



THE UNIVERSITY  
OF BRITISH COLUMBIA

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# Dried and redispersible oat oil emulsion powders stabilized by methyl and nanocellulose



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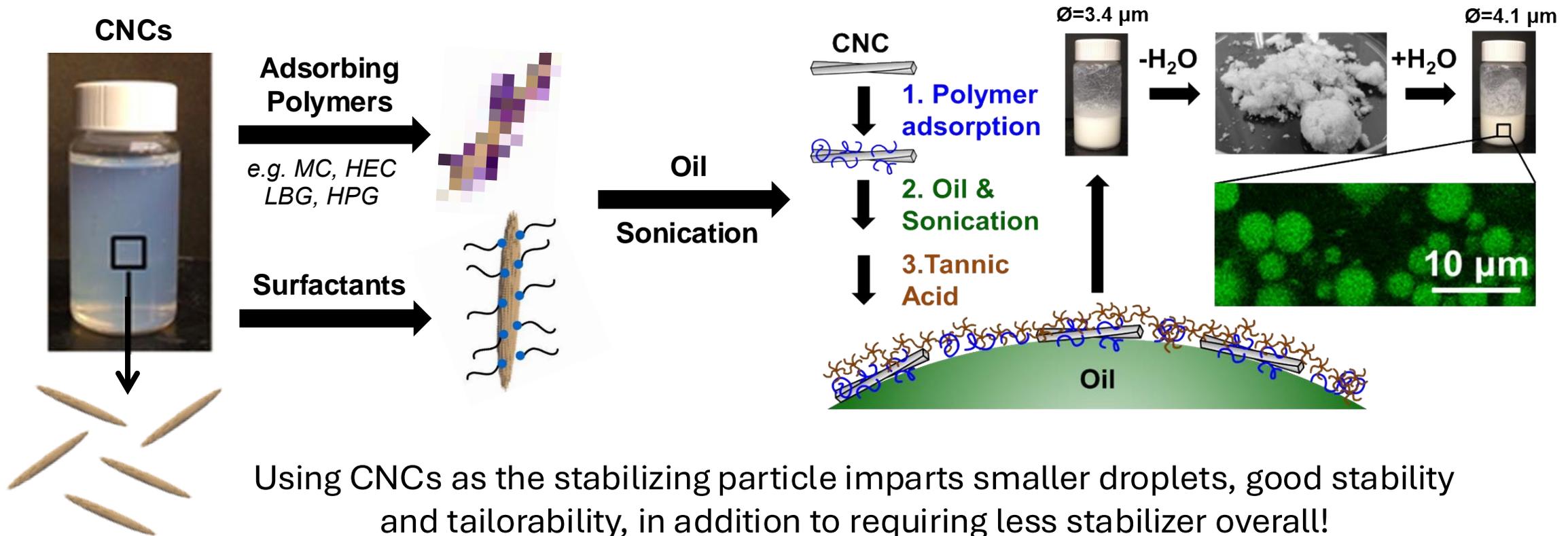
# Our Objectives



- ✓ Produce oat oil-in-water emulsions using well-characterized cellulose-based co-stabilizers
- ✓ Optimize the spray drying process to prepare dried oat oil powders
- ✓ Demonstrate the re-dispersibility of dried oat oil powder back into oil-in-water emulsions
- ✓ Inspire new application avenues for oat oil requiring either wet or dry emulsion formulations
- ❑ Make CNCs from oat hulls (*in progress*)

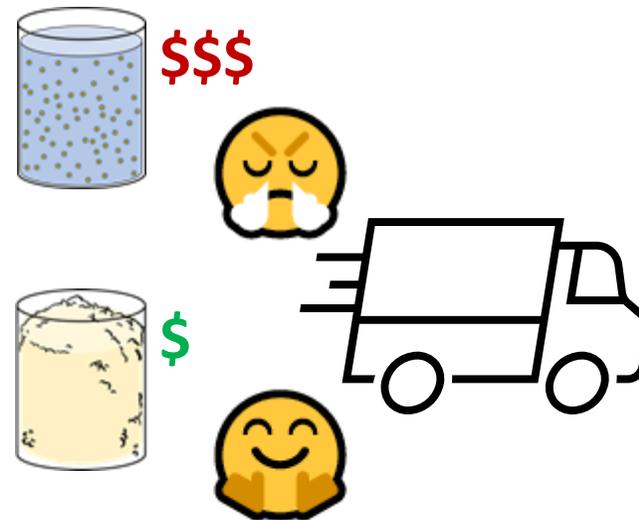
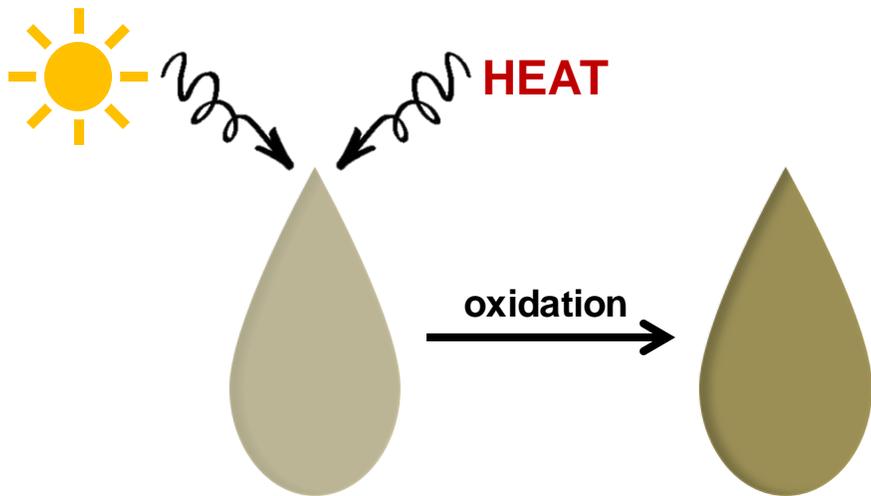
# A Strategy to Protect Oils is Pickering Emulsion Encapsulation

