

P-039 Microencapsulation of inverse emulsions by *in situ* and interfacial polymerisation

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INTRODUCTION AND OBJECTIVES

Polymerization microencapsulation methods, such as *in situ* polymerization and interfacial polymerization, are typically implemented in systems of oil-in-water (O/W) emulsions (Figure 1). In *in situ* polymerisation, all wall materials are water soluble, and originate only from the continuous aqueous phase. Due to a polycondensation reaction and reduced solubility, they evenly precipitate and distribute around the emulsified oily cores, thus forming microcapsules. In interfacial polymerisation, the first monomer is dissolved in the aqueous phase and the second monomer in the oily phase of an emulsion. Wall formation occurs at the interface of the emulsified droplets (Boh et al., 2010). To produce quality microcapsules by *in situ* or interfacial polymerisation in O/W emulsions, the active ingredients (future core materials) should be soluble in the appropriate oil, and should not mix/interact with water. Therefore, the first important technological step is the production of a stable O/W emulsion of a hydrophobic future core material (containing the active ingredient) in the continuous aqueous phase, followed by a polymerisation process for wall formation.

(Lin, 2006) or interact/react with water (Zvonar, 2010). To encapsulate them, formation of multiple emulsions seems to be an appropriate technical solution.

The main **purpose of our work** was to develop a microencapsulation procedure as a potential industrial technological process, based on *in situ* and/or interfacial polymerisation techniques, which would enable microencapsulation of water, aqueous solutions, and water-soluble core materials. The final product would be aqueous suspensions of microcapsules containing water-soluble active ingredients.

MATERIALS AND METHODS

For the preparation of inverse emulsions of water in oil (W/O), water, isopropyl myristate (Evonik), paraffinic oil (Petrol), Span 80 (Fluka) and Tween 80 (Merck) were used. For microcapsules wall formation, melamine-formaldehyde resin (Melamin), adipoly chloride (Aldrich), hexamethylenediamine (Aldrich) and styrene-malein anhydride copolymer (Hercules) were used. Microencapsulation by *in situ* polymerization was carried out in a 400ml glass reactor equipped with a heating and cooling control system (polymerization T = 70°C), and a turbine stirrer (1300 rpm). Microencapsulation by interfacial polymerization was carried out in a 50ml glass beaker, with a magnetic stirrer (400 rpm).

RESULTS AND DISCUSSION

Results of our first experiments are presented in Figures 2 – 5. With the suitable selection of emulsifiers, and adaptation and optimization of process conditions, it was possible to produce aqueous suspensions of microcapsules containing water-soluble active ingredients in multiple emulsions. Both processes, *in situ* and interfacial polymerization, are presented and compared in Figure 2. The advantage of interfacial polymerization are low-temperature operating conditions, compared to *in situ* polymerization, which requires heating (60 – 80°C) and change of pH to start the polycondensation reaction. These conditions have adverse effects on the stability of multiple emulsions. A weak point of the interfacial process is high reactivity of monomers, and fast wall formation. *In situ* process enables the formation of stronger microcapsule walls, and it is easier to control during the polycondensation step, which is important for an industrial scale production. However, it requires specific process conditions, i.e. higher temperatures and pH.

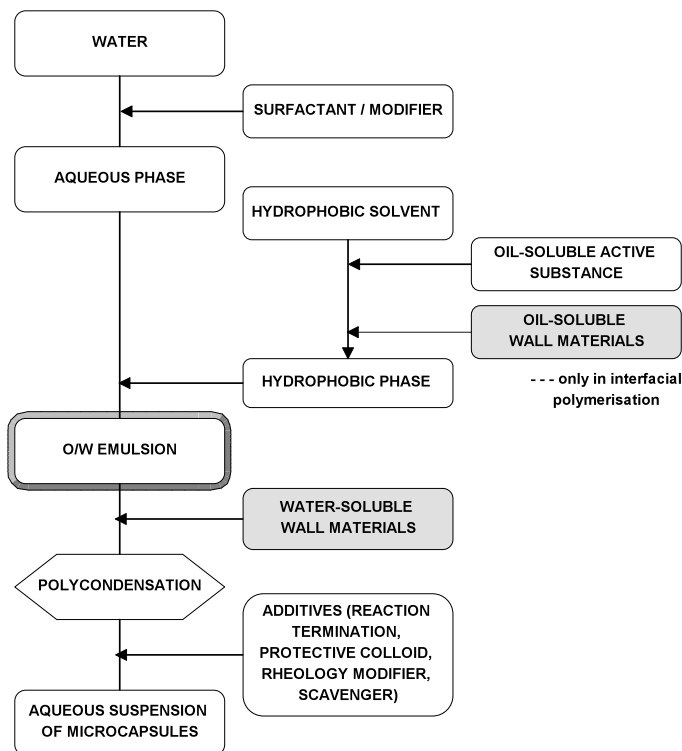


Figure 1: Diagram of *in situ* and interfacial polymerisation process in O/W emulsion

Success of the emulsification step depends on the solubility properties of core materials. However, in industrial applications, several core materials are water soluble

