Optimization of bisguaiacol F Synthesis via Microwave Assistance

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LIBERTY UNIVERSITY

Presentation Overview

- Introduction to BPA
- Background on BGF
- Research Objective, Results, and Plans

Introduction to BPA

BPA Uses

- Widely used chemical
- 5.5 million tons produced annually in United States alone

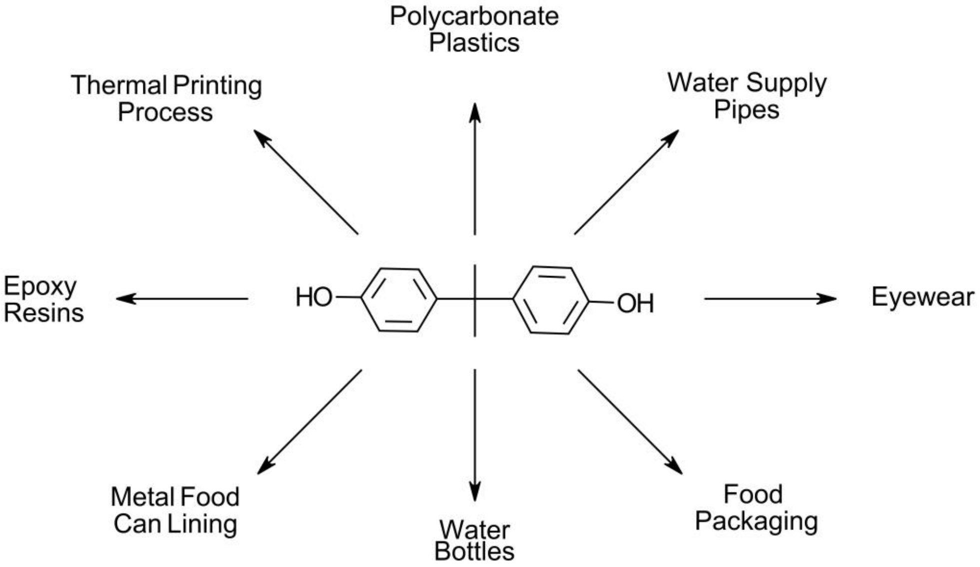

Figure 1. BPA Uses (Created by Jessica Bryant)

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Downsides of BPA

- Synthesized from fossil fuels
- Significant energy requirement
- Estrogenic compound
- Endocrine disruptor that impacts:
  - Diabetes
  - Obesity
  - Reproductive disorder
  - Respiratory Disease
  - Cardiovascular disease

Background on BGF

BGF: Potential Alternative for BPA

- Similar thermal, mechanical, and structural properties compared to BPA
- Synthesized from naturally occurring organic molecules
- Lacking estrogenic and endocrine disrupting characteristics

Figure 3. Bisguaiacol F Product (Photos by Jessica Bryant)

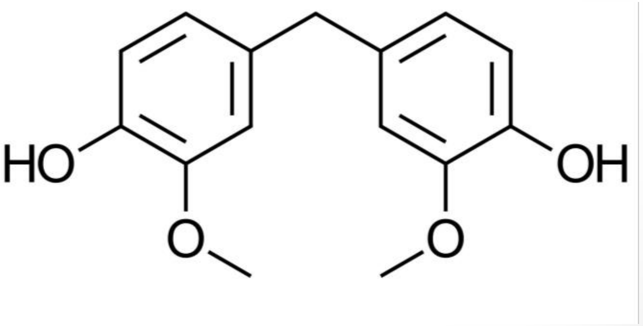
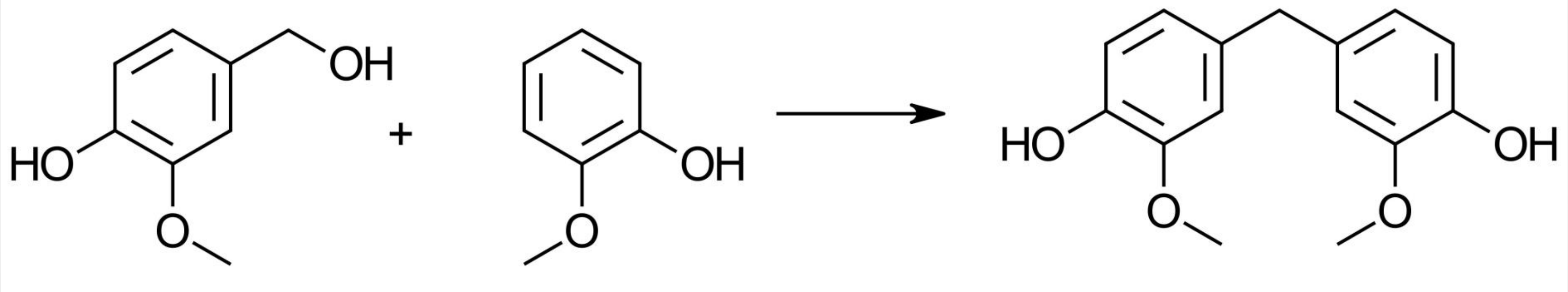


