

# Novel encapsulation technology based on Pickering emulsion systems

M. Sjöö\*, J. Bedi\*\* and A. Ali\*\*\*

\* Speximo AB, Medicon Village, SE-223 81, Lund, Sweden, and Department of Food Technology Engineering and Nutrition, Lund University, PO Box 124, SE-221 00 Lund, Sweden, ms@speximo.com

\*\* Speximo AB, Medicon Village, SE-223 81, Lund, Sweden, jb@speximo.com

\*\*\* Speximo AB, Medicon Village, SE-223 81, Lund, Sweden, and Malmö University, Research Center for Biointerfaces, SE-205 06, Malmö, Sweden, aa@speximo.com

## ABSTRACT

A novel technology based on using particles of biological origin was recently developed for stabilising emulsions. This technology, that utilises Pickering emulsions, was further employed to encapsulate different types of active substances. Advance development and refinement of the technology has been performed in order to suit different encapsulation purposes in areas within personal care, food and pharmaceuticals. Some areas where this technology has been tested include taste masking in food systems, encapsulation and protection of a cosmetic active, production of oil filled powder encapsulates, and on-skin performance.

**Keywords:** Pickering emulsion, personal care, cosmetics, food, starch-based particles

## 1 ENCAPSULATION IN EMULSION BASED SYSTEMS

An innovative technology for encapsulation based on Pickering emulsions, i.e. emulsions stabilized by particles, was recently developed [1]. The use of starch-based particles for stabilizing emulsions provide a completely different stabilization mechanism compared to traditional systems where surfactants or molecules are used. In addition, the system was found very robust at wide ranges of formulation conditions, such as large variations of ionic strength, pH, oil polarity, etc. This technology was further employed to encapsulate different types of active substances. The specific particles used further allowed unique adjustment of the encapsulating barrier. In order to suit different encapsulation purposes and application areas, further development and advanced refinement of the technology was performed. The technology is commercialized by Speximo AB, a spin-off from Lund University in Sweden.

### 1.1 Technology based on particles

Using particles of biological origin is of high relevance, especially for the personal care and food sectors where the awareness regarding sustainability is increasing. The particles used were starch granules with a hydrophobic surface modification. Natural starch granules can be found in

different sizes in the micron range. In order to be used for Pickering stabilization and encapsulation, the particularly small granules, preferably 1 micron, are superior in terms of stability and function [2]. For the encapsulation results reported here, particles classified as naturally derived were used, although clean-label or eco versions have also been shown suitable for similar applications.

### 1.2 Adjusting the encapsulation barrier

Starch granules provide a unique advantage over other particles as they have the ability to swell upon heating. When they are attached to the surface of an oil droplet, this behaviour causes them to cover the droplet completely, see Figure 1. More commonly, molecules rather than particles are used for encapsulation. Compared to hydrocolloids in the molecular form, even of starch origin or other film forming agents, the barrier layer formed using the particle based technology would generally be thicker. The barrier also has different properties in terms of density and porosity.

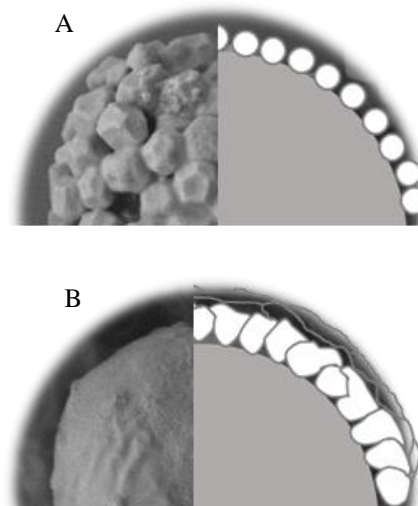


Figure 1: Scanning electron micrograph and schematic image of cross-section of an encapsulated oil droplet. Particles seen on the surface of an oil droplet before (A) and after (B) thermal treatment to cause particle swelling.

