Chemical recycling of amine cured epoxy resins

The optimalization of solvolysis of amine cured epoxide resins and identification of the reaction products.

Cas de Jong, Qian Zhou; Viktor van Mun

Project/Research Group: C-Recycle | Biobased Resources & Energy

Contact information: c.dejong6@avans.nl; q.zhou1@avans.nl; vfg.vanmun@avans.nl

Date: 12 June 2025

Introduction

Epoxy resins are a type of polymer that is hard and chemically resistant, which makes it useful as a material for windmills, electronic devices and coatings. These uses lead to more epoxy resins being made and being thrown away[1]. The epoxy resins that get thrown away often end up on landfills and only a small amount gets recycled. Recycling epoxy resins can be difficult, because it can not be molten and due to its chemical resistance, it is difficult to degrade.

Epoxy resins are molecules made from of a backbone with an epoxide group on it and a curing agent with a group to react with the epoxide. A common example is the diglycidyl ether of bisphenol-A (DGEBA), typically cured with hexamethylenediamine (HMDA). Through solvolysis, the ether bonds in the epoxy resin can be broken, allowing for the recovery of bisphenol-A (BPA))[2].

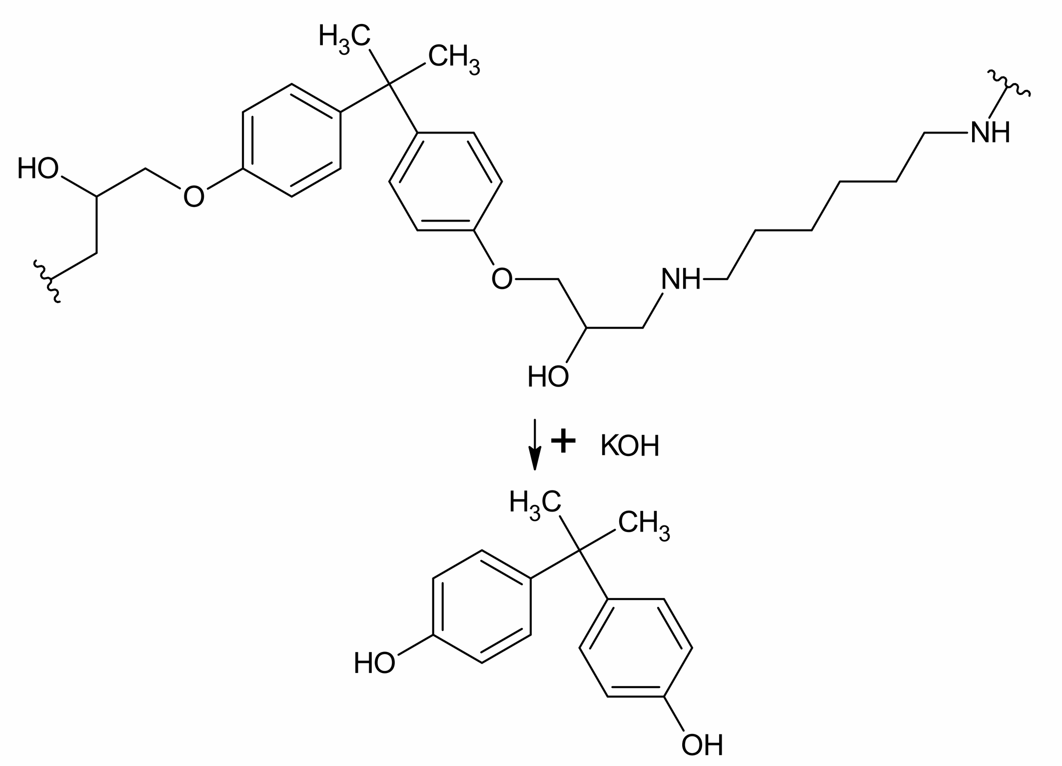

Figure 1: Schematic reaction of an epoxy resin made from the diglycidyl ether of bisphenol-A and hexamethylenediamine to form bisphenol-A using KOH

The aim of this project is to optimize the solvolysis of epoxy resins and to identify the products of this reaction.

