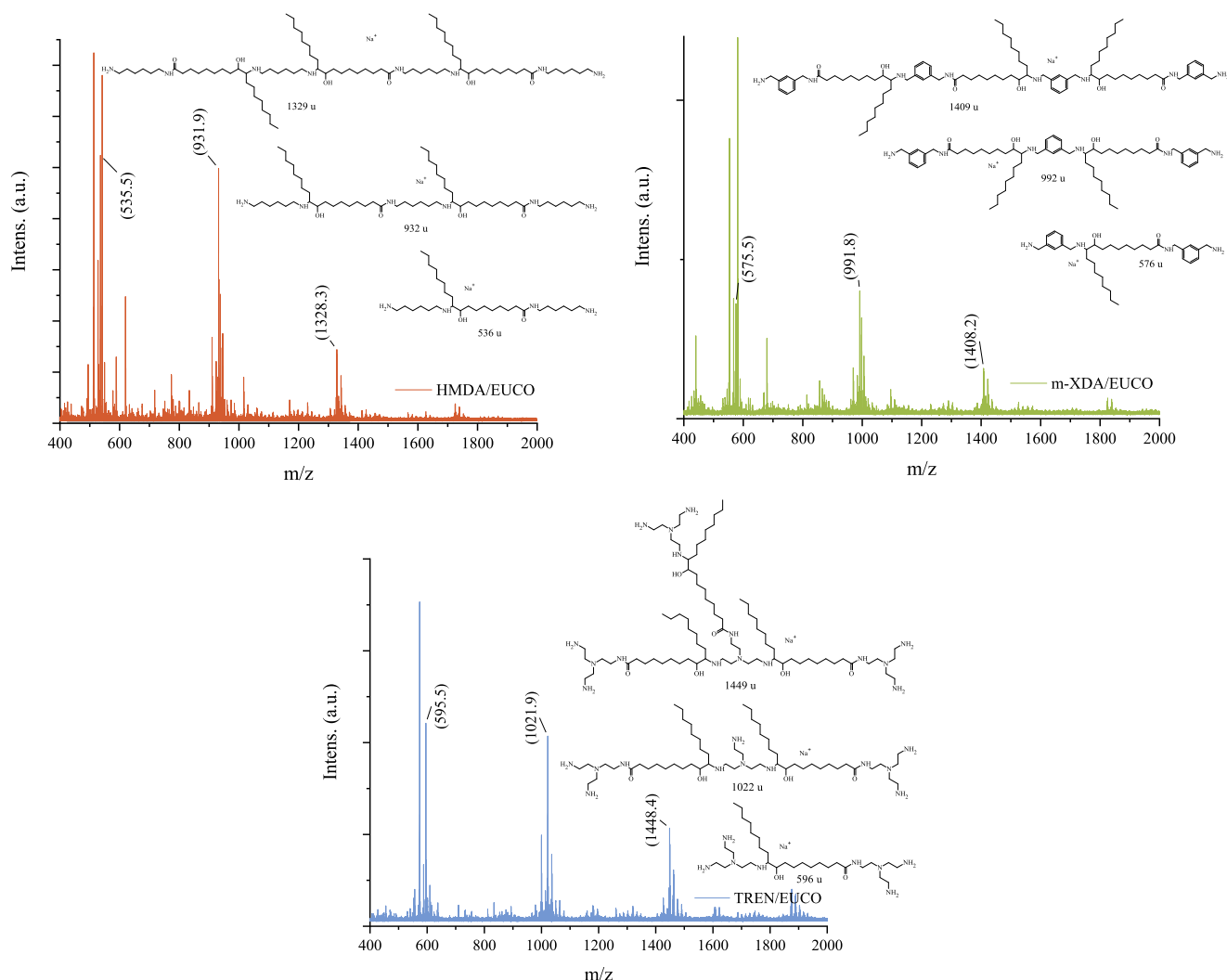


Fig. 5. a. MALDI-TOF spectra (2000 Hz, 355 nm, ionizing agent: CF_3COONa) of HMDA/EUCO. b. MALDI-TOF spectra (2000 Hz, 355 nm, ionizing agent: CF_3COONa) of m-XDA/EUCO. c. MALDI-TOF spectra (2000 Hz, 355 nm, ionizing agent: CF_3COONa) of TREN/EUCO.

