# PLANT BASED RESINS OBTAINED FROM EPOXIDIZED LINSEED OIL USING A HETEROGENOUS HYDROTALCITE CATALYST

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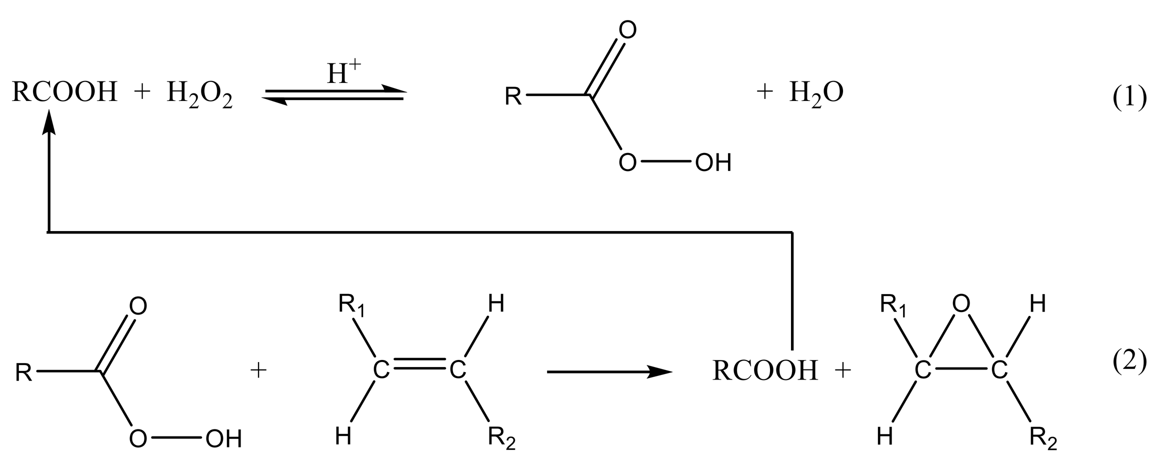
## Introduction

Vegetable oil is a plentiful renewable raw material utilized to replace petroleum-based chemicals used in industry, with the purpose of reducing pollution. Because of the double bonds present in the alkyl chain of the unsaturated fatty acids found in vegetable oil, functionalization reactions such as metathesis, hydroformylation, and epoxidation can be used to produce a wide range of compounds, including intermediaries that can be used as monomers in the polymer industry. The epoxidation reaction is significant for the macro industry because it produces versatile epoxidized vegetable oils (EVO) that can be cured with a range of substances (amines, acids, anhydrides) or undergo ring opening reactions, resulting in useful end-products and intermediaries. Anhydrides are some of the most efficient curing agents for EVO, leading to thermoset polymers with thermo-mechanical properties heavily influenced by the epoxy content of the vegetable oil and the anhydrides used. Lewis bases, such as tertiary amines or imidazole, catalyze the curing reaction. The purpose of this research is to create materials by curing epoxidized linseed oil with phthalic anhydride (PA) utilizing a low-cost base heterogeneous catalyst, resulting in a cleaner and more ecologically friendly approach. The catalyst is MgAl hydrotalcite, which is a layered double hydroxide (LDH), a class of materials with the $[M^{2+}_{1-x}M^{3+}_{x}(OH)_2]^{x+}[A^{n-}_{x/n}]\cdot mH_2O$ general formula. This catalyst was not previously reported in literature for this reaction, but it is instead well characterized and used for a broad range of reactions.

