Optimization of the curing of liquified wood into epoxy networks

Thermosets for a greener coatings industry

Joumad, H.; Rump, E.; Coulibaly, M; Zhou, Q.
Project/Research Group: Rewoody/BBB&P
Contact information: h.joumad@student.avans.nl
Date: June 2025

Introduction

With the continuous depletion of fossil fuels and the many complications that come with the use of it, the search for alternatives has become an increasingly important point of research. The sources should not only be renewable, but the products should also either be biodegradable or recyclable. High fractions of liquified wood (HLW) is a product that was found to be applicable to both. It has great potential to serve as a recyclable coating for wood once cured into thermosets [1].

Figure 1: HLW usage cycle [2]

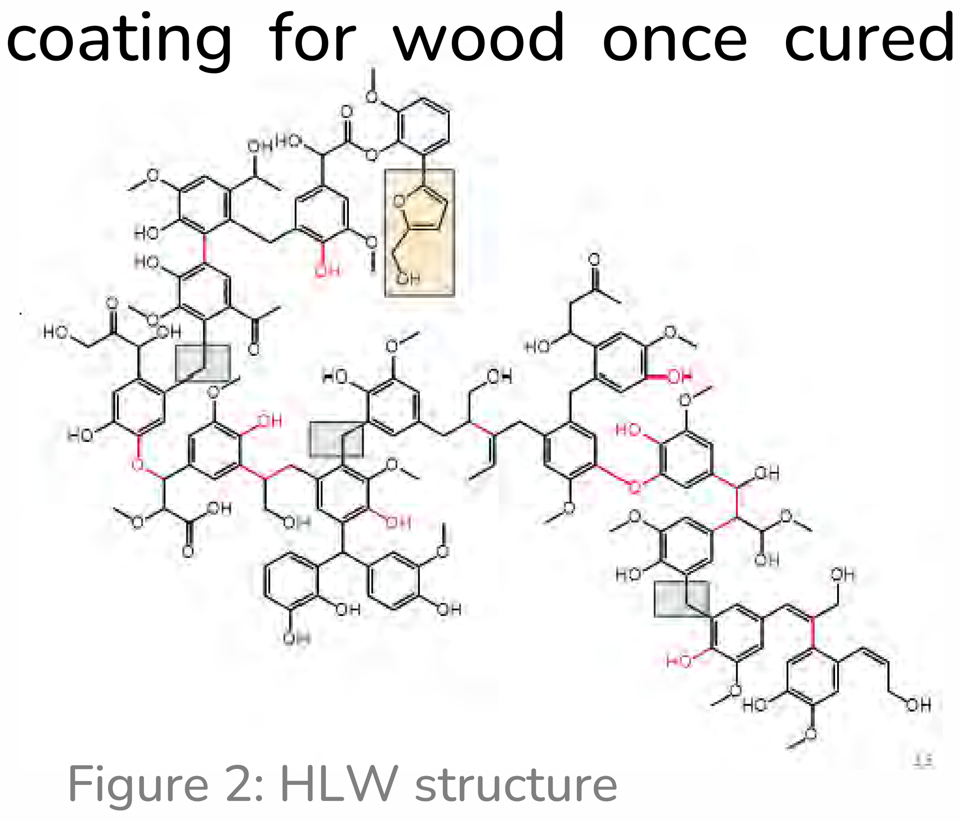

Figure 2: HLW structure

This research sought to optimize the reaction of HLW with glycerol diglycidyl ether (GDGE) to create these thermosets, using different concentrations of N,N-dimethyl benzylamine (BDMA), 2-ethyl-4-methylimidazole (EMI-24), and zinc acetylacetonate (Zinc(acac)$_2$)

Methodology

Figure 3 shows an overview of the curing process and the different values used for the samples with a 1:1 and 1,2:1 epoxy:hydroxyl ratio.

1,2:1

0,5 / 1 / 4 wt%
BDMA / EMI-24 / Zinc(acac)$_2$

Time: 1 / 7,5 / 15h
Temp: 150C

Mix HLW, GDGE and catalyst

⇩

Cure

⇩

Characterize with DSC and Soxhlet extraction

1,2:1

1 wt% EMI-24

Time: 7,5 + 15h
Temp: 150 + 180C

Figure 3: Curing process

DSC analysis was used to determine thermal properties and the gel content was determined by Soxhlet extraction with tetrahydrofuran.