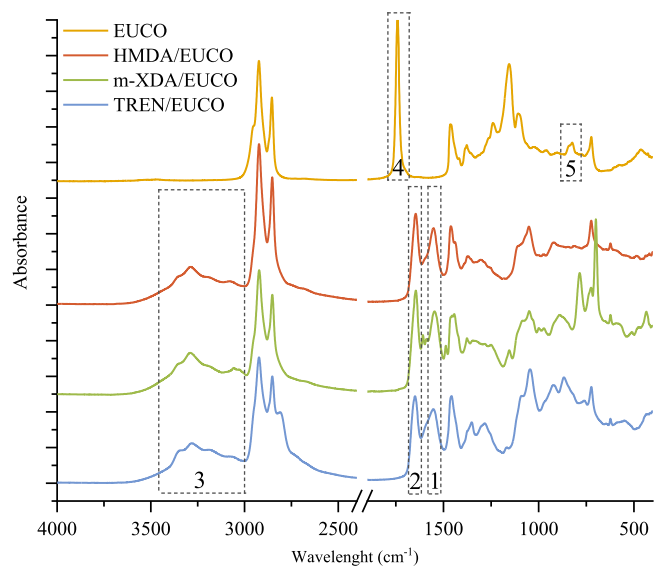


Fig. 6. FTIR spectra of synthesised bio-based amidoamines and EUCO.

Table 1

FTIR acquired information about functional groups of EUCO and bio-based amines.

Product	Region	Wavelength (cm^{-1})	Functional group	Vibration
EUCO	5	~ 840	Epoxide ring	ring stretching
	4	~ 1740	Ester band	$\text{C}=\text{O}$ stretching
HMDA/EUCO	3	3000–3400	Primary amines	NH_2 stretching broad
m-XDA/EUCO	2	~ 1640	Amide band	$\text{C}=\text{O}$ stretching
TREN/EUCO	1	~ 1545	Amide band	N-H deformation C-N stretching

3.6. GPC analysis of synthesized bio-based amines

The GPC chromatograms of the synthesized bio-based amines are depicted in Fig. 7. The chromatographic profiles clearly indicate the formation of both monomeric and oligomeric species of the ester bond aminolysis and epoxide ring-opening reactions, resulting in bio-based amines with β -amino alcohol and fatty acid amide functionalities. The principal peak corresponding to monomeric species appears at a retention time of approximately 15.7 min, whereas oligomeric structures, including dimers and trimers, are represented by a broad shoulder-like

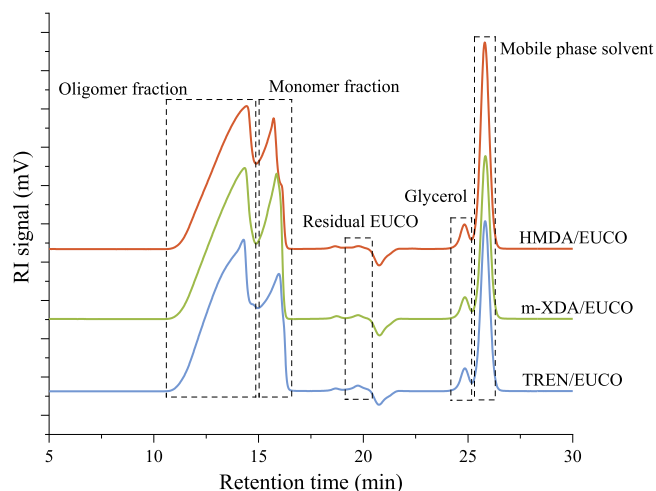


Fig. 7. GPC chromatograms of synthesised EUCO-based bio-based amines.

peak centered at approximately 14.4 min, indicating species with higher molecular weight. Following the complete conversion of triglyceride ester functionalities into amide groups, glycerol, released as a by-product of the aminolysis reaction, is detected at a retention time of approximately 25 min. The presence of glycerol was also confirmed by ^1H NMR spectroscopy, supporting the occurrence of aminolysis of ester bonds during synthesis. In addition, a small signal observed slightly below 20 min can be attributed to residual EUCO. Overall, the GPC results are in good agreement with the structural information obtained from NMR and MALDI-TOF analyses, confirming the formation of both monomeric and oligomeric species. Although chromatographic resolution does not allow precise differentiation between dimers and trimers, the broad oligomeric peak strongly suggests the presence of a mixture of higher molecular weight species, such as dimers and trimers. A similar approach has been reported by Pomilovskis et al. (2022), who employed GPC analysis to characterize bio-derived polyol product mixtures consisting of monomeric, dimeric, and trimeric species.

