Encapsulation of Oils for Use in Biodegradable Hygiene Products

PURDUE UNIVERSITY
Materials Engineering

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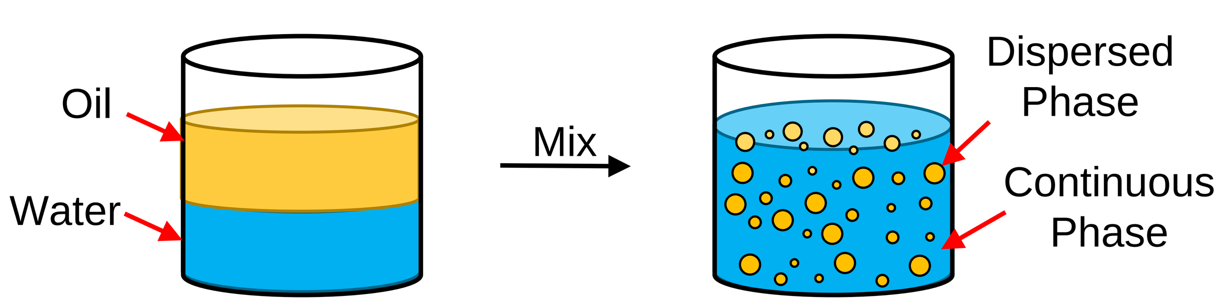
Procter & Gamble is looking to develop new microencapsulation methods and materials selection for use in products they currently have on the market. These new techniques will be utilized in biodegradable products that reduce packaging such as single use personal care products. Limiting the packaging will in turn cut transportation costs and emissions. The group was tasked with forming stable emulsions that successfully encapsulated oil droplets in the 10s to 100s micron scale. The emulsions were casted and allowed to dry so that the final product was a dry powder containing encapsulated oil drops that dissolve upon contact with water.

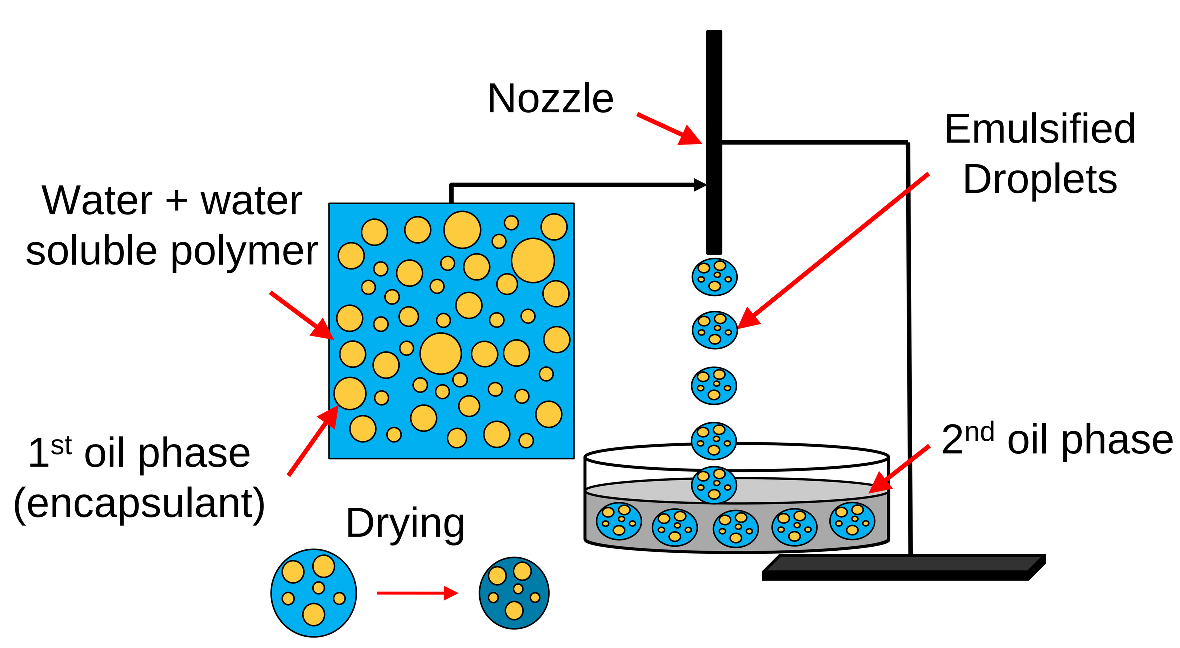
This work is sponsored by Procter & Gamble, Cincinnati, OH

Project Background

- Encapsulation is utilized in cosmetic, pharmaceutical, and agriculture industries to control the delivery of encapsulated material.
- Oil in Water Emulsions:
  - ➔Two insoluble liquids are mixed via agitation or ultrasonication to produce one phase dispersed within the other.