Cellulose nanocrystals (CNCs) are amphiphilic and can stabilize oil/water interfaces:



# The Motivation for Encapsulating Oat Oil with CNCs

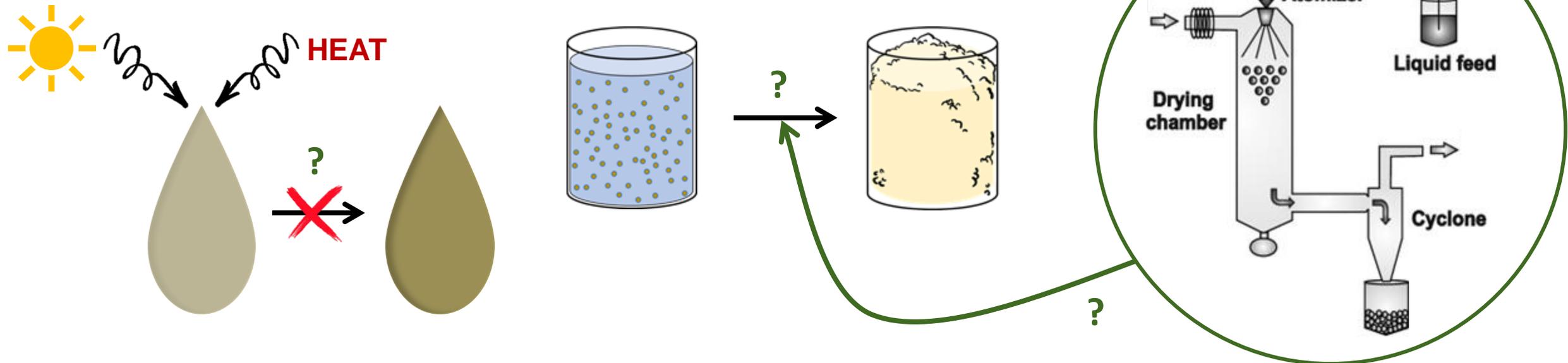
1. The oil is protected against oxidation (i.e., takes longer to become rancid)
2. It becomes possible to produce dry oil powders



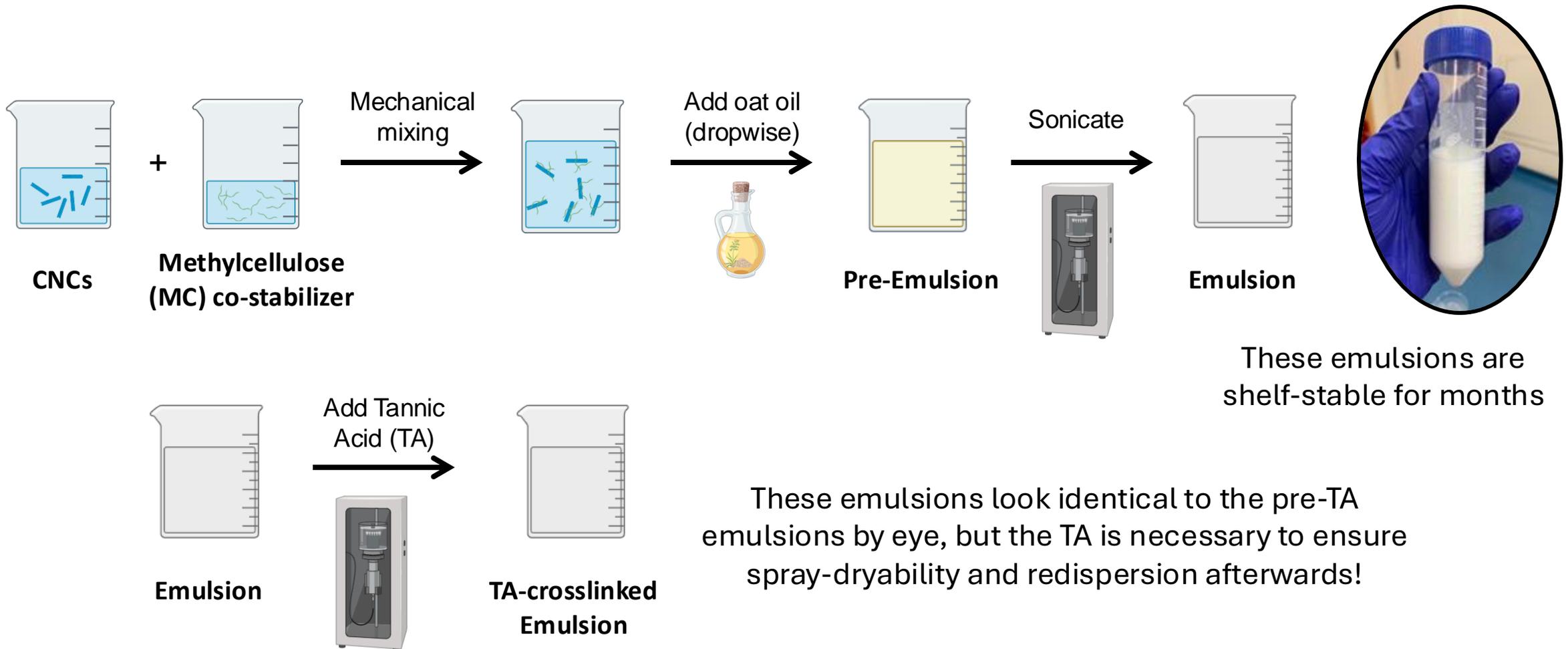
The oil powders can be used dry or redispersed in liquid media!

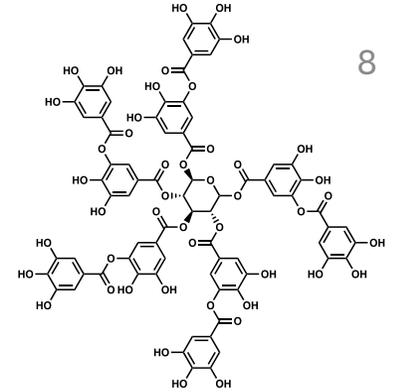
# Revisiting Our Research Questions Around Oat Oil Emulsions

1. Can we make dry oil powders by encapsulating oat oil with CNCs?
2. Can we use these emulsions to probe variable interactions during SD?
3. Can CNCs be used as Pickering stabilizers to protect the oat oil against oxidation over time?