These properties make the particle based technology unique for encapsulation and barrier design. The ability to efficiently adjust and control the barrier properties has previously been demonstrated by measuring lipase digestion of encapsulated oil in an oil-in-water emulsion system [3]. The availability of the oil towards lipase digestion was successfully reduced and could be altered.

## 2 ENCAPSULATION EXAMPLES

The technology has been tested for encapsulation in different systems for various application areas. Some examples are given below, including food and cosmetic applications. The examples cover barrier adjustments as well as more advanced emulsion systems.

### 2.1 Taste masking

The present technology has been shown to encapsulate substances with a strong off-flavor for taste masking [1]. The patent demonstrated taste masking of omega-3 oil as an example of a food application, whereas a pharmaceutical example was the encapsulation and taste masking of penicillium. More recently, a paired comparison sensory study was performed using another strong tasting model oil. In this study, 86% of the participants experienced an efficient taste masking effect when using the present technology and a certain barrier treatment compared to a commonly used encapsulation system [4]. The traditional gum encapsulation was not significantly masking the oil flavour compared to free oil, see Figure 2, whereas the present encapsulation technology was efficient also with the two different barrier treatments according to Figure 1.

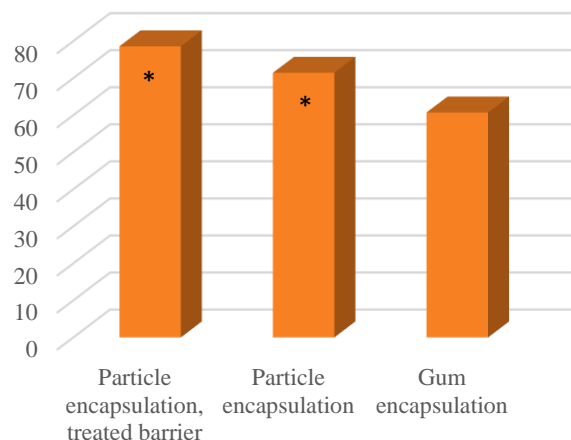


Figure 2: Taste masking effect of encapsulated oil compared to free oil. Paired comparison test n=28. Significant masking is noted \*.

### 2.2 Emulsion encapsulations

The technology was based on Pickering emulsion formation, causing highly efficient encapsulation in wet emulsion system. In different investigations dyes have been used as model substances, both in more common oil-in-water systems [2] and in more advanced double emulsions, i.e. water-in-oil-in-water systems [5]. The difference between these two encapsulation systems is shown in Figure 3. An oil soluble active is generally added directly to the oil phase, whereas a water soluble active is protected in a double emulsion system.

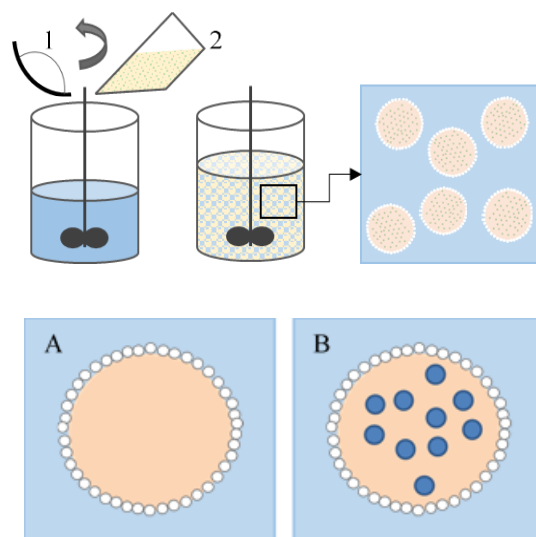


Figure 3: Encapsulation in oil-in-water system (A). First particles (1) were added to the aqueous phase, then the oil (2). Emulsification was performed by high shear mixing. For double emulsion encapsulation (B), the active substance was added in water droplets inside the oil before being mixed as above.