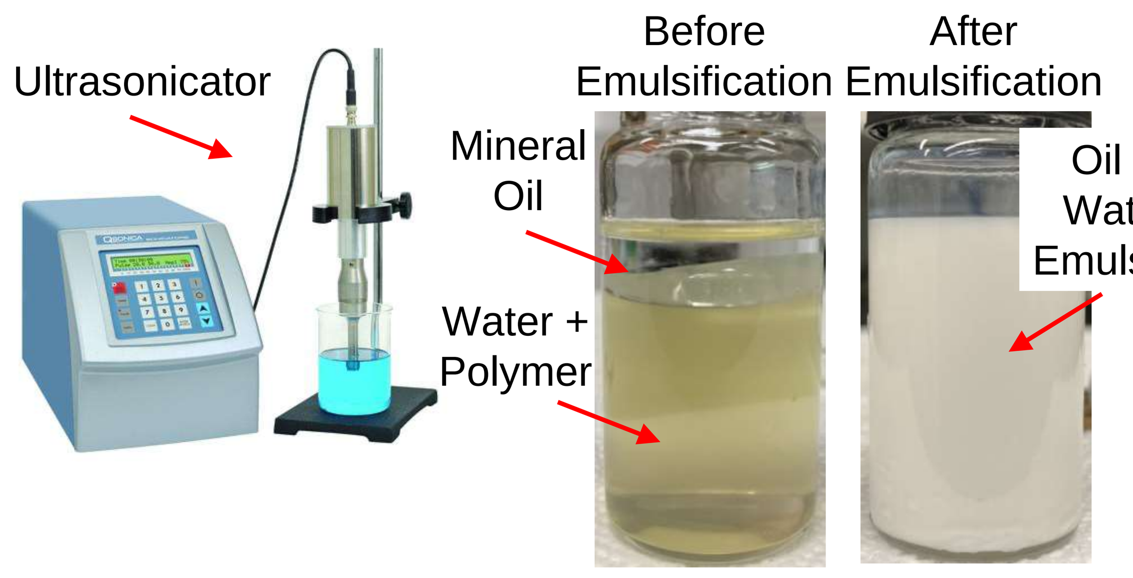
- Microencapsulation: a method to microscopically envelope one material within another.
  - ➔Matrix microencapsulation: Core material (oil) is distributed throughout a continuous phase of material (e.g., polymer):



Experimental Procedure

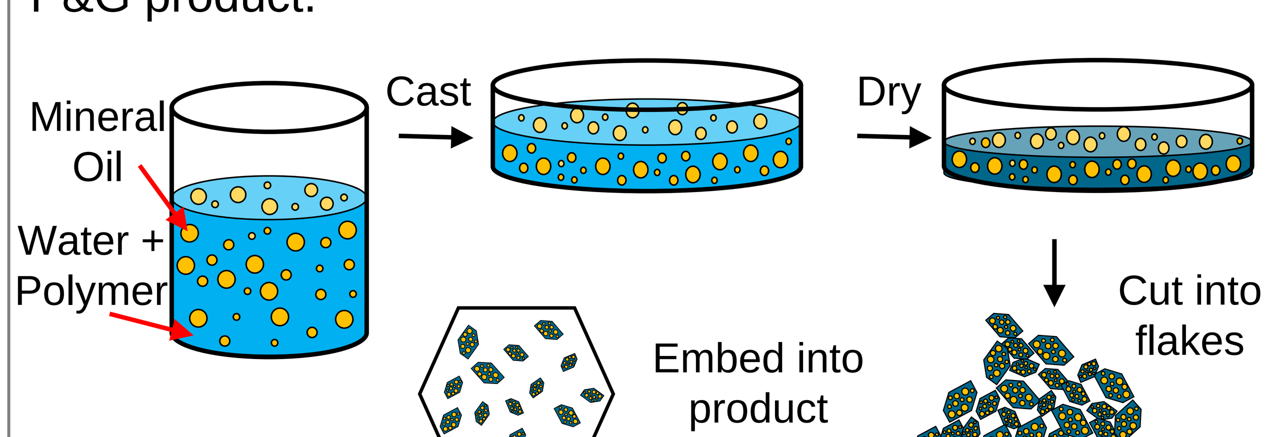
Emulsification

All emulsions were made by ultrasonication at 75W for 20 min. (1s on/1s off); The probe on the sonicator was positioned at the interface of oil and solution.