3.7. Amine value analysis of synthesized bio-based amines

Amine values of the synthesized bio-based amines obtained via aminolysis of ester bonds and oxirane ring-opening reactions are summarized in Table 2. The contributions of different nitrogen functionalities are reported as total amine value (AV_{total}), as well as estimated primary (AV_{prim}), and a combination of secondary and tertiary ($\text{AV}_{\text{sec+tert}}$) amine values.

Titrimetric determination of amine values for fatty amines and related systems is described in ASTM D2073 and ASTM D2074. However, both standards impose important limitations regarding their applicability to fatty amidoamines. ASTM D2073 supports the

determination of total, primary, secondary, and tertiary amine values for fatty amines and diamines, but only the total amine value is considered applicable for fatty amidoamines. Similarly, ASTM D2074 enables the determination of individual amine functionalities in fatty amines but is not intended for fatty amidoamines or fatty diamines.

Thus, quantitative determination of individual amine functionalities in multifunctional amidoamine systems remains challenging. Conventional titrimetric methods were developed for relatively simple fatty amines and rely on blocking primary amines with salicylaldehyde, followed by non-aqueous titration of the remaining basic nitrogen atoms. In contrast, triglyceride-derived amidoamines typically consist of mixtures of monomeric, dimeric, and oligomeric species containing amide groups and multiple nitrogen environments. The presence of amide functionalities and diverse steric environments around nitrogen atoms can influence the apparent basicity and accessibility of amine sites during titration. Consequently, differentiation between primary, secondary, and tertiary amines using classical titrimetric procedures may only provide approximate values. Therefore, the reported amine group distribution should be interpreted as an estimation derived from titrimetric analysis.

The content of primary amine groups was calculated as the difference between the total amine value and the sum of secondary and tertiary amine values. In theoretical calculations based on monomeric structures, about 66% of the amine value is contributed by primary amines, and 33% by secondary amines, for HMDA/EUCO and m-XDA/EUCO. If we compare these values to titration data, it can be seen that the titrated secondary/tertiary amine for HMDA/EUCO has a slightly lower value, with a difference of about 15 mg KOH/g, while m-XDA/EUCO has blocked primary amine more, making the difference between theoretically calculated and titrated at about 10 mg KOH/g. These values being close suggest that, while primary amine blocking with salicylaldehyde is hindered to some extent in amidoamines systems, the results show that the titration method can be used to estimate the contribution of different amine groups to the total amine value.

In case of using TREN as a reactant to obtain bio-based amines it makes each amine value input more difficult because there is a tertiary amine nitrogen already within the TREN molecule. In this case, using the titration method, we cannot separate secondary and tertiary amine values. The separation of two, as suggested by standards, is usually done using acetic anhydride; however, it will also cause a reaction with the hydroxyl groups present in the synthesised product mixture, making the titrated value unreliable.

Nevertheless, assuming idealised molecules, we can calculate the theoretical input in total amine value for each amine using NMR and MALDI-TOF analysis. In this case based on monomeric structure, we concluded that primary amine input is roughly 57%, secondary is 14% and tertiary amine input is 29%. When these values are compared to titrated data, it can be seen that the difference is around 50 mg KOH/g, which is substantially higher when compared to differences using bifunctional amines. In TREN-derived bio-based amines, the

Table 2
Amine value contribution of the obtained EUCO-based bio-based amines.

Bio-based amine	Amine value in mg KOH/g					Bio-based content ^{****} (wt%)
	AV _{total}	AV _{prim}		AV _{sec+tert}		
		Titr.	Theor.	Titr.	Theor.	
HMDA/EUCO	310 ± 5	222 ± 4	207	88 ± 3	103*	55
m-XDA/EUCO	280 ± 2	200 ± 2	190	80 ± 2	90*	51
TREN/EUCO	465 ± 6	210 ± 5	268	255 ± 5	66*	49
Priamine™ 1074	205 ± 4	205 ± 4	-	-	-	100 ^{***}

* Calculated secondary amine contribution.