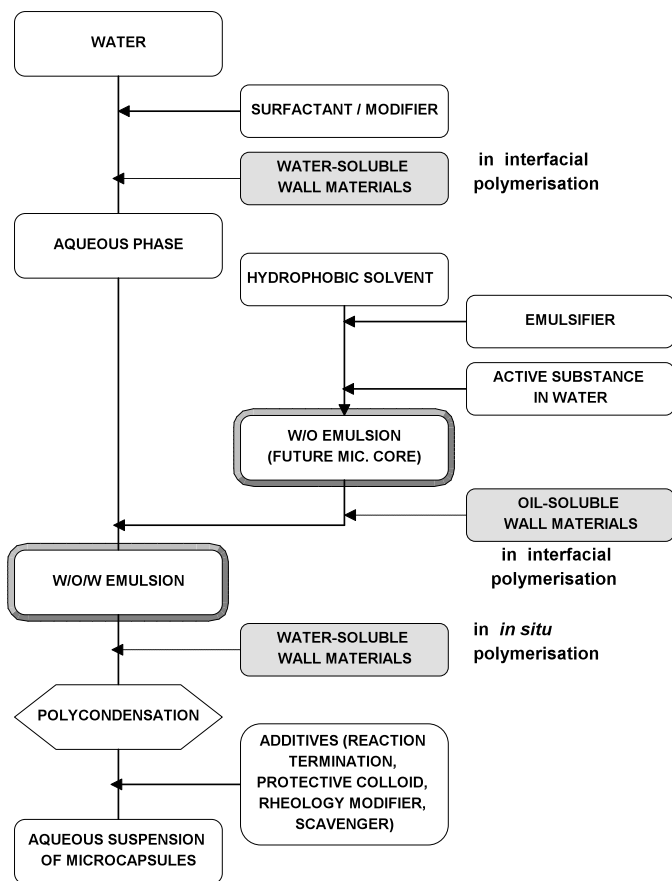


Figure 2: Diagram of *in situ* and interfacial polymerization process in W/O/W inverse emulsions

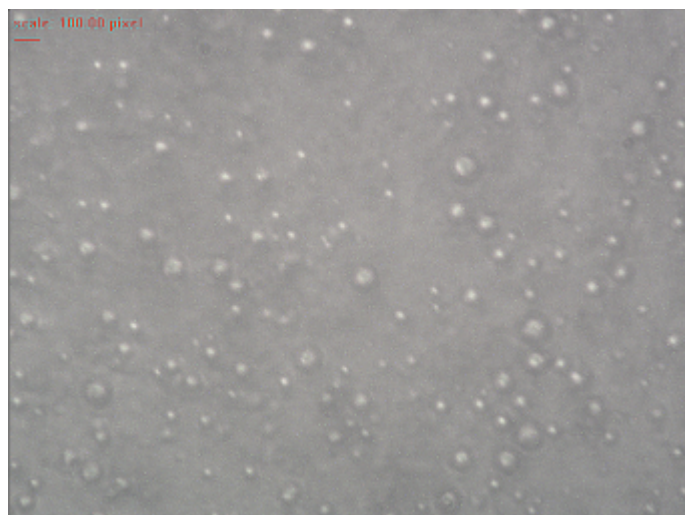


Figure 3: Microcapsules (1 μ m in diameter) prepared by *in situ* polymerization of an inverse emulsion with isopropyl myristate (aminoplast wall)

CONCLUSIONS

Laboratory experiments show that microencapsulation of water soluble active ingredients can be achieved by *in situ* and interfacial polymerization methods applying inverse/multiple emulsion systems. With further adaptations, optimization and scaling-up, the process may become attractive for different industrial applications.

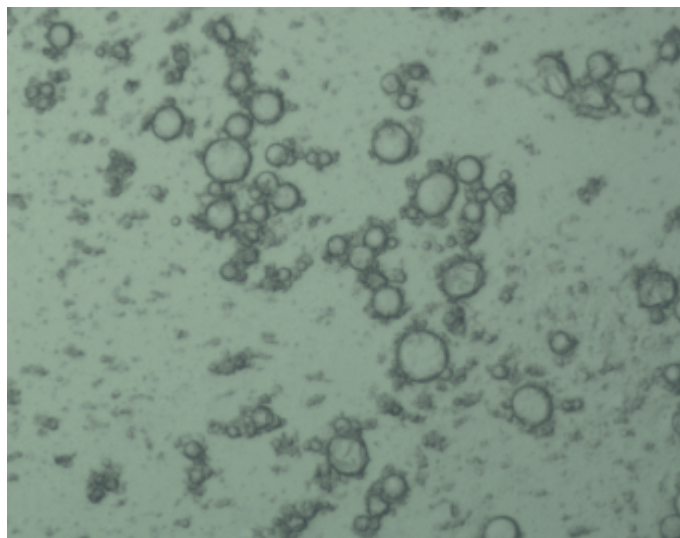


Figure 4: Microcapsules prepared by interfacial polymerization of inverse emulsion with paraffinic oil (nylon wall)

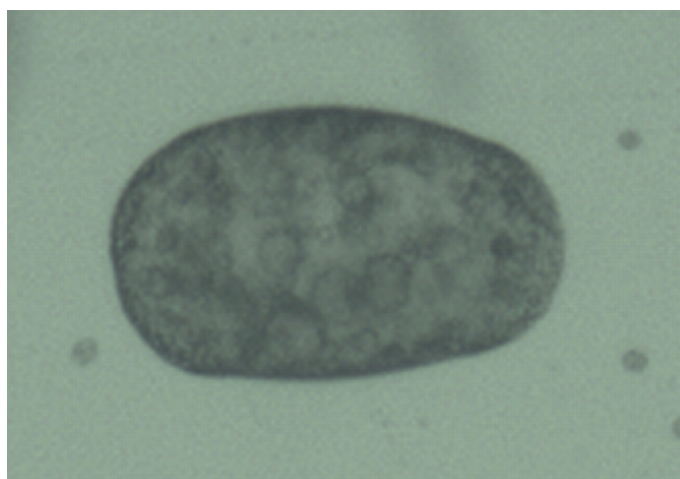


Figure 5: Enlarged microcapsule with nylon wall, containing an inverse emulsion of water in paraffinic oil

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