Casting

In order to eliminate the need for the second oil phase to stabilize individual droplets, the emulsions were casted into petri dishes and allowed to dry completely in ambient conditions. They were then flaked into a powder with a razor so that they could be readily embedded into the P&G product.

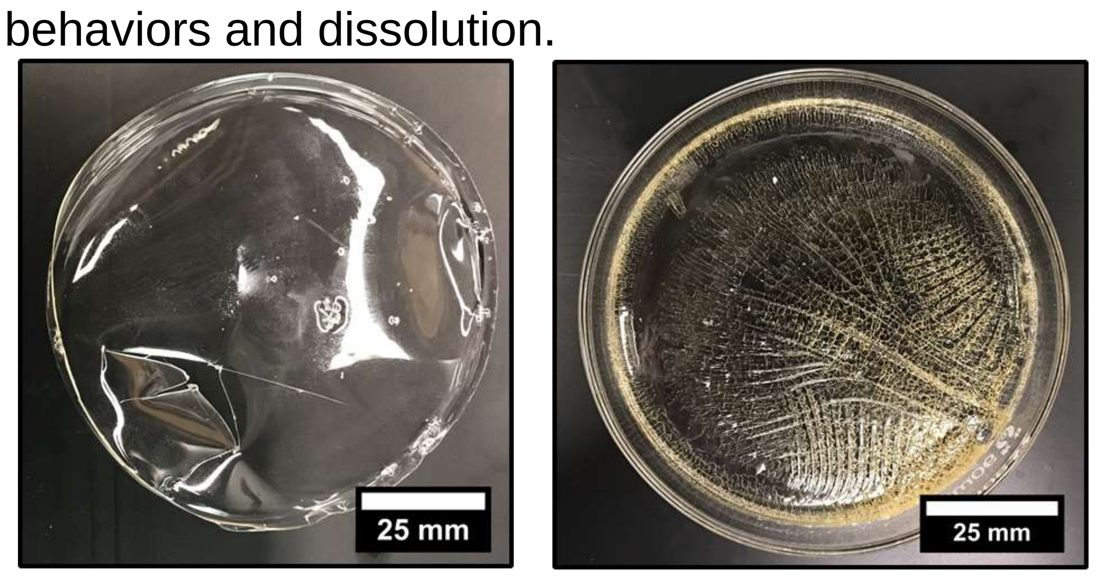


Redispersion

Flakes were added to palm with a few drops of water and sheared until dissolved. This process took around 5-8 seconds.

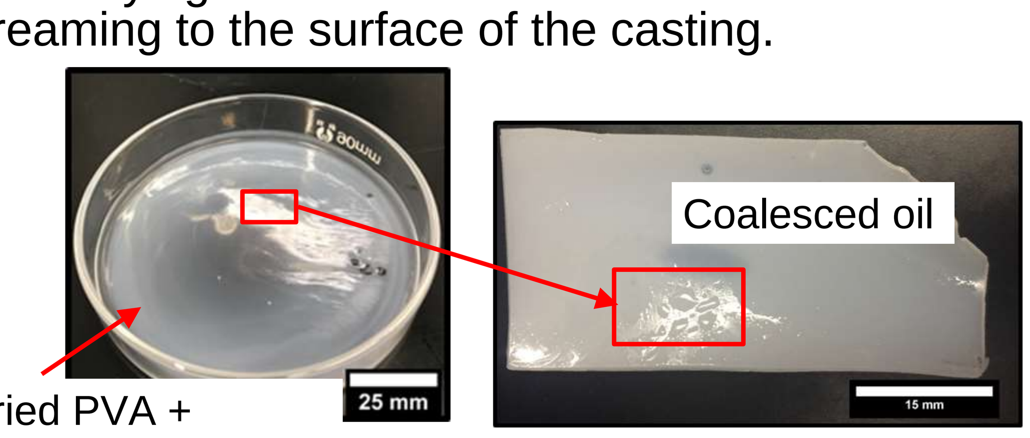
Results: Testing Matrix Material

Polyvinyl Alcohol (PVA), a water-soluble synthetic polymer, and Capsul®, a water-soluble food grade starch, were tested as matrix materials for encapsulation. Films of different concentrations of each material were casted to observe drying behaviors and dissolution.