Figure 4. BGF Structure (Created by Dr. Stephen Hobson)

BGF Synthesis



Vanillyl Alcohol	Guaiacol	BGF

Created by Dr. Stephen Hobson

CEM Discover 2.0 Microwave Reactor

- Rapidly reaches high temperatures
- Decreases reaction time
- Reduces side reactions

Figure 6. Microwave Reactor (Photo by Jessica Bryant)

CEM Discover 2.0 Microwave Reactor

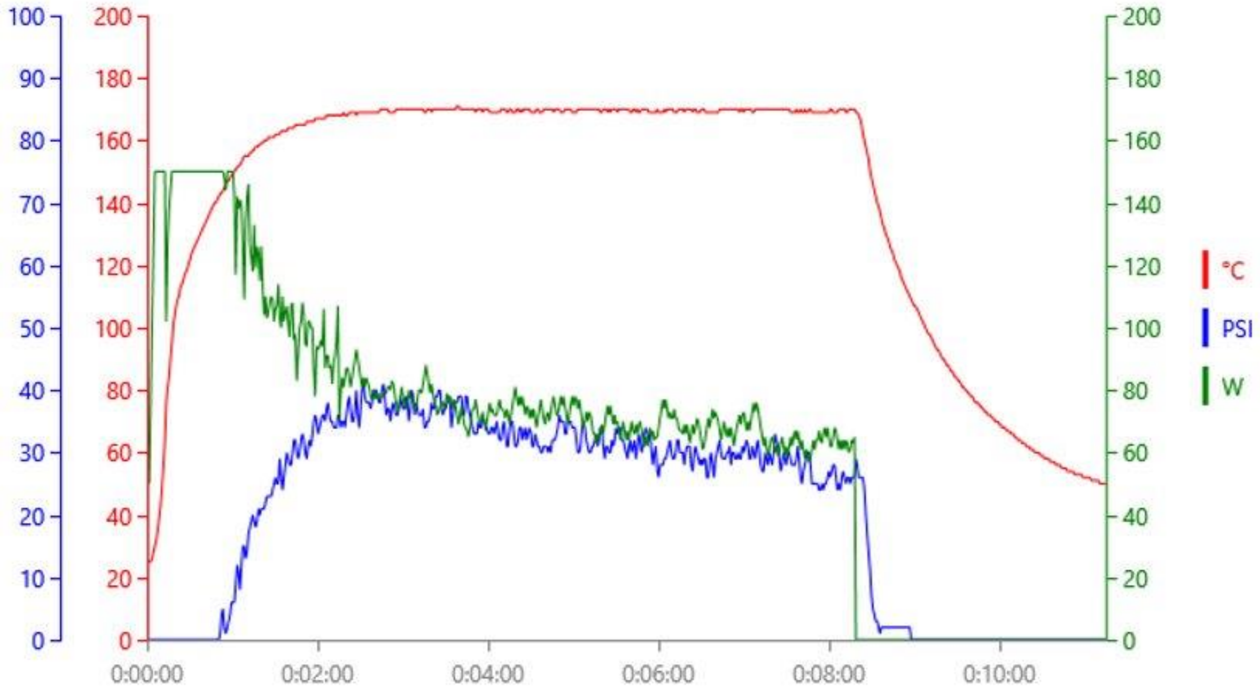


Figure 7. Temperature/Pressure/Power During Microwave Reaction (Photo by Jessica Bryant)