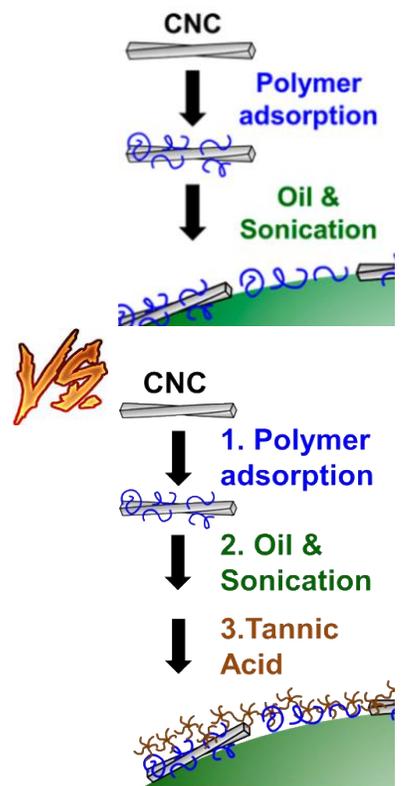
# The Recipe for Preparing CNC-Stabilized Oat Oil Emulsions



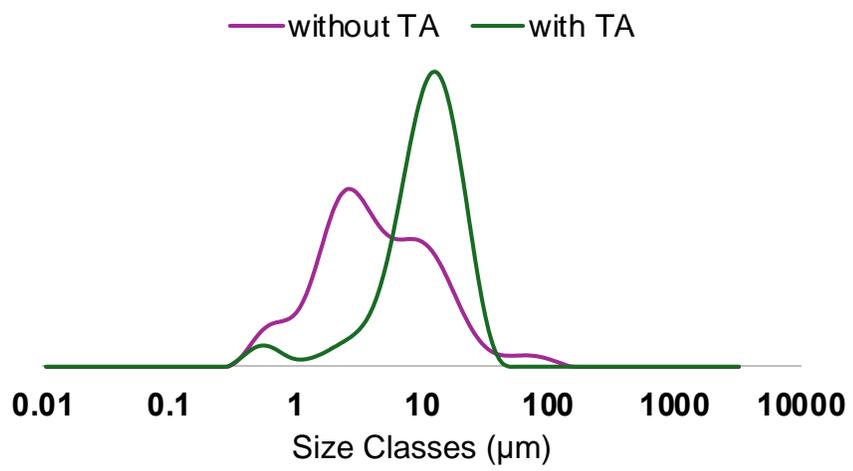


# The Effect of Adding the Tannic Acid (TA) Crosslinker

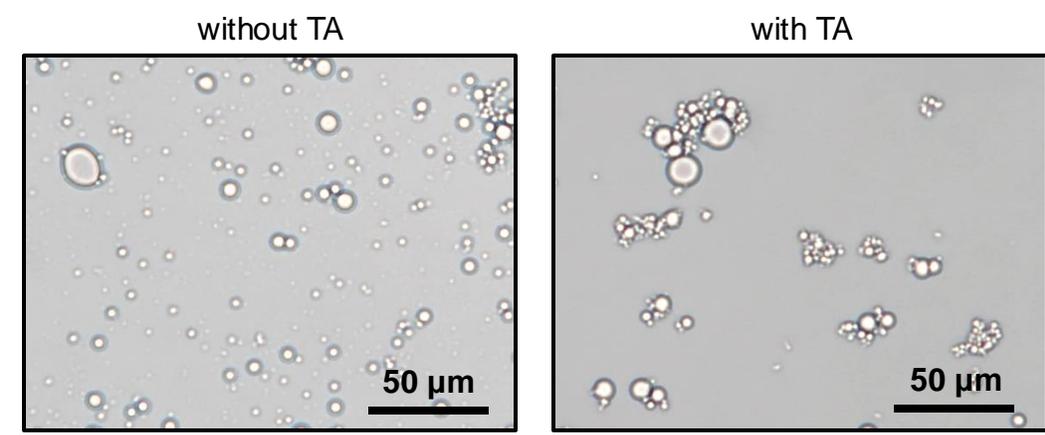
TA strengthens the CNC-MC shell around the oil droplets to enhance stability during drying, prevent coalescence after drying, and allow for redispersion.



D[3,2] from MasterSizer:

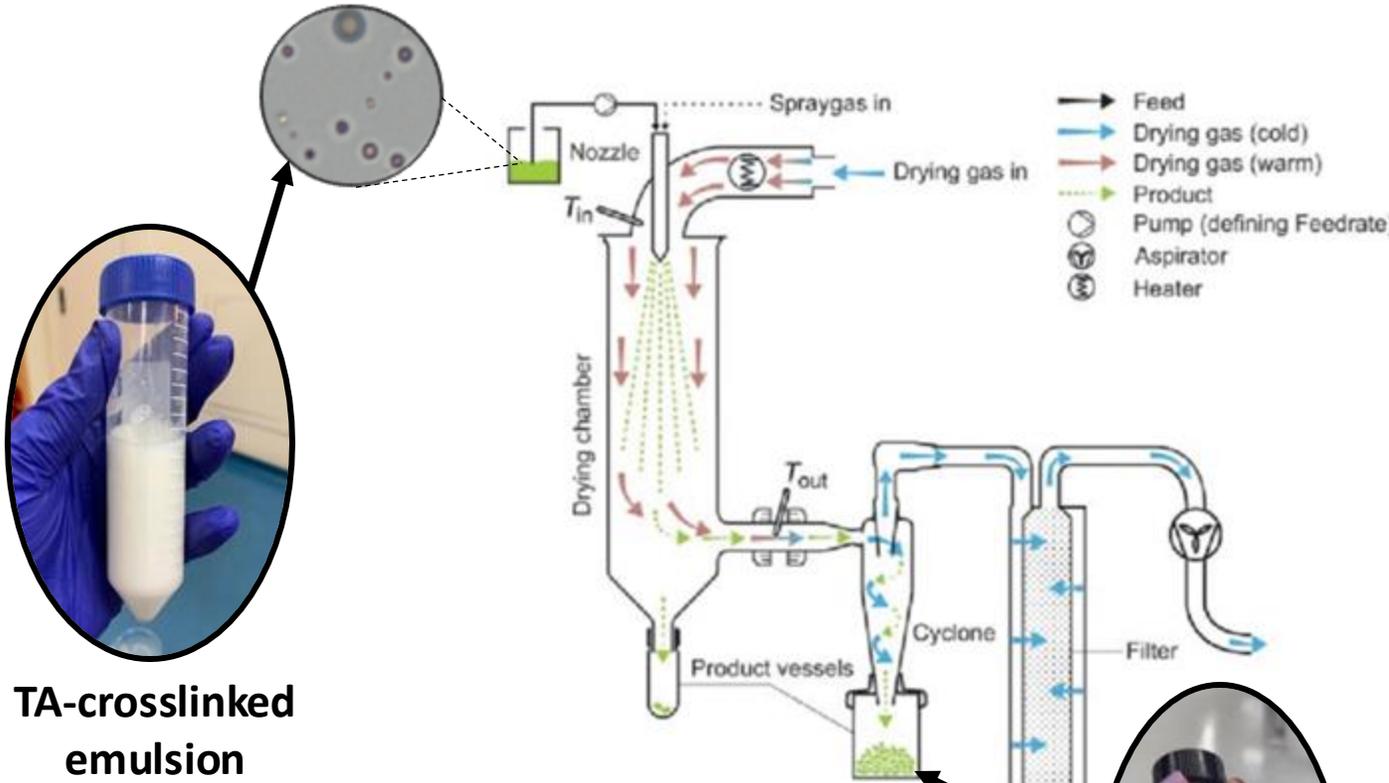


Polarized Light Microscopy:



Average droplet size goes up but dispersity goes down. Under the microscope, we see this is the result of droplet aggregation.

# Spray Drying Emulsions and Optimizing for Yield



TA-crosslinked emulsion

## Advantages

- Fast and continuous
- Less energy intensive than FD
- Less expensive for scale up

## Challenges

- Greater exposure to external stresses
- Greater loss of yield
- Dry product contains less oil

!! Many variables affect the output of a SD process.



Dry oil powder



Redispersed oil powder

# Design of Experiments (DOE) for Yield of Oil Powders from SD

DOE is a systematic, efficient method enabling scientists to study **the relationship between** multiple **input variables** (aka factors) and key **output variables** (aka responses).

When should we use DOE?

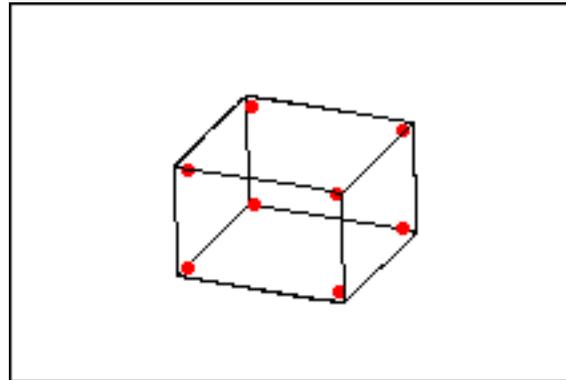
1. To determine whether a factor, or a collection of factors, influences the response.
2. To determine whether factors interact in their effect on the response.
3. To model the behavior of the response as a function of the factors.
4. To optimize the response.

# Design of Experiments for Yield of Oil Powders from SD

**Half factorial design** of DOE meant we selected **5 input variables** and did **19 spray drying trials**.

## Input parameters:

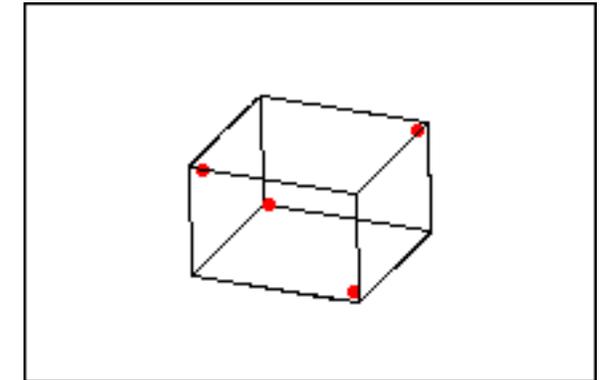
1. Aspirator rate
2. Inlet temperature
3. Feed rate
4. Dilution
5. CNC concentration



**Full factorial design**

*The number of runs necessary for a 2-level full factorial design is  $2^k$  where  $k$  is the number of factors.*

This would have meant 35 runs ☹️



**Half factorial design**

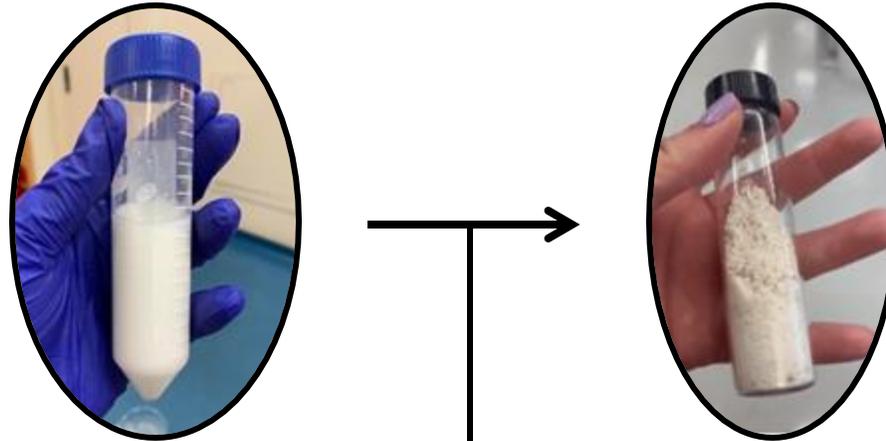
*Selective statistics limits the number of runs necessary to determine input interactions.*