** Calculated tertiary amine contribution.

*** Bio-based content, according to ASTM D6866 and EN 16640 (Cargill, 2024) <https://www.cargill.com/bioindustrial/doc/1432234433707/adhesives-brochure.pdf> (accessed 11 March 2026).

**** Calculated.

salicylaldehyde blocking efficiency can be influenced by tertiary amine groups in product mixture. The presence of these tertiary centers, together with the diverse steric environments around nitrogen atoms, may alter the basicity and accessibility of amine sites during titration. This may result in incomplete masking of primary amines and consequently an overestimation of secondary and tertiary amine content.

Also, for comparison, the fully bio-based Priamine™ 1074, a commercial fatty amine, shows an AV_{total} of 205 ± 4 mg KOH/g, which corresponds exclusively to primary amine groups. The synthesized bio-based amines exhibited higher total amine values, reflecting the incorporation of additional reactive functionalities through the combined oxirane ring-opening and ester aminolysis reactions. Unlike conventional fatty amines, the obtained products contain not only primary amine groups but also secondary amines, β -amino alcohols, and amide functionalities. Consequently, the synthesized products possess a multifunctional structure that may provide additional opportunities for network formation and intermolecular interactions during subsequent polymer synthesis. Furthermore, the use of different amines (HMDA, m-XDA, and TREN) enables tuning of the chemical structure, amine functionality, and physicochemical properties of the obtained products. Overall, the synthesized bio-based amines achieve 49–55% renewable content (Table 2), thereby combining renewable feedstock utilization with a multifunctional molecular structure.

However, it should be acknowledged that, despite the use of a renewable waste-derived feedstock, several reagents and catalysts employed during the synthesis, including zinc (II) perchlorate hexahydrate and multifunctional amines, are not inherently renewable and may present environmental and safety concerns. The synthesized amines are therefore considered bio-based due to the incorporation of used cooking oil into their molecular structure. Nevertheless, the developed methodology benefits from solvent-free reaction conditions, a one-pot synthesis strategy, and does not require washing, extraction, or extensive purification steps, thereby reducing solvent consumption and process waste generation.

4. Conclusions

The synthesis and comprehensive characterization of three bio-based amines derived from epoxidized used cooking oil (EUCO) were successfully demonstrated under solvent-free conditions using $Zn(ClO_4)_2 \cdot 6H_2O$ as a Lewis acid catalyst. The combined FTIR NMR, MALDI-TOF, and GPC/SEC analyses confirmed that the reaction proceeds through both nucleophilic oxirane ring-opening and triglyceride ester aminolysis, resulting in multifunctional bio-based amines containing amide, β -amino alcohol, and amine functionalities. Furthermore, the analytical data demonstrated the presence of both monomeric and oligomeric species within the synthesized products. The obtained bio-based amines exhibited total amine values ranging from 280 to 468 mg KOH/g and viscosities ranging from 12,000 to 90,000 mPa.s. By combining amine titration, primary amine determination, and detailed structural characterization, reliable estimations of amine group contributions were obtained. In addition to primary amine groups, the synthesized products also contain secondary amine functionalities, providing increased functionality that may influence network formation and dynamic behavior in subsequent polymer synthesis. Overall, this work demonstrates a viable route for the valorization of used cooking oil into multifunctional bio-based amines and provides detailed structural insight into these materials. The obtained products broaden the range of multifunctional bio-based amines available from renewable and waste-based feedstocks and provide promising intermediates for subsequent polymer synthesis. Future work will explore their utilization in dynamic polymer networks and other advanced polymer materials.

CRedit authorship contribution statement

Ralfs Pomilovskis: Writing – review & editing, Software, Formal

analysis, Data curation. **Arnis Abolins:** Writing – review & editing, Writing – original draft, Visualization, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Sergejs Gaidukovs:** Writing – review & editing, Supervision. **Mikelis Kirpluks:** Writing – review & editing, Supervision, Formal analysis. **Olga Kockova:** Formal analysis. **Anda Fridrihsone:** Writing – review & editing, Project administration, Funding acquisition, Formal analysis.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Conflicts of interest

The authors declare no conflict of interest.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.indcrop.2026.123854](https://doi.org/10.1016/j.indcrop.2026.123854).

Data availability

Data will be made available on request.

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