BGF Preliminary Results

- Control syntheses of BGF completed using conventional heating methods with water as the solvent and Amberlyst resin as an acid catalyst at 65 °C and 100 °
- BGF synthesis at 100 °C resulted a mixture of *p,p'*-BGF and *o,p'*-BGF isomers as indicated by the broader MP (88.4-94.5 °C).
- Moderate yields of BGF isomers obtained through laborious workup and purification

Reaction	Temperature	Percent Yield	Melting Point
1	65 °C	26.7%	82.5-95.6 °C
2	100 °C	56.7%	88.4- 94.5 °C

Table 1. Control BGF Synthesis (Created by Jessica Bryant)

TLC Comparisons

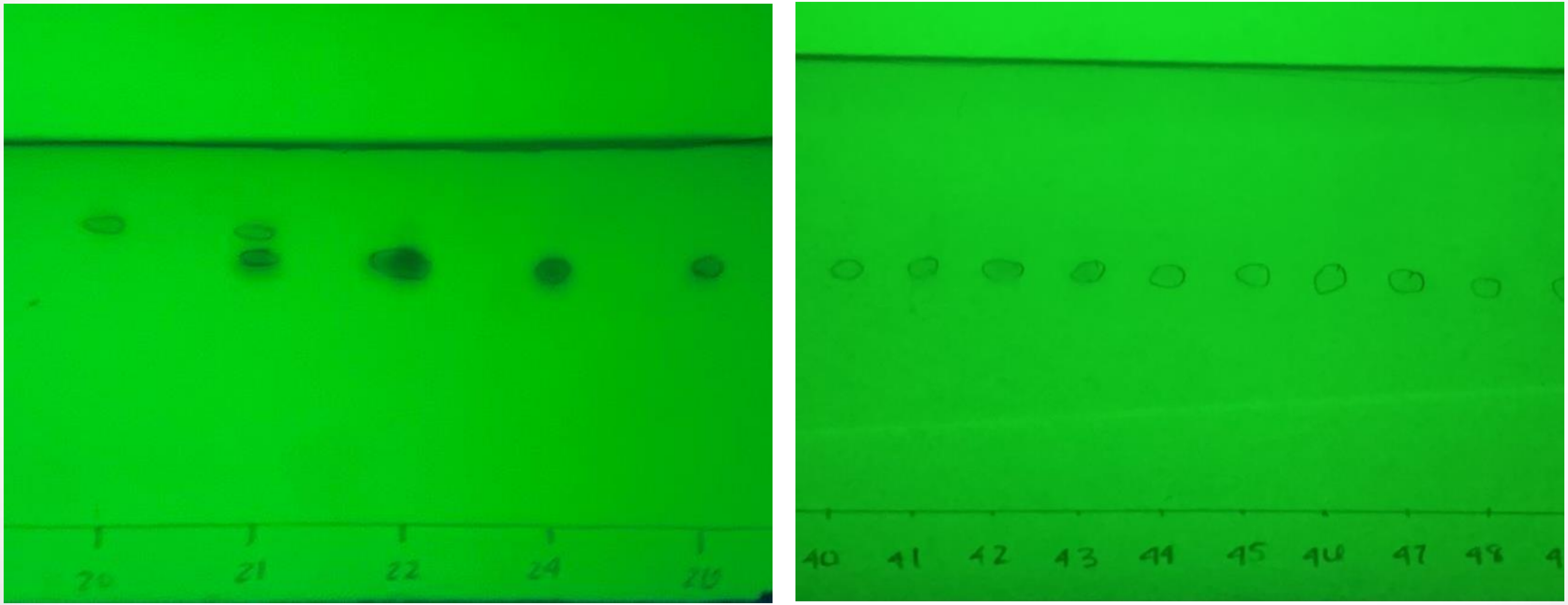


Figure 5. BGF Products (Photos by Jessica Bryant)