## Results and discussions

**The mechanism of the epoxidation reaction used**



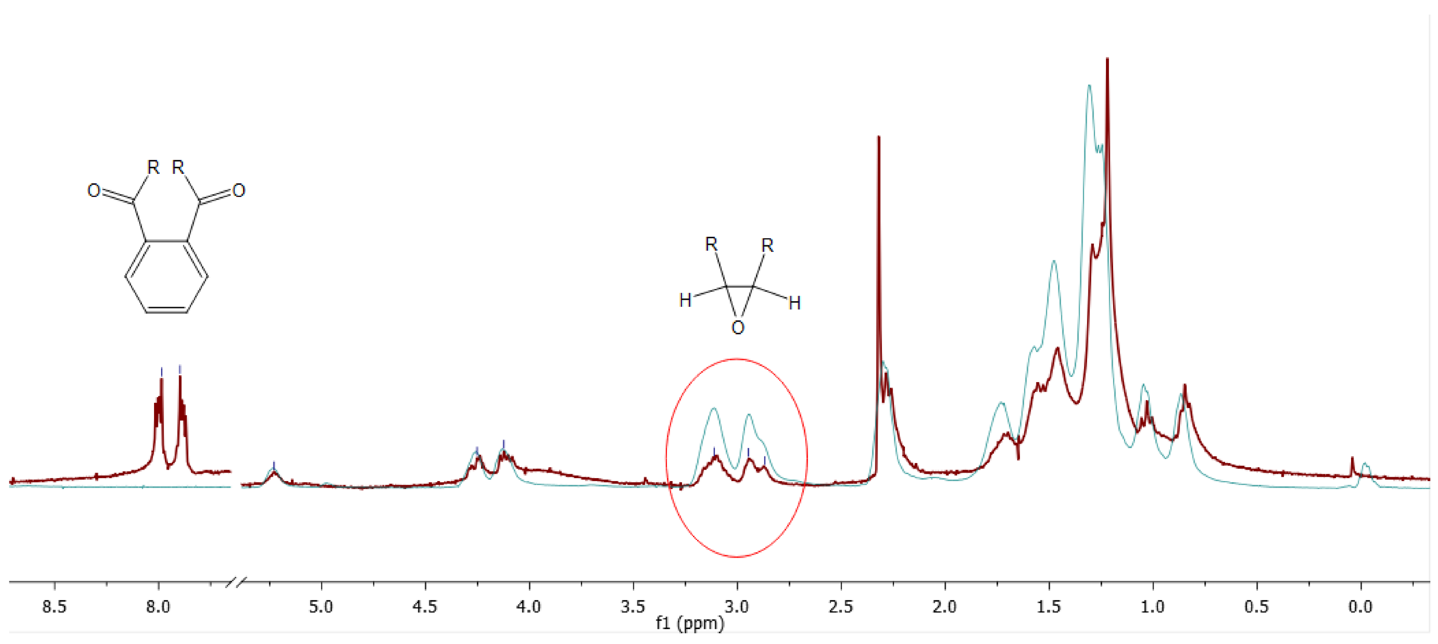

(1) the generation of the peracid in situ

(2) the subsequent epoxidation of the substrate

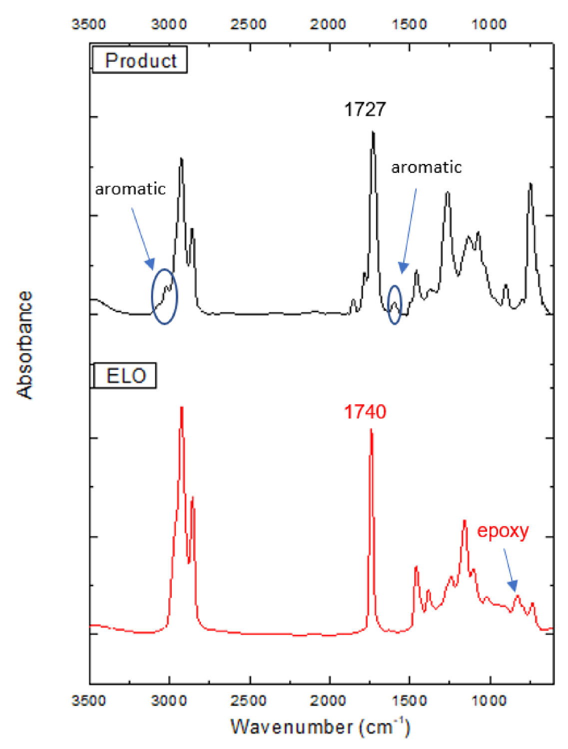
**Curing reaction conditions:**

- ELO (epoxidized linseed oil) and PA in different molar ratios were dissolved in toluene solvent
- MgAl LDH catalyst (10% wt)
- The mixture was heated to 100 °C under constant magnetic stirring for 24 h
- The reaction mixture was cooled, and the catalyst was filtered
- The organic liquid phase was washed with water 4 times for the removal of unreacted PA
- The solvent from the organic phase was removed under vacuum.

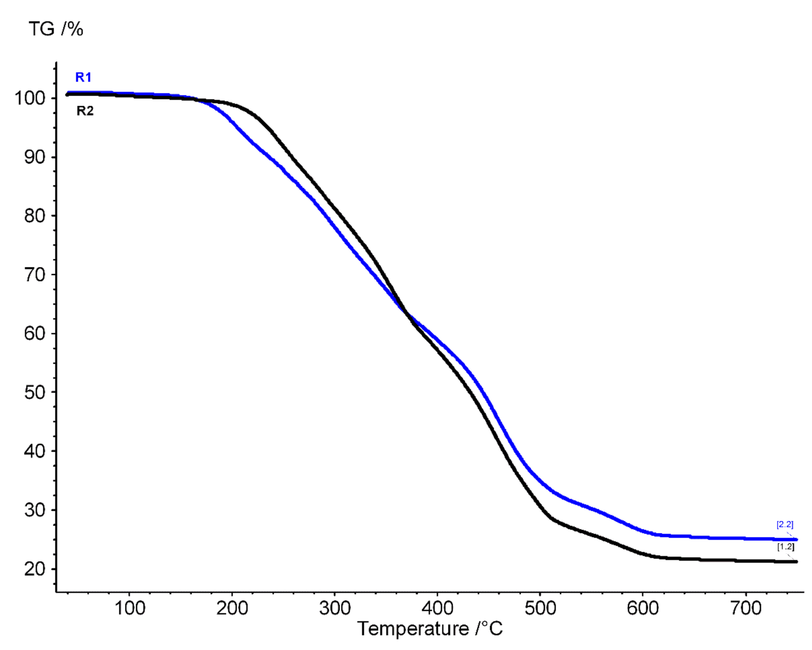
**The superimposed $^1$H NMR spectra of the ELO (green) and the partially epoxy ring-opened liquid product (red) – molar ratio of epoxy groups : PA = 1 : 0.6**




