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Synthesis and Characterization of a Tris(phenolate)amine Ligand for the Production of an Iron(III)-centered Catalyst

Caden Pensak
Juniata College, PENSACX17@juniata.edu

Marie Heidledr
Juniata College

Abby Betar
Juniata College

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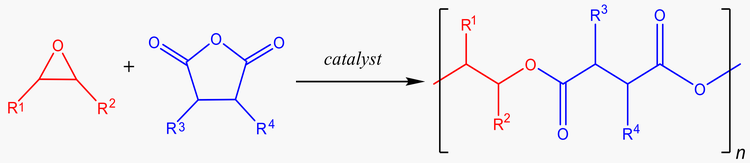
Caden Pensak, Marie Heidler, Abby Betar, Dr. Ursula Williams
Department of Chemistry
Juniata College

Introduction

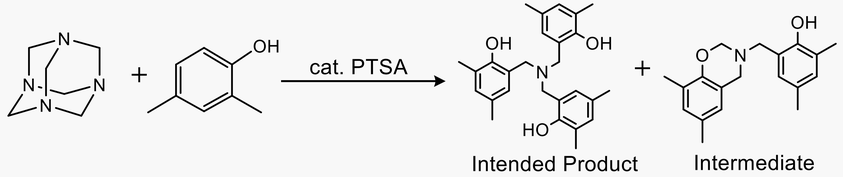
- The use of tris(phenol)amines in the synthesis of iron(III)-based catalysts has been essential to the production of high glass-transition aliphatic and semiaromatic polyesters from epoxides and cyclic anhydrides.[1]



- Many aliphatic and semiaromatic polymers can be produced from sustainable resources, which serve as a great alternative to petroleum based products.
- Aliphatic polymers degrade very easily and can be used for a range of medical purposes, due to their high biocompatibility.
- High glass-transition polymers can be used in higher stress environments, such as machinery.

Research Objectives

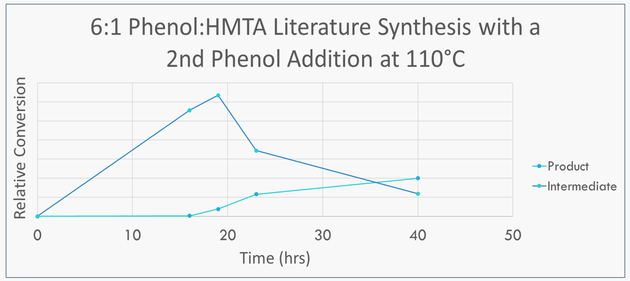
- Our goal is to optimize the literature synthesis of the catalyst by improving the ligand synthesis considering the following variables:[2]
  - Time
  - Temperature
  - Stoichiometric ratio
  - Purification methods

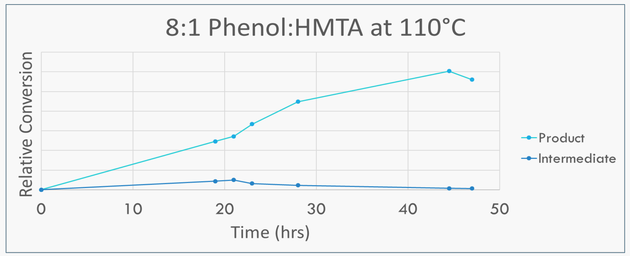


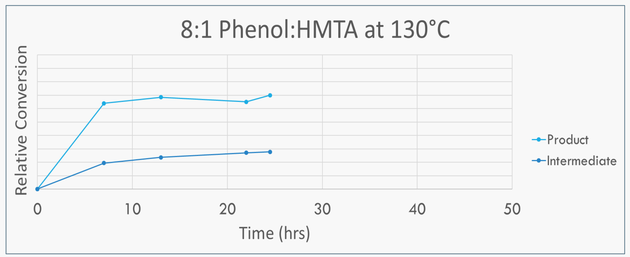
Results

Stoichiometric ratio (phenol:HMTA)	Temperature (°C)	Time (hrs)
8:1	110	24
6:1	130	40
		48

Experiments monitored via ^{1}H NMR







Purification Methods

- 4 methods were tested to purify crude reaction mixtures:
  - Recrystallization from acetone
  - Column chromatography in dichloromethane
  - Recrystallization in diethyl ether and hexanes
  - Trituration in 2-propanol, followed by acetone recrystallization
- The only method that resulted in pure product at reasonable yields was the trituration technique.

Conclusions

- Increased stoichiometric ratio eliminates the need of a 2nd addition of 2,4-dimethylphenol
- Longer reaction times maximize conversion to product
- Trituration in 2-propanol is a critical purification step.

Future Goals

- Establish a proper internal standard for quantitative NMR
- Use isolated product for future metalation reactions to synthesize the catalyst
- Evaluate the potential of this and related compounds to act as a catalyst in polymer chemistry

Acknowledgments

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References

(1) Sanford, Maria J., et al. *Macromolecules*, **2016**, 49, 6394.
(2) Chandrasekaran, A., Day, R. O.; Holmes, R. R., *J. Am. Chem. Soc.*, **2000,** 122, 1066