

Process manufacturing of functional food particles from lignin-rich feed

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Introduction

After cellulose, lignin is the second most abundant natural polymer found in vascular plants. As such, it finds its way into many waste streams and by-products of the food industry. Two such examples are spent coffee (20-27% lignin¹) and brewers' spent grain (BSG) with its main component being barley (28% lignin^{2,3}). In 2015, European beer production totalled at 51.7 billion litres with an approximation of 20 kg BSG per 100 L of brewed beer. This results in ≈25.85 million tpa BSG in Europe alone⁴.

Lignin represents a class of amorphous complex organic molecules providing structural support to plant cell walls. It has been hypothesised to be the cause of the Pickering emulsifying ability observed for small cocoa particles. The particles appeared to be hydrophilic since they readily stabilised oil-in-water emulsions^{5,6}. Demonstrated on consumer coffee waste, also rich in lignin, hydrothermal treatment renders the hydrophilic particles hydrophobic imparting water-in-oil emulsifying ability. Hydrothermal treatment relocates the hydrophobic lignin from the cell walls onto the surface of the particles where it appears in the form of small droplets. Building on these lab scale findings, the research hypothesis of this PhD project is that lignin-rich waste/by-products can be processed into functional food particles with emulsifying property at economically viable food ingredient process scale.

Overall aim of this project: Validation of thermal processing at pilot scale as a process manufacturing route for functional food particles from lignin-rich feed in comparison to microwave processing as alternative technology.

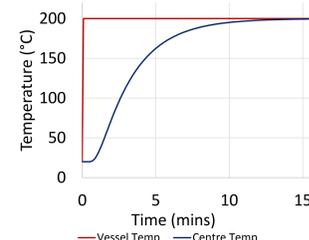
Specific objectives of year 1 (2016/17):

- Choice and characterization of feedstock
- Understanding material - process interactions
- Putting assays in place for characterization of processed material



Lager based brewers' spent grain

Thermal processing in aqueous suspension – dry heat



Theoretical heating profile – Process T of 200°C

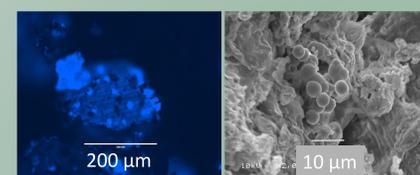
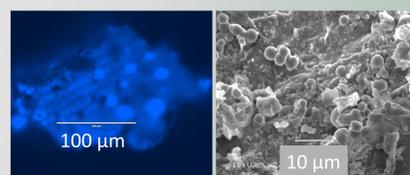
- Conduction heating model shows that the suspension in the geometric centre of the steel reactor reached temperature in 15.5 min
- ✓ Consideration of convection heating in the model reduces this time to just under 10 min

Relocation of lignin during hydrothermal processing at 200°C for

90 min

v

120 min

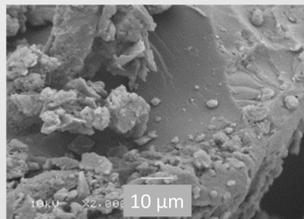


- Lignin successfully relocated
- Benefit of prolonged processing not obvious

Next steps

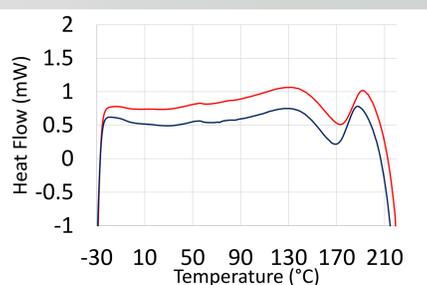
- Process mapping including temperature, time, pressure and pH as variable since dissociation of water at 150°C hypothesised to be critical to move lignin → can conditions be identified to run this process in a commercial steam retort?

