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Précipitation en émulsion pour le contrôle de morphologie de poudre à l'échelle industrielle

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I. Background

Emulsion precipitation can be implemented on a large scale through the use of pulsed columns, which disperse large quantities of aqueous phase in the form of droplets in an organic phase. Precipitation occurs after coalescence of two drops each containing two different reactants. If the feasibility of such a process for use in the field of nuclear waste treatment is proven, partial knowledge of the reaction mechanism is a limiting step. To study the process of precipitation the use of controlled systems, regarding mixing and feeding of reactants is necessary. In order to study the precipitation in emulsion under controlled conditions, three original operating modes are explored.

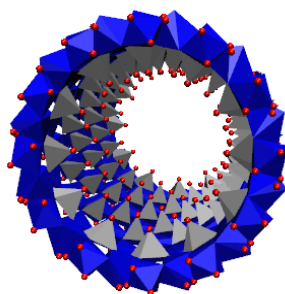


Figure 1 : Imogolite structure. Hydrogens are omitted, Oxygen as red dots, Silicon as grey tetrahedra and Aluminium as blue octahedra.

II. First approach

Our first approach is based on the use of a nano sized tubular clay named imogolite with following chemical composition: $(\text{OH})_3\text{Al}_2\text{O}_3\text{Si}(\text{OH})$. (Figure 1) It was first discovered in volcanic ash soils by Aomine et Yoshinaga in 1962.^[1] After modification of the outer tube surface it is able to stabilize droplets through the formation of a Pickering emulsion and was found to align itself in a perpendicular fashion on the droplet interface.^[2] Since the tubes are also filled with water, they fulfill the prerequisites to be tested for the control of the solute transport between two connected droplets. In order to investigate on the feasibility of this control mechanism, imogolite has been synthesized and modified according to the protocols found in literature. An in depth study of the surface modification by WAXS, IR and MAS NMR showed that instead of grafted tubes, the reported grafting reaction yields a composite material of a lamellar phase and ungrafted tubes. The material is not able to transport solutes, but is able to stabilize droplets against coalescence.^[3]

Since using imogolite as transport control turned out to be not feasible, two other pathways are investigated.

III. Current approaches

Two alternative processes are proposed to achieve controlled solute transport in the droplets. In the first one, droplets are created containing the two reactants which are initially separated by a buffer solution. In the second one, the droplets contain only one of the reagents. The other one is stripped from the organic phase, where it is solubilized by a complexing agent).

To realize these experiments in a reproducible manner, a microfluidic device has to be developed. It will create a segmented flow of aqueous droplets separated by an oil phase. The solute transport rates and mechanisms will be studied by comparing two different experiments (i.e with or without reactant transfer at the droplet interface).

Several requirements have to be fulfilled by the used device material to be suitable to conduct the studies in an application-like chemical environment. OSTE(+) fulfills those requirements, but lacks the ideal surface properties and has several drawbacks regarding the chip-to-world connectors, compared to PDMS. In order to overcome those handicaps, several solutions had to be developed for this not so commonly used material.

IV. Perspective

After successful reactant feeding is achieved, the investigations will concentrate on the precipitate formation by characterizing the effluents and precipitates and by developing an *in-situ* SAXS experimental setup. This will give insight into the mechanism of nucleation and growth in reactive emulsions and will allow us to model the transport phenomena at play. Furthermore, the results will allow for morphology control by controlling the mixing conditions in reactive emulsions.

References :

- [1] N. Yoshinaga and S. Aomine, *Soil Science and Plant Nutrition* **1962**, 8, 22-29.
- [2] P. Picot, *et al.*, *Faraday Discuss* **2016**, 191, 391-406.
- [3] T. Lange *et al.*, submitted.