Characterization: IR

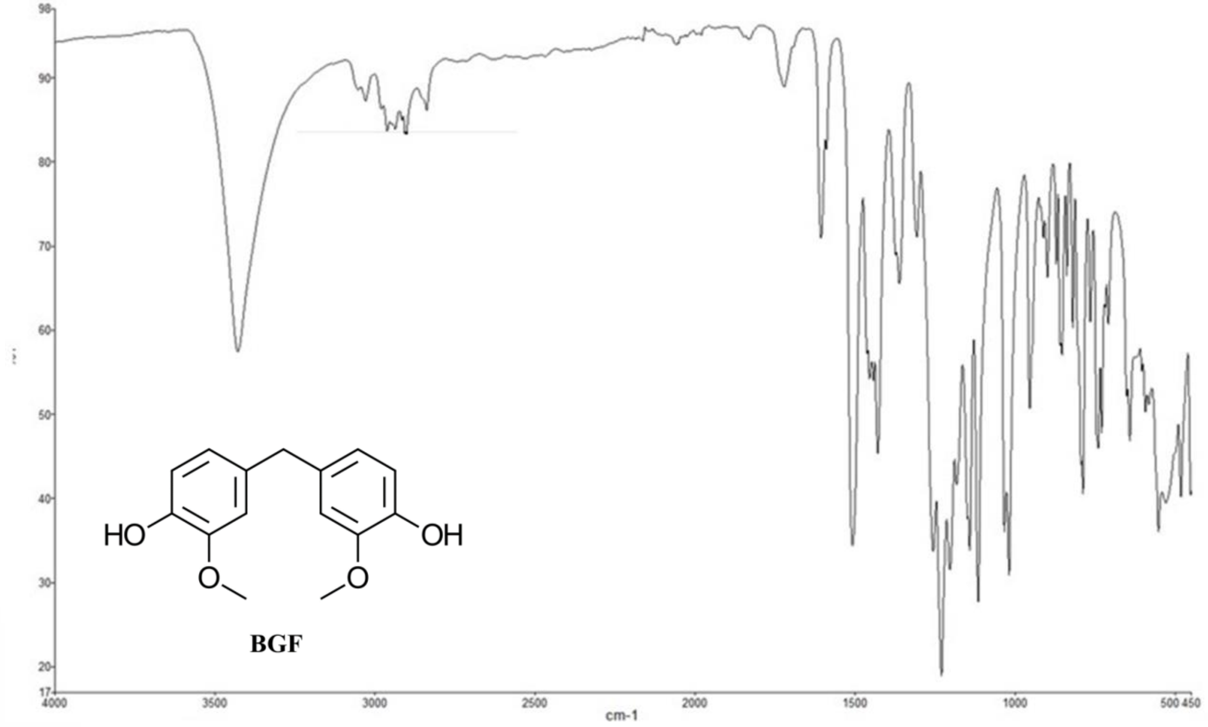


Figure 9. FTIR spectrum of BGF

BGF Preliminary Results

- Control syntheses of BGF completed using microwave reactor
- Higher purity obtained compared with conventional heating methods

Reaction	Solvent	Catalyst	Conditions
1	Water	Amberlyst	135 °C/ 6 min
2	Water	Amberlyst	170 °C/ 6 min
3	Ethanol	H_2SO_4	170 °C/ 6 min
4	Water	Amberlyst	170 °C/ 12 min

Table 2. Microwave Control BGF Synthesis (Created by Jessica Bryant)