So, we only need 19 runs!! 😊

## Output parameters:

1. **Yield (Y)**
2. Emulsion droplet size
3. Aggregate “powder” size

# Design of Experiments for Yield of Oil Powders from SD



Input Variable	Low Point (-)	Mid Point Recipe	High Point (+)
Feed rate ( $x_3$ )	4 %	8 %	12 %
Aspirator rate ( $x_1$ )	76 %	88 %	100 %
Dilution ( $x_4$ )	2 x	4 x	8 x
Inlet temperature ( $x_2$ )	130 °C	145 °C	160 °C
CNC concentration ( $x_5$ )	0.0825 wt%	0.165 wt%	0.33 wt%

... 19 experiments later

# Results from Design of Experiments for Yield

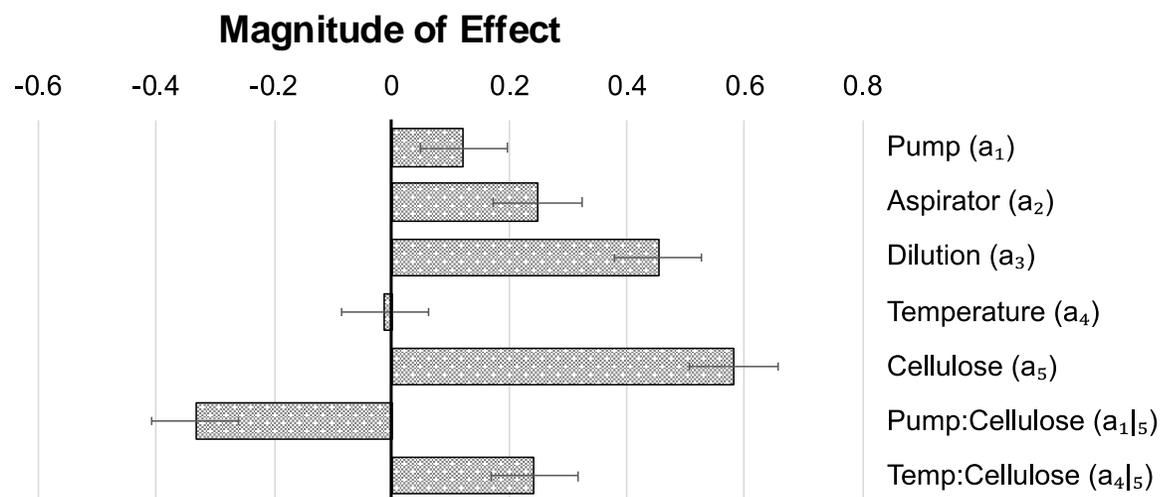
Linear Regression Analysis:

$$Y = a_0 + a_1x_1 + a_2x_2 + a_3x_3 + a_4x_4 + a_5x_5 + a_{1|2}x_1x_2 + a_{1|3}x_1x_3 + a_{1|4}x_1x_4 + a_{1|5}x_1x_5 \\ + a_{2|3}x_2x_3 + a_{2|4}x_2x_4 + a_{2|5}x_2x_5 + a_{3|4}x_3x_4 + a_{3|5}x_3x_5 + a_{4|5}x_4x_5$$

Linear Regression Analysis (simplified):

$$Y = a_0 + a_1x_1 + a_2x_2 + a_3x_3 + a_5x_5 + a_{1|4}x_1x_4 + a_{1|5}x_1x_5$$

Coefficients for parameters in our model for yield:



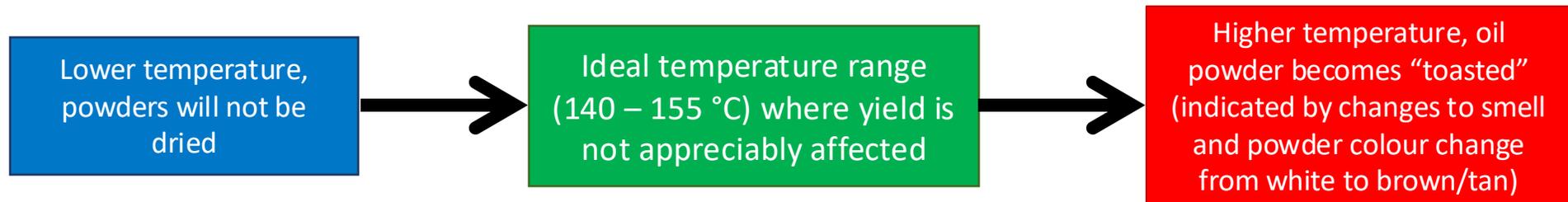
n.b., positive values indicate an increase in the input parameter results in a increase of the output property. Negative values indicate the opposite.

Cellulose content and dilution factor have the greatest effect on yield of oil powder from spray drying!

# Our Design of Experiments Results Allow Us to Suggest:

1. Work using a high CNC content (0.33 wt%)
2. Dilute the emulsion feed (8× worked best)
3. Increase the aspirator pressure to 100% (ca. 100 mbar)