In addition to more general storage stability, the photo stability of a light sensitive cosmetic active was investigated in an encapsulated system based on the particle stabilization technology. The active was dispersed in the oil phase before emulsification. The method used for analysis followed guidelines for pharmaceutical tests and showed an increased stability. The projected activity at the end of the storage time was 2.8 times higher than for the reference sample.

### 2.3 Powder encapsulations

In many applications, for example when being used as an ingredient, it is beneficial to dry the encapsulate into an oil filled powder, see Figure 4. The ability to dry Pickering emulsions and also to subsequently reconstitute the powder with water has been demonstrated previously [6]. Even for a dried and reconstituted double emulsion an encapsulated

marker was retained at over 97% [7]. The properties of the encapsulated oil in combination with the barrier treatment was seen to affect the outcome of the drying process.



Figure 4: Dried oil filled powder for mixing with water or to be used as is to deliver encapsulated material to skin.

## 2.4 Fragrant oil filled powders

Encapsulation of flavours and fragrances is also relevant for personal care and cosmetic applications. In this case, the powders can also be applied directly on skin to deliver the oil and actives present. To demonstrate that, a fragrant oil was encapsulated using the Pickering system and freeze dried. The powder was found to be easily spreadable and gave a stronger fragrance when rubbed onto the skin, showing effective encapsulation. The powder did not leave visible remnants on the skin after application, see Figure 5.

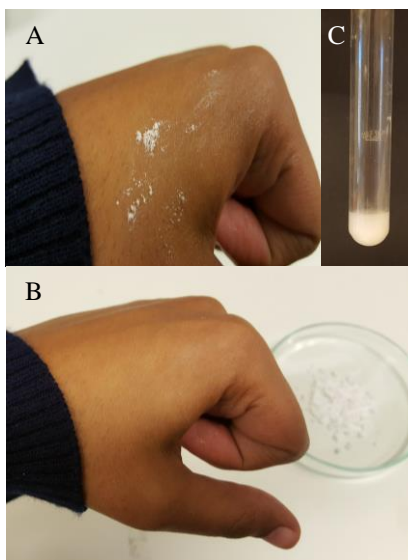


Figure 5: Fragrant oil filled powder upon application before being spread (A), showing ease in spreadability and good absorption (B) and upon reconstitution after drying (C).

As the powder also had good reconstitution ability in water as seen in Figure 5, it increases the ways in which this technology can be applied by the personal care and food industry.

## 3 ON-SKIN DELIVERY

The cosmetic feel and on-skin behavior of particle based creams was experienced as very positive in sensory tests [8, 9]. The cosmetic properties are highly important for the ability of an end product containing an active substance to reach the market. The present technology has shown a capacity of both encapsulation and on-skin delivery with a concurrent positive cosmetic feel [8]. Furthermore, Pickering emulsions have been seen to substantially increase transportation of active substances through skin [10, 11]. Consequently, the present technology may find use in a range of products for topical use, both within personal care and pharmaceuticals.

Skin delivery is related to permeability and absorption. A sensory test showed reduced stickiness and on-skin residues for a particle stabilized system compared to two commercial samples [12], see Figure 6. The commercial lotion was based on paraffin, therefore paraffin oil was used also for the particle stabilized system, which contained 56% oil. The permeability was sensed similar for all samples tested.

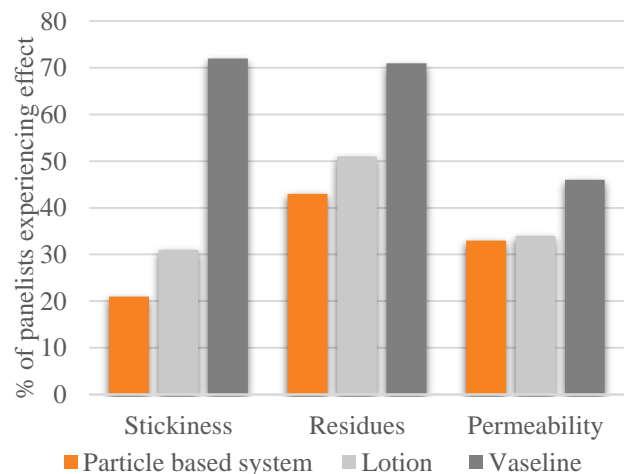


Figure 6: Sensory effect after absorption onto skin. The products tested were a simple model cream emulsion stabilized by particles, a commercial lotion, and commercial vaseline, n=9.