Methodology

1: Method optimization

- Temperature & time
- Amount of solvent
- Amount & type of base

2: Product identification

Solid product

- Infrared spectroscopy
- Thermogravimetric analysis

Product in solution

- Mass spectrometry
- High Pressure Liquid Chromatography

Figure 2: Schematic overview of the 2 parts this project and planned tests

Results & discussion

Figure 3-5 present the effects of potassium hydroxide (KOH) amount, solvent amount, and reaction temperature and time on the BPA yield. The results suggest that having at least twice as much KOH as ether bonds is sufficient to achieve a high BPA yield, and excess KOH does not provide a substantial benefit.

Lower amounts of solvent lead to higher BPA yields. This likely happens because the concentration of KOH is higher in less solvent, making the reaction more efficient.

Increasing the temperature accelerates the reaction and raises the BPA yield, but too high a temperature (like 190°C) may eventually lead to a decrease in BPA yield after 3h, possibly due to degradation of BPA.

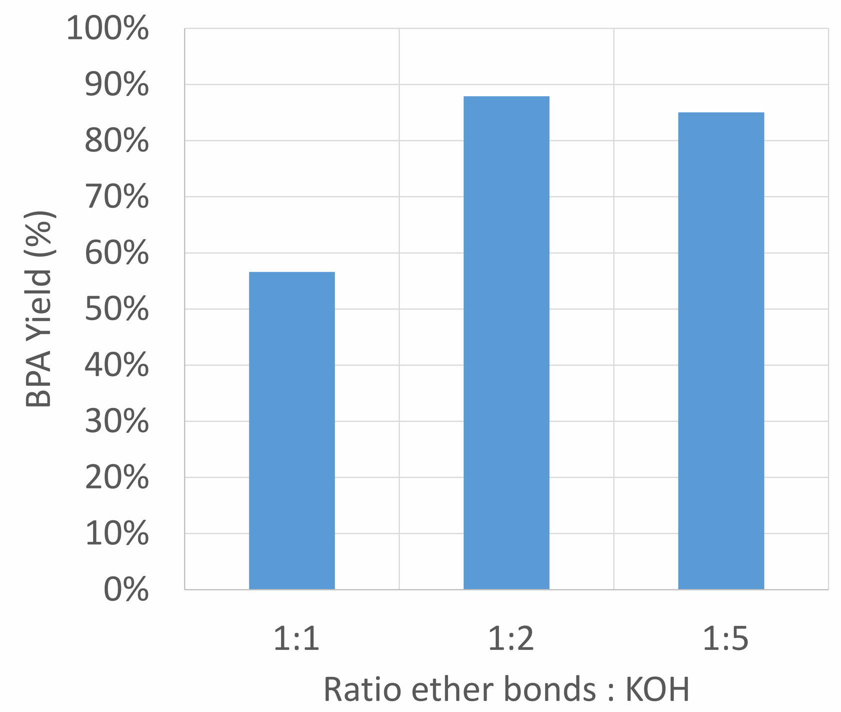

Figure 3: Effect of KOH amount on BPA yield

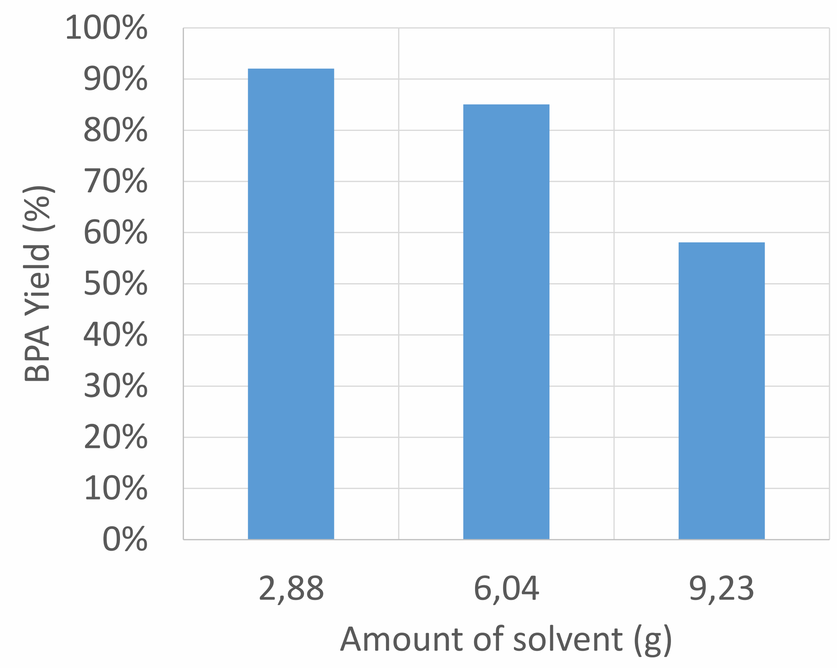