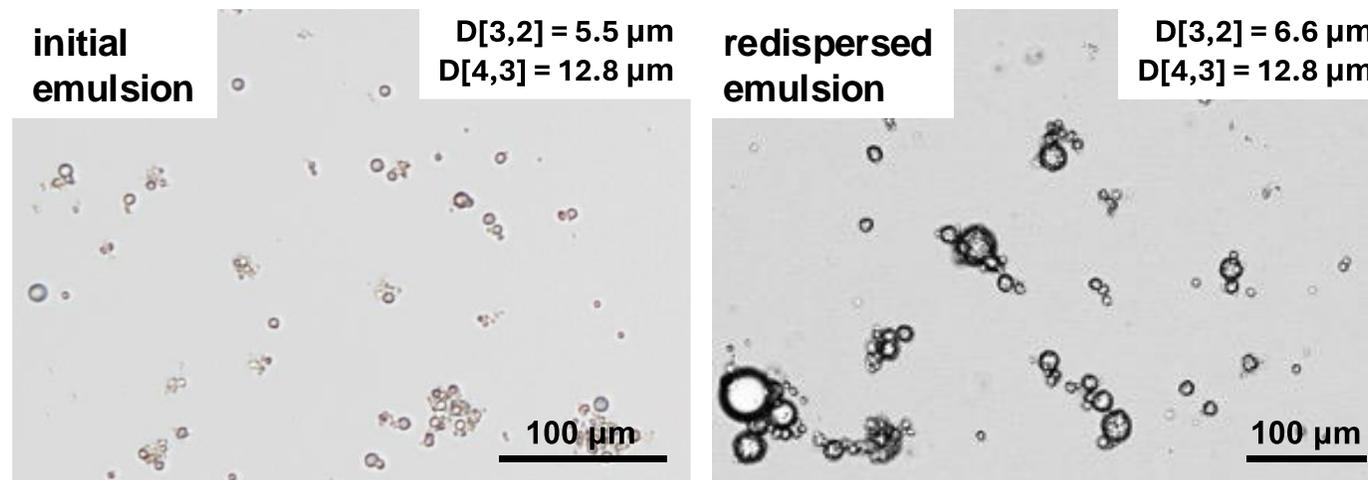
Note that it is likely temperature is a binary parameter:



While the feed rate was not found to be highly significant using our model, it does affect exposure of emulsion droplets to stresses (i.e., thermal stress, and capillary effects from tubing).

# Understanding Powder Redispersibility

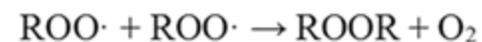
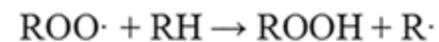
Individual droplets of the re-dispersion appear less defined and spherical than the original emulsion under the microscope.



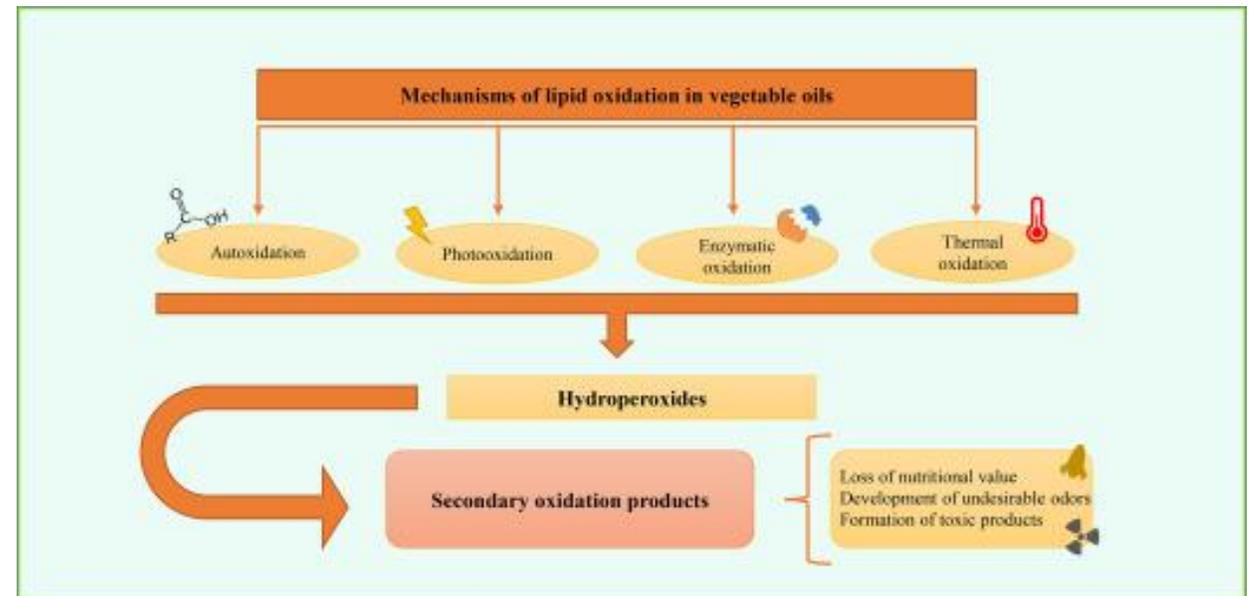
Both  $D[3,2]$  and  $D[4,3]$  remained close after re-dispersion!