PVA (left) formed a stiff film that was unable to dissolve in water at room temperature while Capsul® (right) dissolved readily.

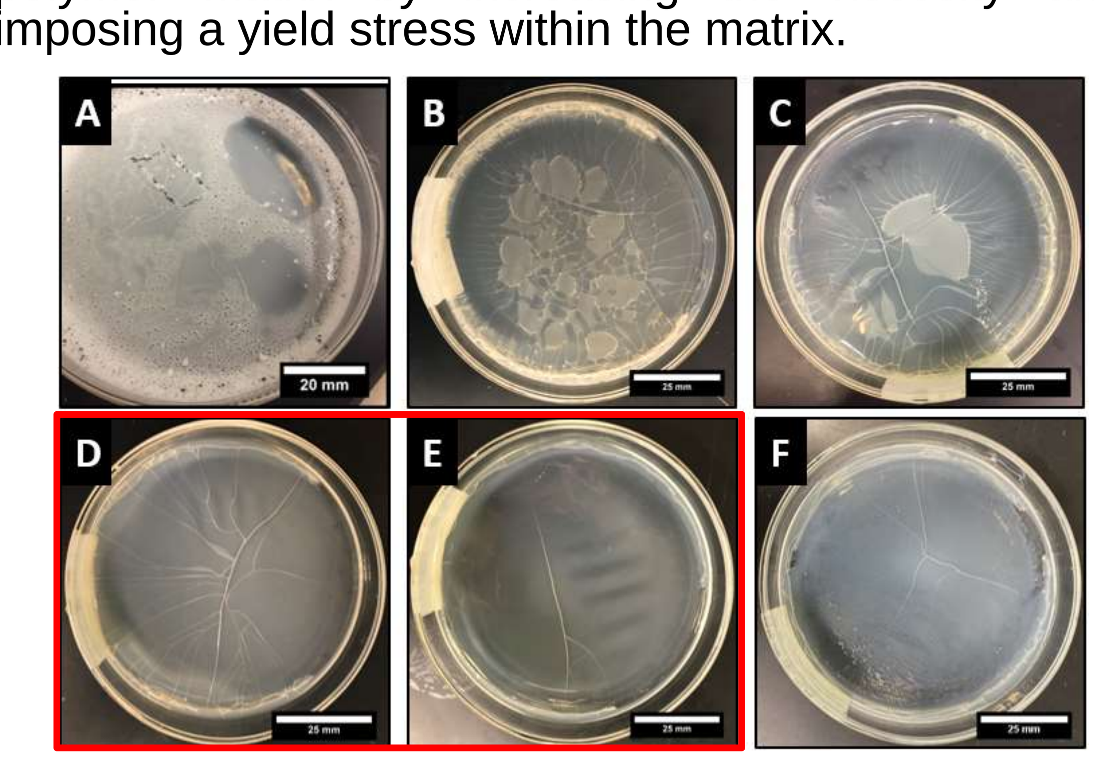
Emulsifying mineral oil in PVA led to issues with oil creaming to the surface of the casting.



Dried PVA + mineral oil emulsion

Results: Emulsion Casting

Carbopol® 941 (polyacrylic acid, PAA) was added as a thickening agent to keep oil suspended within the polymer matrix by increasing the viscosity and imposing a yield stress within the matrix.