Figure 4: Solvent amount on BPA yield

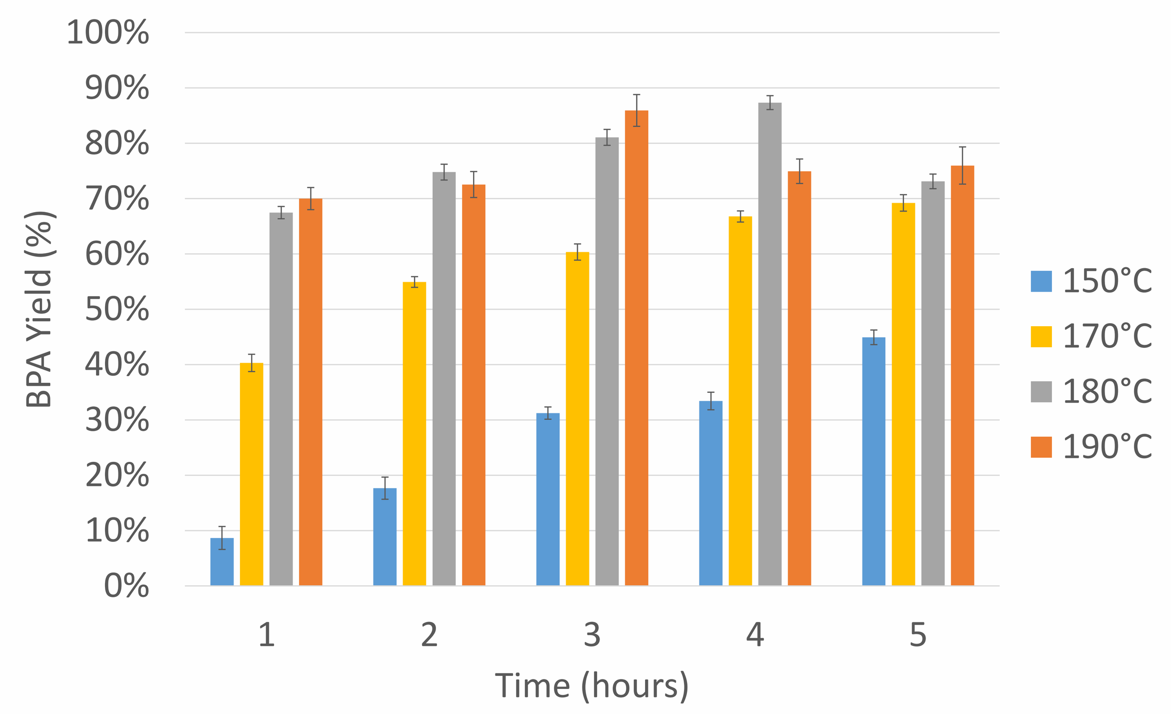

Figure 5 Effect of reaction temperature and time on BPA yield

Conclusion

It was found that it is possible to degrade amine cured epoxy resin using solvolysis. A 2:1 ratio of KOH to ether bonds is sufficient for high BPA yields (~88% and reducing the amount of solvent increases the yield (up to 92% at 2.88 g solvent). Higher temperatures accelerate the reaction and increase BPA yields (maximum at 85% in 3 hours at 190 °C), though BPA yields may decline if the temperature is too high or the reaction runs too long. The next step in this project will be to identify and characterize the byproducts formed during the reaction.

References

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2. Y. Minami, T. Tsuyuki, H. Ishikawa, Y. Shimoyama, K. Sato, and M. Yoshida, “Degradation of stable thermosetting epoxy resins mediated by bases in amide solvents,” Polym J, 2024, doi: 10.1038/s41428-024-00979-6.

Acknowledgements

The authors acknowledge the financial support of the Just Transition Fund (JTF) grant from the European Commission (grant number JTFZ-00101).

JTF Fonds voor rechtvaardige transitie

Medegefinancierd door de Europese Unie