# Side Objective: Do CNCs Protect the Oat Oil from Oxidation?

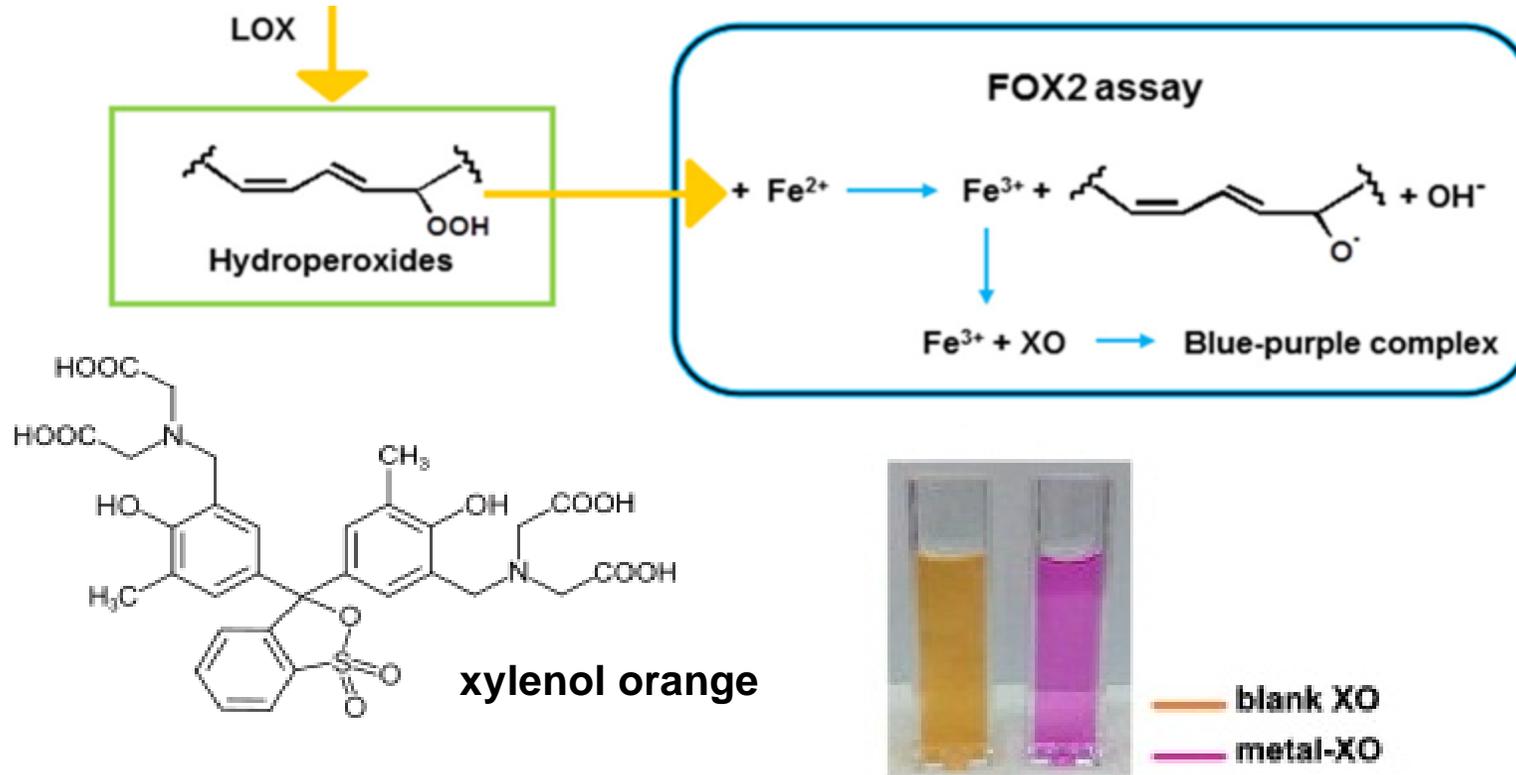
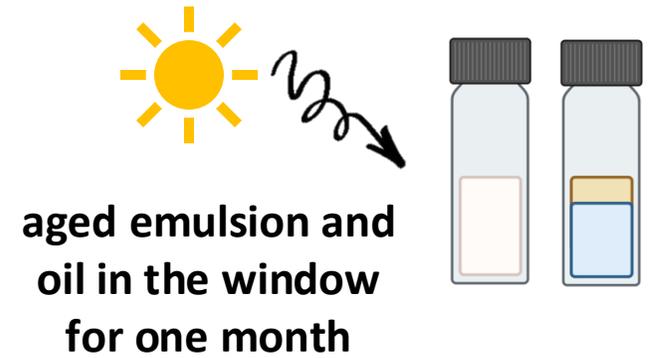
- **Lipid oxidation is a major cause of deterioration in oils**
- Hydroperoxides are generated as primary oxidation products. Then, these break down into volatile and non-volatile secondary products that affect the oil's taste and characteristics



There are assays that will detect these peroxide oxidation products.

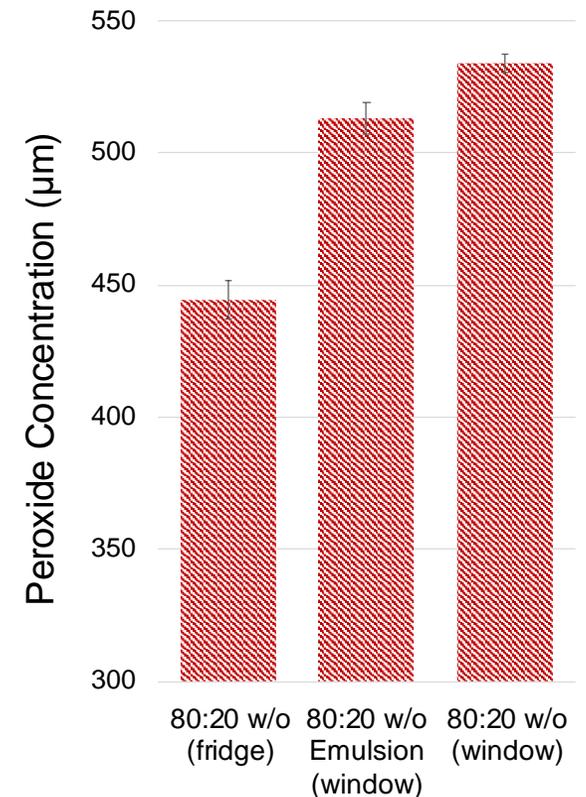


# The Peroxide Assay (i.e., the FOX2 assay)

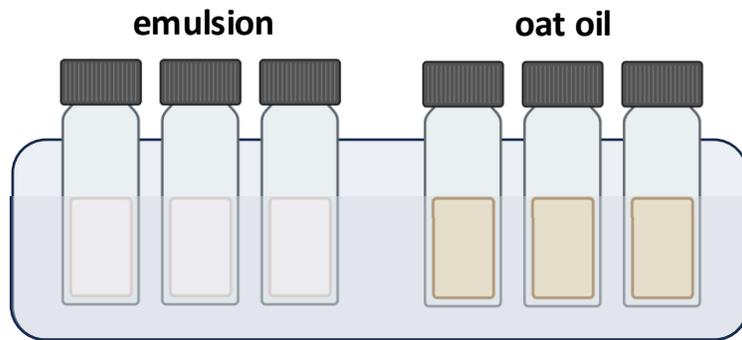


Compared to traditional titrations, this assay is way faster and easier!