**FT-IR spectra of ELO (red) and the obtained product from the partial ring-opening (black)**



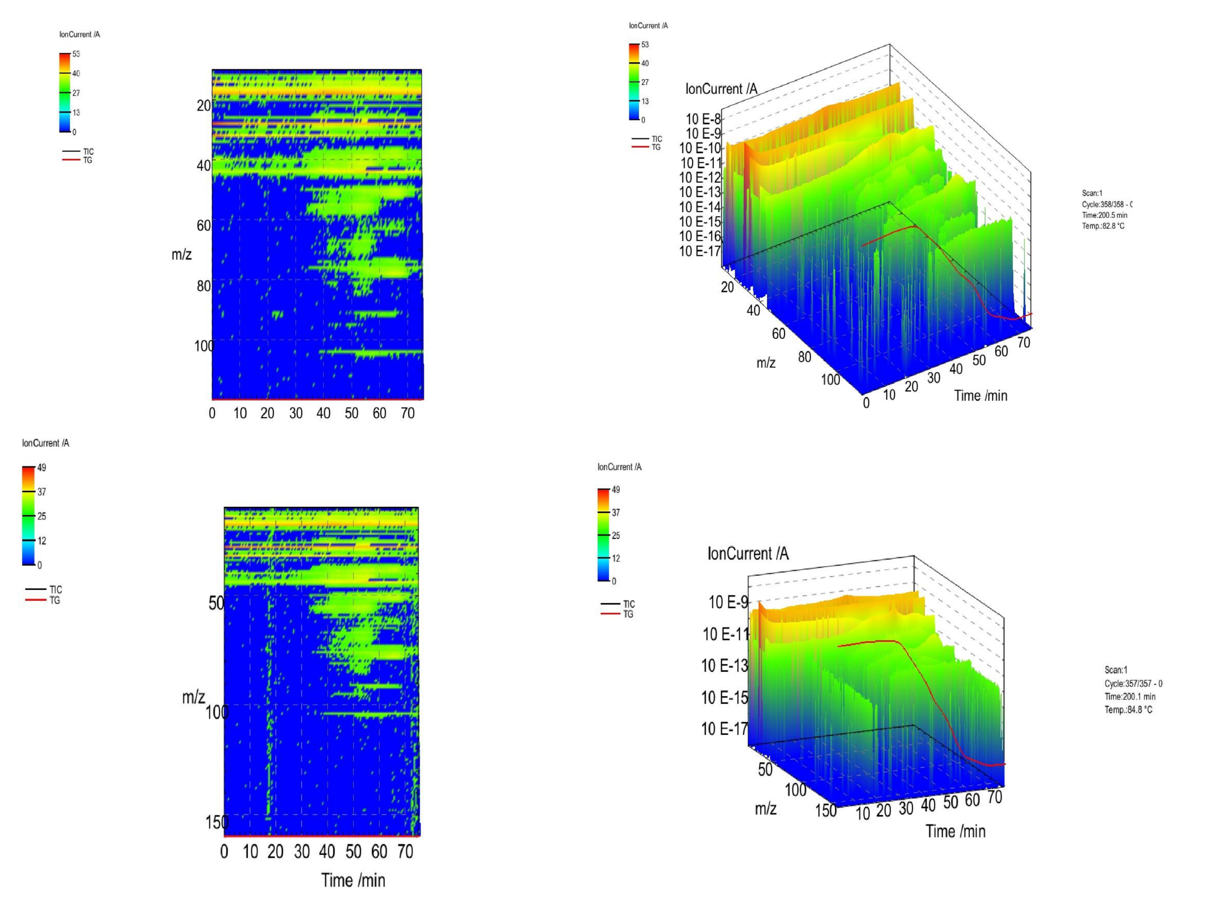

**The TGA thermogram showing the degradation behavior of the resins**




R1 – epoxy : PA ratio = 1 : 1.2

R2 – epoxy : PA ratio = 1 : 1.5

**MS spectra (2 D – left, and 3 D – right) of R1 (up) and R2 (bottom), showing the thermal degradation behavior of the samples**




## Conclusions

- Cured materials with different thermo-mechanical properties were obtained by reacting ELO and PA in different molar ratios, in the presence of a solid reusable catalyst, MgAl LDH.
- The obtained materials were analyzed using $^1$H-NMR, FT-IR spectroscopy, and TGA-DSC-MS. Spectroscopy showed the ring-opening of the epoxy groups and the appearance of phthalic moieties in the obtained product.
- The curing was confirmed by the thermal analysis of the solid products, which shows the thermal degradation behavior of the samples, as well as the fragments leaving the product during this process.
- These results show that using MgAl LDH catalyst is a viable alternative for curing epoxidized vegetable oil with PA, in order to obtain interesting materials.

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