Results

The gel contents of the stoichiometric variations didn't vary much between 7,5 and 15 hours, so only 7,5h is showcased. it is likely that the crosslinking stops around 7.5 hours but never reaches 100%

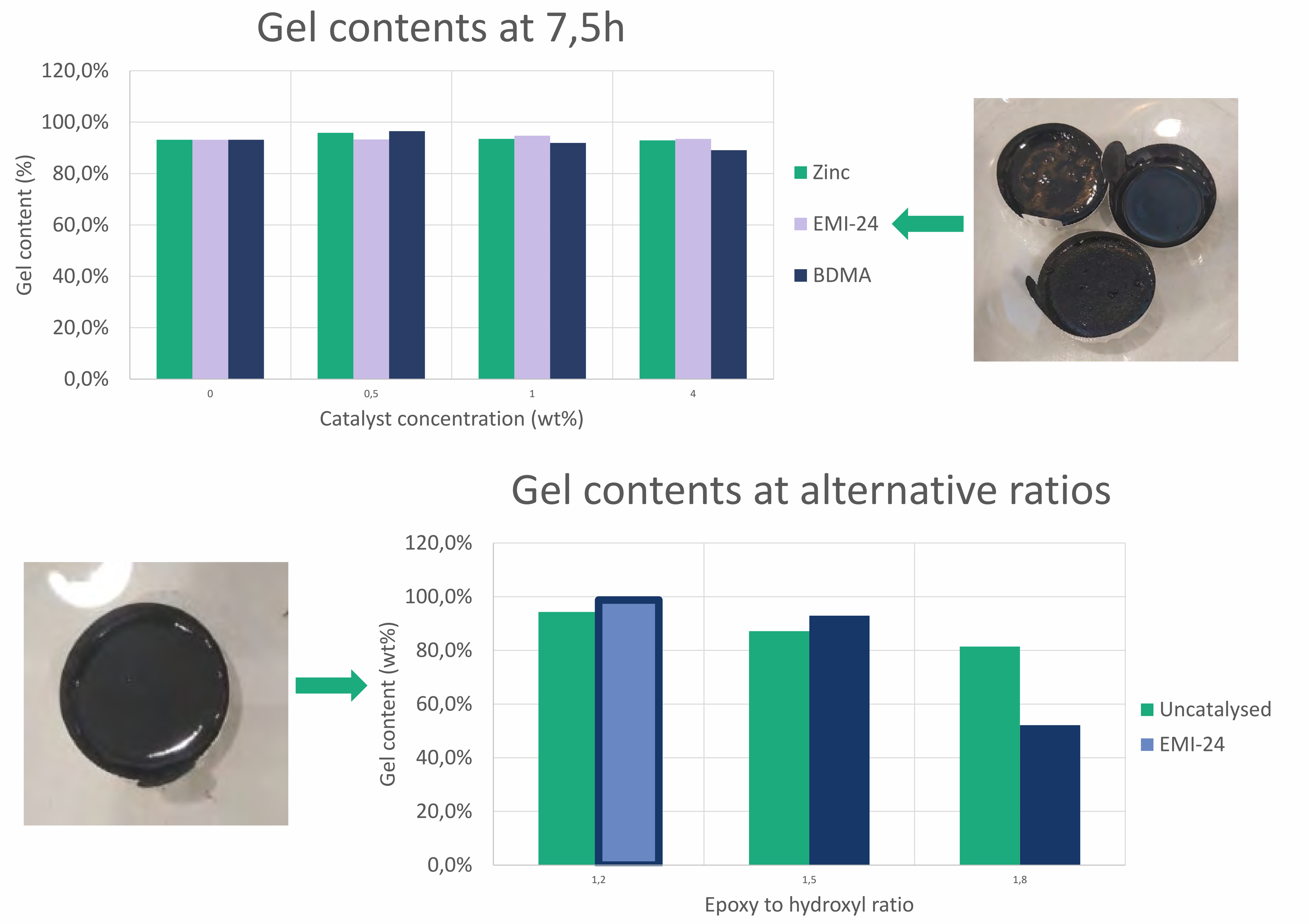

Figure 4: Gel contents of 1:1 at 7,5h (top) and alternative ratios (bottom) and examples of the samples