TLC Comparisons

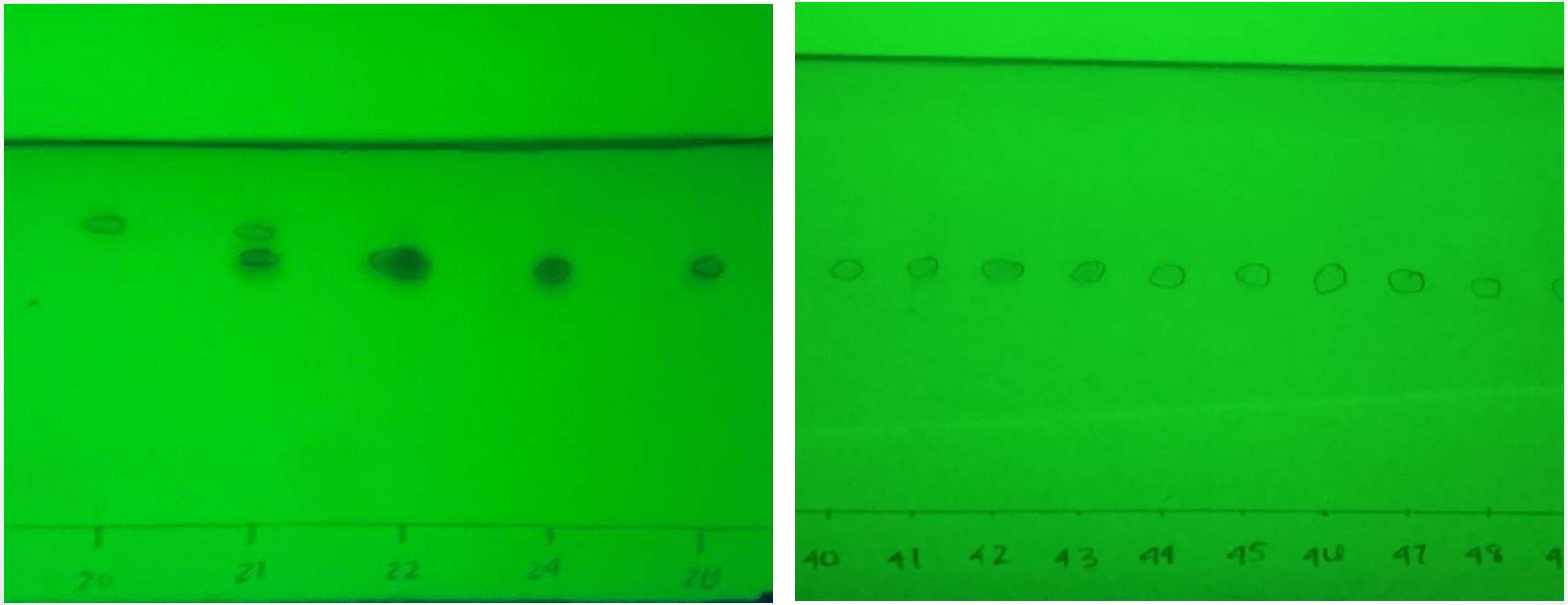


Figure 5. BGF Products (Photos by Jessica Bryant)