In another sensory test, a particle stabilized cream was designed to resemble a replica of a popular commercial hand cream. Again, the positive effects regarding afterfeel were confirmed, see Figure 7. The Pickering system significantly reduced the perceived stickiness, greasiness and residues on the skin after absorption.

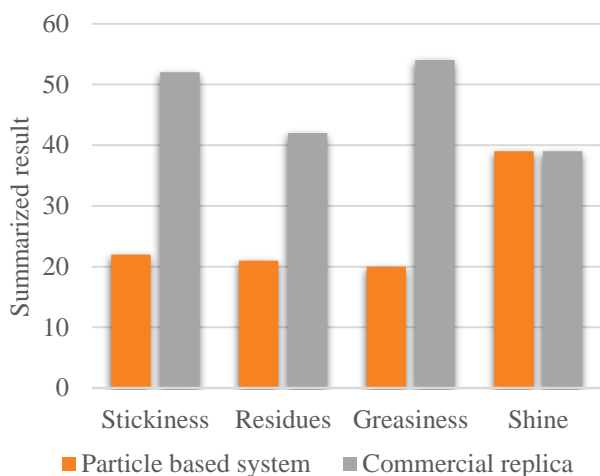


Figure 7: Sensory effect after absorption onto skin. The products tested were particle stabilized replica and a corresponding commercial replica., n=26.

## 4 SUMMARY

With the investigation undertaken at Speximo AB and in collaboration with Lund University, it can be seen that Pickering emulsion based encapsulations not only offer a way to solve sustainability issues but also give the industry a novel solution to achieve better stability and afterfeel in their end products. The particles, derived from an organic source, provide a strong alternative to available options in the market, which can appeal to several end consumers.

## REFERENCES

- [1] Dejmek, Rayner, Sjöo, and Timgren, Patent application, PCT/SE2011/051522, 2010
- [2] Timgren, Rayner, Dejmek, Marku and Sjöo, Food Science & Nutrition, 1 (2) 157-171, 2013.
- [3] Sjöo, Cem Emek, Hall, Rayner and Wahlgren, Journal of Colloid and Interface Science, 450, 182-188, 2015.
- [4] Knopp, Taste masking in Pickering emulsions, MSc Thesis, Lund University, 2015.
- [5] Matos, Timgren, Sjöo, Dejmek and Rayner, Colloids and Surfaces A: Physicochem. Eng. Aspects 423, 147– 153, 2013.
- [6] Marefati, Rayner, Timgren, Dejmek and Sjöo, Colloids and Surfaces A: Physicochem. Eng. Aspects 436, 512-520, 2013.
- [7] Marefati, Sjöo, Timgren, Dejmek and Rayner, Food Hydrocolloids, 51, 261-271, 2015.
- [8] Marku, Wahlgren, Rayner, Sjöo and Timgren, International Journal of Pharmaceutics, 428, 1-7, 2012.
- [9] Ali, Yilmaz and Sjöo, SOFW Journal, 141 (11) 1-7, 2015.

- [10] Frelichowska, Bolzinger and Valour, International Journal of Pharmaceutics, 368, 7-15, 2009.
- [11] Wahlgren, Engblom, Sjöo and Rayner, Current Pharmaceutical Biotechnology, 14, 1222-1234, 2013.
- [12] Marku, Pickering emulsions for topical delivery, MSc Thesis, Lund University, 2011.