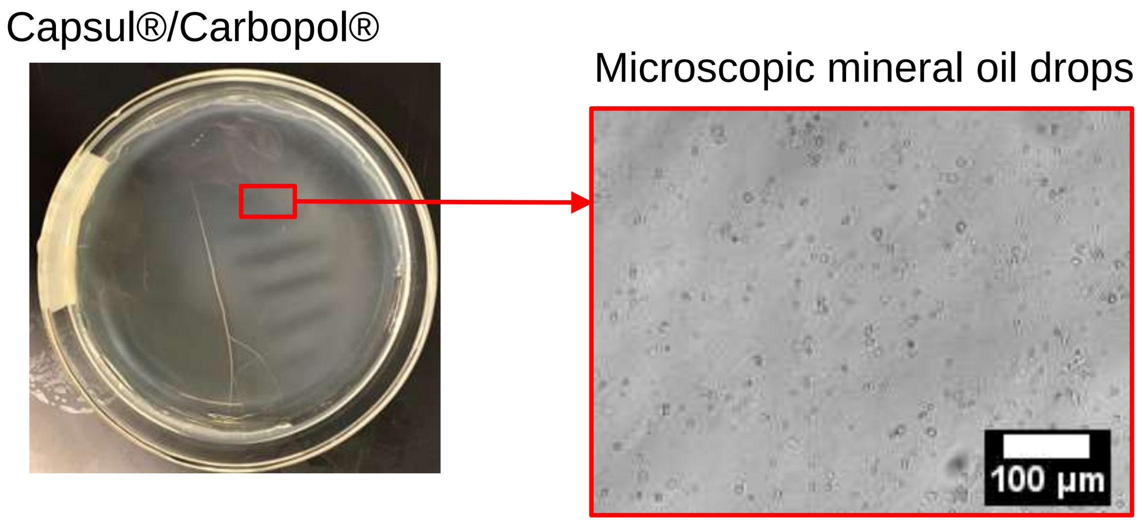


Capsul® and Carbopol® concentrations were varied but the ratio of mineral oil to aqueous solution was held constant at 1:4 oil to aqueous phase.

Emulsion	Capsul/Carbopol Solutions [%/%]
A	100/0
B	80/20
C	70/30
D	60/40
E	50/50
F	40/60