Brewers' spent grain (BSG)



Surface structure

- Scanning electron micrograph of dried and milled brewers' spent grain (scale bar: 10 µm)
- Smooth particle surface structure

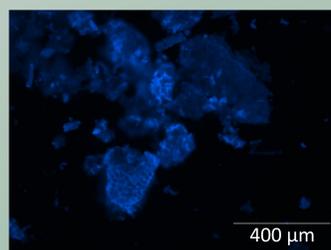


Thermal properties

- DSC thermogram of dried and milled BSG (heating rate 5°C/min)
- Softening and glass transition at 180°C
- "Relocation" processing requires T > 180°C

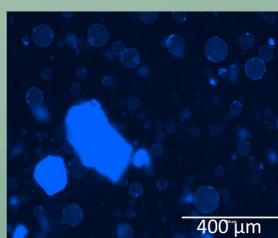
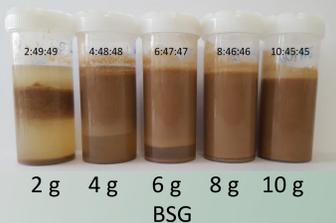
Localisation of lignin

- Lignin autofluoresces
- Fluorescence microscopy at excitation 357 nm⁷ (scale bar: 400 µm)
- Lignin in the cell wall structure clearly visible



Emulsifying properties

- 20% of oil emulsified into 80 g of water containing increasing amount of milled BSG particles
- Stable at 8 g of BSG with no coalescence observed for at least 6 weeks
- Fluorescence microscopy validates interfacial adsorption of the lignin-rich BSG particles

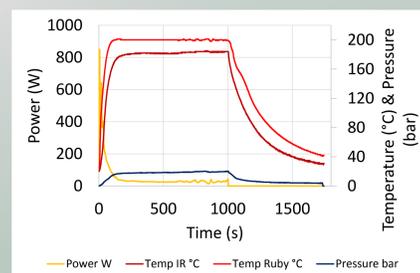


Microwave processing in aqueous suspension

Actual heating profile

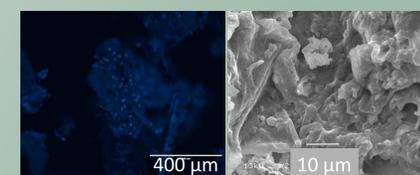
– Process temperature of 200°C

- Anton Paar Monowave 300 microwave synthesis reactor
- Holding time of 15 min



Micrographs

- Surface droplets not identifiable
- Lignin remains in cell wall structure
- Surface structure of BSG particle has changed though



Next Steps

- Mapping impact of process time on particle appearance
- Comparison of microstructures to those obtained in dry heat process
- Determination of dielectric properties of biomass to optimise process parameters (time and temperature)

Conclusions

- Brewers' spent grain represents another lignin-rich by-product which can be functionalised as a Pickering particle
- Softening transition was identified by thermal analysis
- Processing time impacts on visible amount of lignin relocation at temperature above softening transition though further experiments are required to validate that microwave heating has no fundamentally different impact on material properties compared to traditional heat processing
- Development of quantitative methods to characterise surface properties and composition of particles before and after treatment is required

References: ¹Ballesteros, L.F., J.A. Teixeira, and S.I. Mussatto, Food and Bioprocess Technology, 2014, 7(12): p. 3493-3503. ²Mussatto, S.I., G. Dragone, and I.C. Roberto, Journal of Cereal Science, 2006, 43(1): p. 1-14. ³Mussatto, S.I., G. Dragone, and I.C. Roberto, Industrial Crops and Products, 2007, 25(2): p. 231-237. ⁴Kirin. Kirin Beer University Report - Global Beer Production by Country in 2015, 2016. ⁵Gould, J., J. Vieira, B. Wolf, Food & Function 2013, 4, 1369-1375. ⁶Gould, J., G. Garcia-Garcia, B. Wolf, Materials 2016, 9. ⁷Vavrick H; Gryc, V.R., M, TRACE Conference. 2008, Association of Tree-Ring Research. p. 149-153. ⁸Fan, S., et al., Current Organic Chemistry, 2016, 20: p. 2799-2809.



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