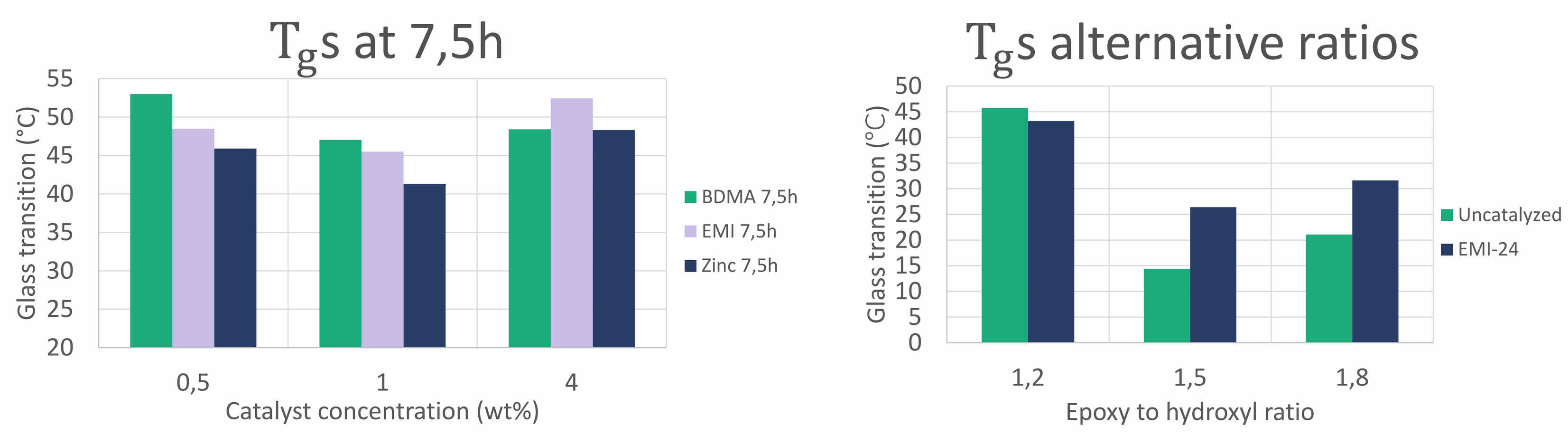

Figure 5: Overview of the T_gs as determined from DSC analysis

1:2 with 1wt% EMI-24 as coating on Spruce

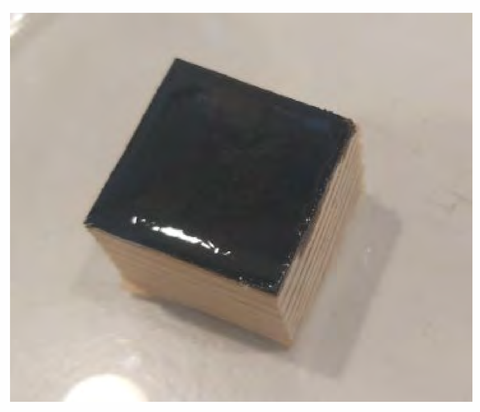

Figure 6: Coating tested on spruce wood

Conclusion

From the gel content determinations it can be concluded that from the variations used, a 1,2:1 epoxy:OH ratio catalyzed with 1wt% EMI-24 cured for 7,5h at 150°C and post-cured for 180°C results in the best crosslinking, with a conversion of 99% and a T_g of 45°C as determined by the DSC analysis.

Acknowledgements & References

This publication is part of the project Rewoody with file number KICH1.ED01.20.002 of the research programme Circulariteit which is financed by the Dutch Research Council (NWO)

1. Q. Hu, R. P. Martinho, S. Azhdari, J.-P. Lange, M. van Drongelen, M. P. Ruiz en F. R. Wurm, „Fully Bio-Based Epoxy Resins from Liquified Wood for Chemically Recyclable Wood Coatings," *Adv. Funct. Mater.*, 16 April 2025.

2. M. P. Ruiz, J. Mijnders, R. Tweehuysen, L. Warnet, M. van Drongelen, R. A. Kersten en J.-P. Lange, „Fully Recyclable Bio-Based Thermoplastic Materials from Liquefied Wood," *ChemSusChem*, pp. 4395-4399, 2 September 2019