Ideal compositions

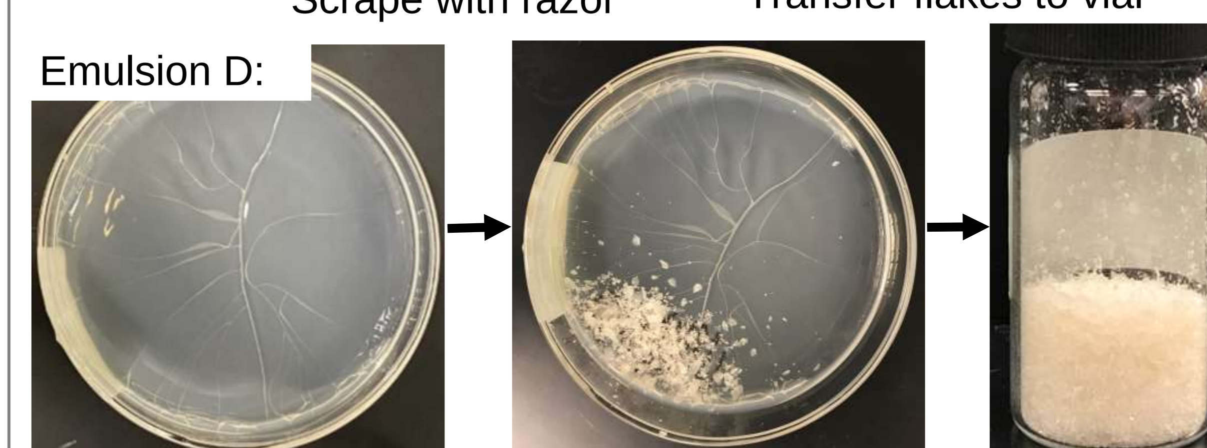
Emulsion E: 50/50 Capsul®/Carbopol®



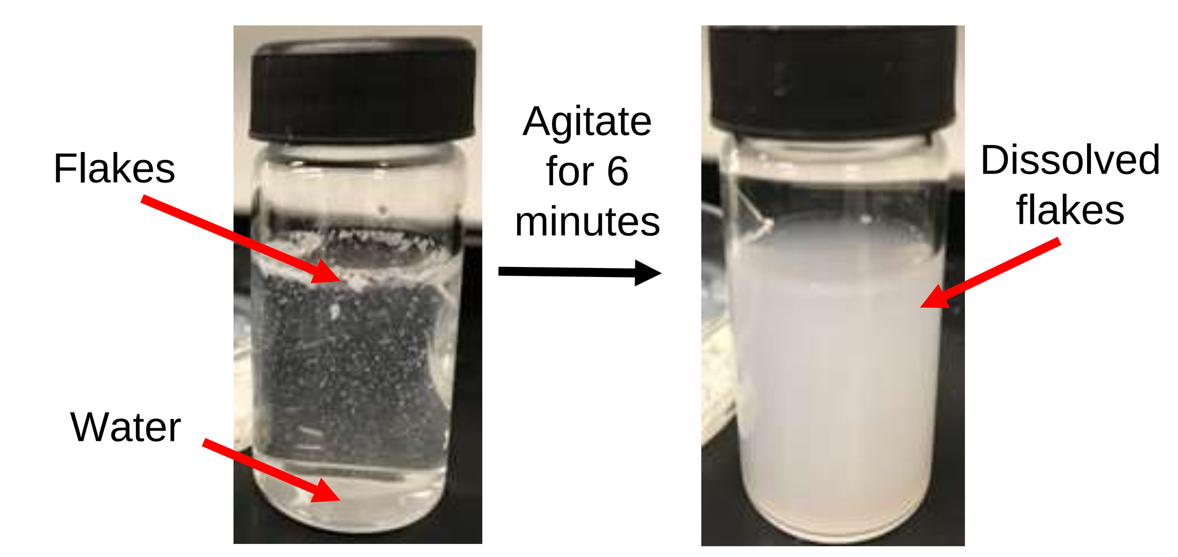
Optical microscopy showed successful encapsulation of mineral oil in a Capsul®-Carbopol® solution. Drops were evenly dispersed and had an average diameter of 1.14 µm. Casted film was continuous and dry to the touch, suggesting no oil loss.

Results: Flake Production

The dried films were scored with a razor blade, where the ensuing flakes were collected.



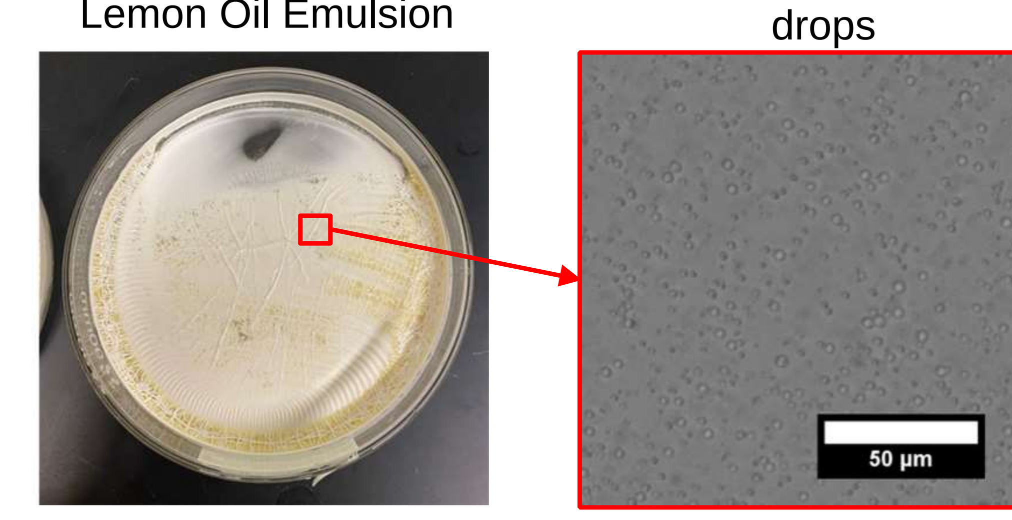
1 wt.% of flakes were added to room temperature DI water to observe dissolution of flakes:



1 wt.% of flakes was successfully dissolved in room temperature DI water. Flakes also dissolved readily with water and shear against the palms.

Results: Change in Core Material

Lemon oil was used to test if a volatile oil would yield the same results as the non-volatile mineral oil. No oil loss was observed, and optical microscopy showed that drops were evenly dispersed.



Lemon oil flakes were larger and tougher, but still dissolved readily in room temperature water.

Conclusions

- Mineral oil was successfully encapsulated within 50-60% Capsul® and 50-40%Carbopol® 941 via ultrasonication.
- Flakes were formed by scraping dry films casted from oil/polymer solution emulsions.
- Flakes were able to fully dissolve upon contact with water and shear.

Recommendations

The group has recommended P&G to use either the 50/50 or 60/40 ratios of the 25 wt.% Capsul®/1 wt.% Carbopol® polymer solutions and manufacture similarly to the flaking experiment to produce larger volumes of flakes. To comply with biodegradability and avoid using a crosslinked additive like Carbopol®, it is recommended to use a higher molecular weight starch that is compatible with Capsul®.