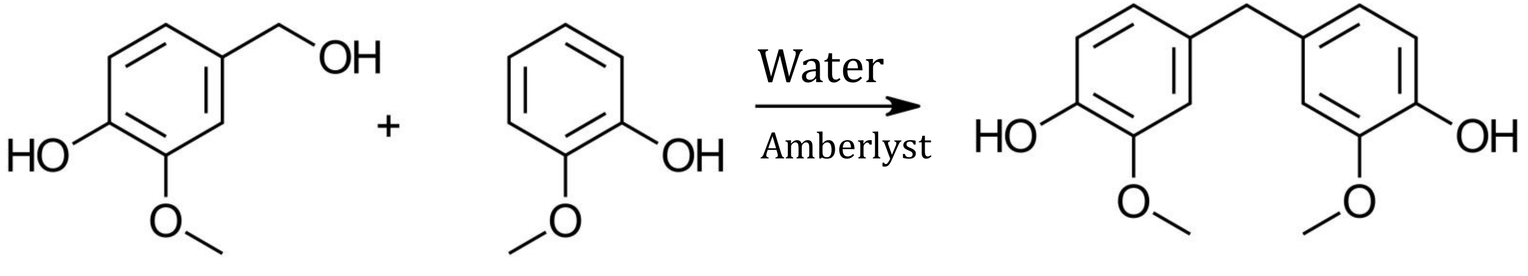
Research Objectives

- Optimize Microwave Synthesis Conditions
- Vary Polarity of Solvents and Determine Effect of Acid Sources
- Characterization and Analysis
- Optimize workup/ purification of BGF
- Peer-Review, Publication of Results

Created by Dr. Stephen Hobson

Optimize Microwave Synthesis Conditions

- Use Water solvent and Amberlyst Resin as acidic catalyst
- Establish optimal microwave assisted synthesis conditions in terms of:
  - Time
  - Temperature
  - Heating rate



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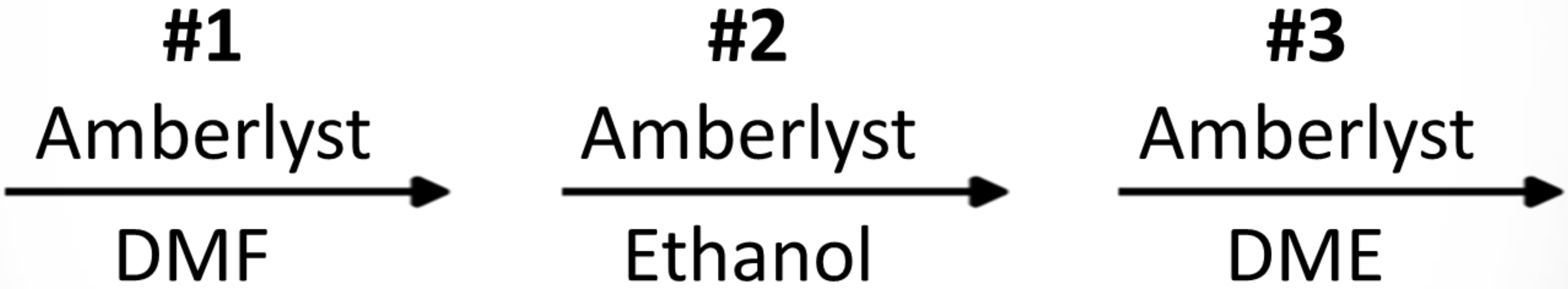
Vary Polarity of Solvents

- Vary solvent polarity with optimal conditions for water



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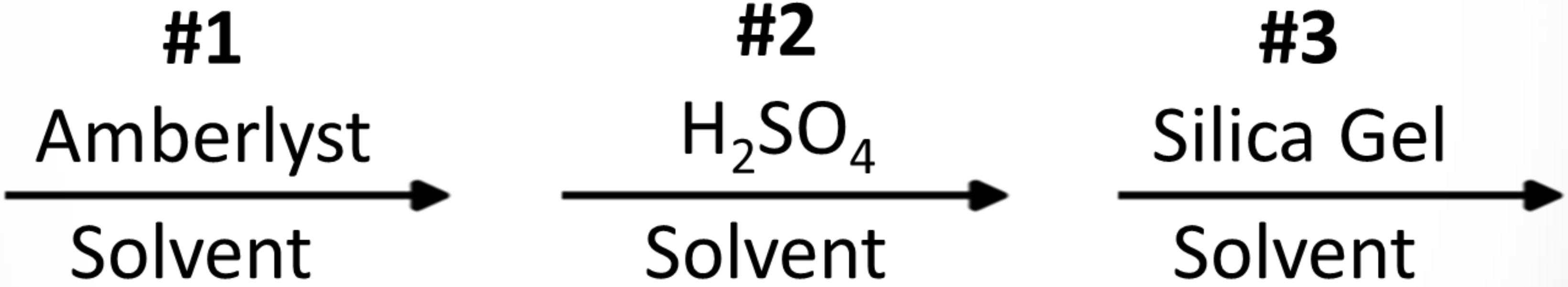
Determine Effect of Acid Sources