Peroxide Concentration After Aging

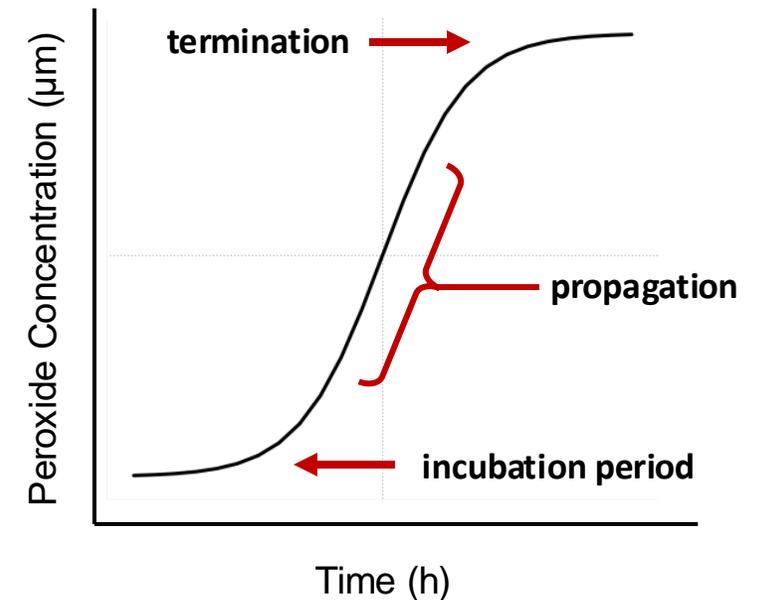


# A Series of Oxidation Kinetic Experiments Were Conducted



1. Heat at 60 °C for 72 hours
2. Remove aliquots every few hours
3. Measure peroxide content of oil and emulsion aliquots

We should have gotten a profile like this:



The experiment showed no difference in oxidation behaviour between the oat oil and the emulsion. In 72 h, neither peroxide concentration increased above the “incubation period.”



# Ongoing Objective: “All Oat” Emulsions 😊



oat hulls from C-Merak

Pretreatments?  
Hydrolysis conditions?



CNCs

Aspect ratio,  
Surface charge,  
Dispersion quality,  
etc...

We have tried:

- (1) Alkaline treatment with NaOH + Peroxide treatment (×2) ❌ (did not work)
- (2) Bleaching treatment using chlorite ❌ (did not work)
- (3) Alkaline treatment with NaOH + Bleaching treatment using chlorite ✓ (worked)

Followed by sulphuric acid hydrolysis

# Some Key Challenges and How We Overcame Them

**Problem 1:** Even though ground oat hulls looked uniform, they were not. This caused there to be a lot of “gunk” left at the end of hydrolysis that were not CNCs:



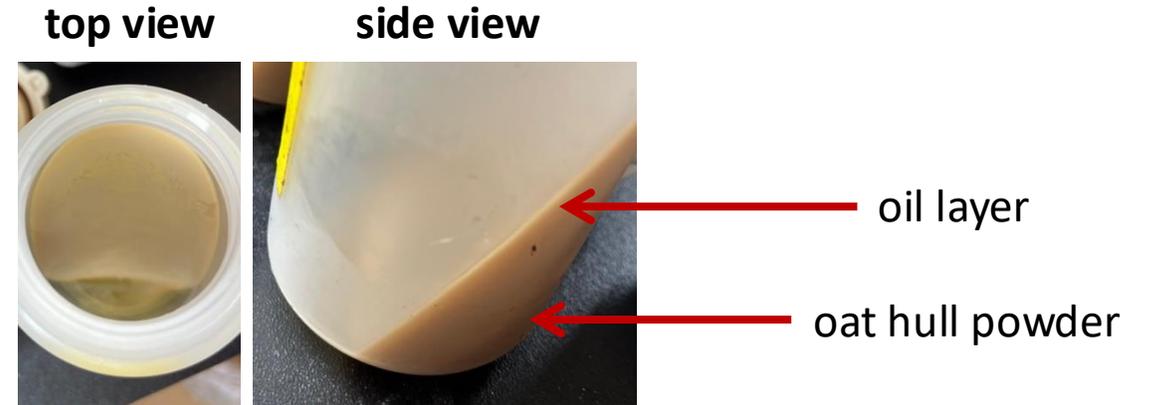
**Solution to Problem 1:** Sieving the oat hulls showed there is variety in powder size. We progressed using the fine powder produced using a 250  $\mu\text{m}$  sieve:

*(we started with 200 g of oat hull powder)*



# Some Key Challenges and How We Overcame Them

**Problem 2:** The fine powder was very oily. After alkali treatment, oil separated from the powder:



**Solution to Problem 2:** This oil layer was removed using acetone to leave a much less clumpy powder for subsequent bleaching step and hydrolysis:



# Characterization Experiments are In Progress

**We are working to characterize the CNCs produced from oat hulls.**

We are using:

1. Size using atomic force microscopy (AFM)
2. Surface charge using titration for sulphate half ester groups
3. Dispersion quality using dynamic light scattering (DLS)

# Summary

- CNCs can be used to stabilize the oat oil-water interface to make emulsions
- These emulsions are spray dryable to make redispersible oat-oil powders
- A design of experiments approach taught us that cellulose content and dilution factor had the greatest effect on yield of oil powder from spray drying (**highest yield achieved was ca. 80 %**)
- Oxidation kinetic studies were performed but did not have conclusive results.
- Using a combination of alkaline and bleaching treatment steps, we can isolate CNCs from oat hulls.
- To avoid complications, oat hull powders should be sieved prior to treatments and oil should be extracted using acetone.
- A manuscript focused on the design-of-experiments approach to spray drying emulsions is in progress!

# Thank You!



THE UNIVERSITY  
OF BRITISH COLUMBIA

Prof. Emily Cranston  
Keanna Yu  
Marcus Johns  
Golshan Matinfar

All group members past  
and present!



Darren Walkey



**NSERC**  
**CRSNG**