- Use optimal solvent and reaction conditions to evaluate different acid catalysts



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Characterization: TLC

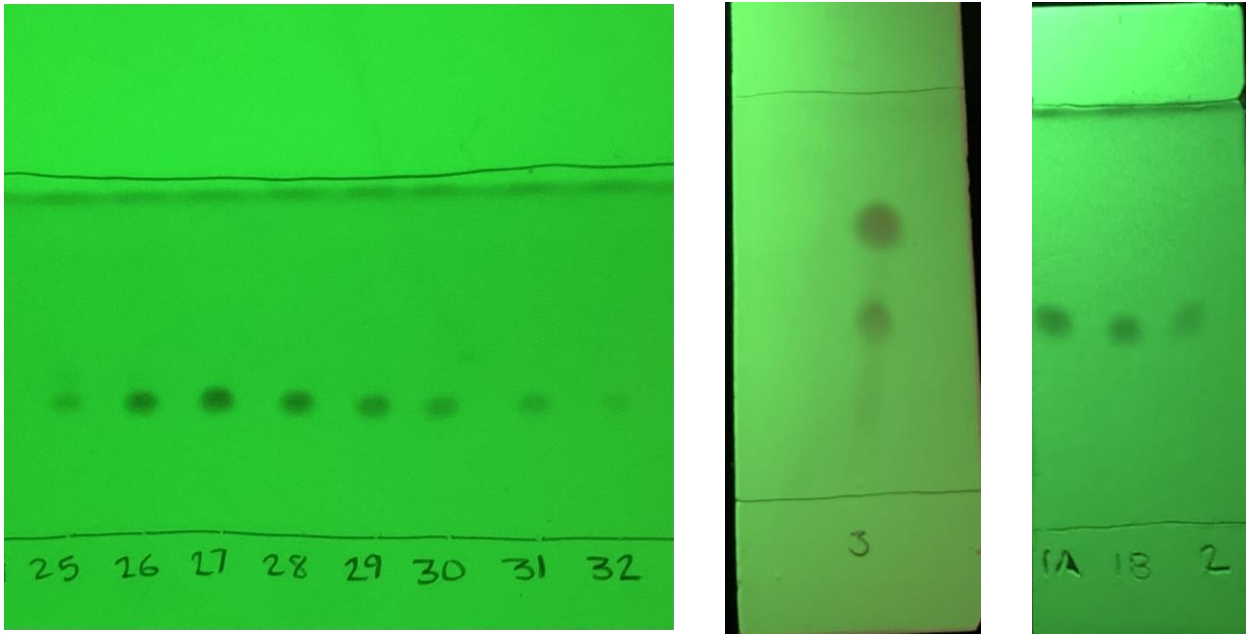

Figure 8. TLC Analysis of BGF (Photos by Jessica Bryant)

Characterization: IR

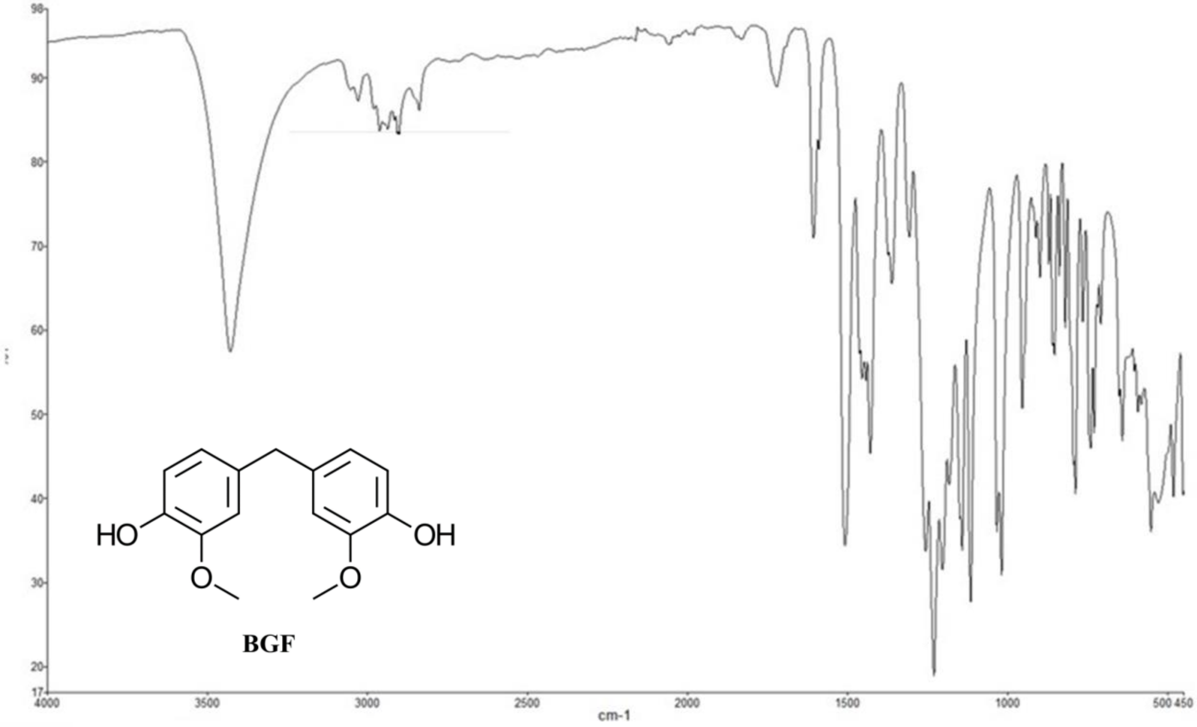

Figure 9. FTIR spectrum of BGF

Optimize BGF Workup/Purification

Figure 10. Column Chromatography
(Photo by Jessica Bryant)

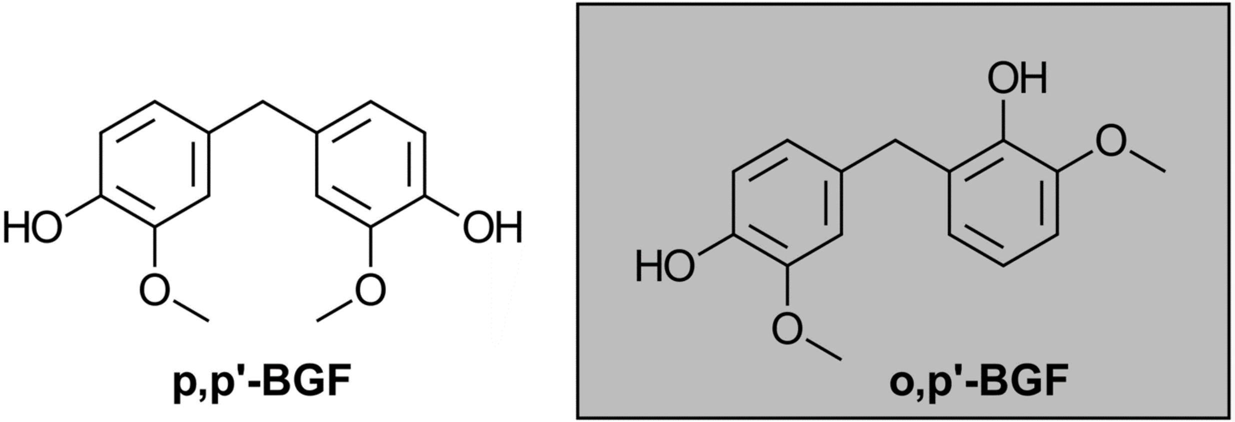

Figure 11. BGF Isomers (Created by Dr. Stephen Hobson)

Acknowledgements

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Questions?