

## Protection Of Oxygen-Sensitive Ingredients Using Composite Microencapsulation Materials



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### INTRODUCTION

Many ingredients in food and pharma products are sensitive to degradation by oxygen, leading to changes in taste or flavour, or even unhealthy effects. Microencapsulation of the ingredient, surrounding the ingredient with a shell material that prevents or reduces oxygen diffusion, can be used to create a longer shelf life for the envisioned product (Schrooyen 2001, Gouin 2004). Different shell materials can be used, depending on the ingredient, the type of encapsulation technology and the envisioned product. In particular, product and/or storage conditions with a high humidity or water content pose challenges to the choice of shell material (Sagalowicz 2010), as this might swell or dissolve, thus strongly reducing the protective effect.

We have investigated a new type of encapsulation material that is composed of a combination of a hydrophobic matrix material combined with crystalline particles to create oxygen protection under humid conditions. All selected components are generally recognized as safe (GRAS) for use in food products, and can be dissolved or digested in gastrointestinal conditions to allow release of the encapsulated ingredient.

### MATERIALS AND METHODS

A mixture of zein and lauric acid (70/30 w/w) was dissolved in an ethanol/water mixture (96/4 w/w) at 65°C. One solution was used as such, while calcium citrate (11w% or 21w% with respect to the zein/lauric acid) was dispersed in the other two. Subsequently, linseed oil was added and emulsions were obtained after sonification and used for spray-drying. As a reference, linseed oil was also encapsulated in gelatin matrix. This emulsion was prepared in the same way, but at room temperature. The exact composition of the different emulsions is shown in Table 1.

**Table 1 : Composition of spray-drying emulsions**

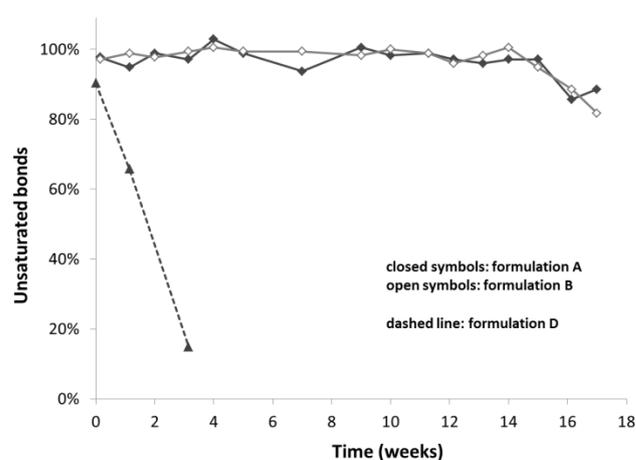
	A	B	C	D
Ethanol	279 g.	279 g.	280 g.	-
Water	11.7 g.	11.7 g.	11.8 g.	583 g.
Zein	56 g.	50.6 g.	50.7 g.	-
Lauric acid	22.6 g.	20.3 g.	20.4 g.	-
Gelatin	-	-	-	78.6
Calcium citrate	-	7.6 g.	15.2 g.	-
Linseed oil	31.2 g.	30.4 g.	30.9 g.	30.5 g.

Microencapsulated linseed oil was produced by spray-drying using a Büchi Mini Spray Dryer B-290, under N<sub>2</sub> atmosphere. The obtained powder was stored at room temperature at 90% relative humidity and stability of the encapsulated linseed oil was studied by monitoring the double bond conversion using FTIR spectroscopy. In a separate series of experiments, the powder (0.15 g.) was mixed with carboxymethylcellulose (1.5 g.) and water (25–29 g.) or 1M NaCl solution (17–18 g.) was added to create a hydrogel, stored at room temperature or 40°C, and stability of the linseed oil was again monitored by FTIR.

### RESULTS AND DISCUSSION

Zein, a hydrophobic plant protein, was selected as the major component for the shell material, since it combines good oxygen barrier properties with low swelling in the presence of water or water vapour (Lin 2007). It was mixed with lauric acid, which acts as a plasticizer to create a shell material with good film forming and mechanical properties. Crystalline calcium citrate particles were dispersed in the material to create additional oxygen barrier as well as increased mechanical stability of the shell material under humid conditions.

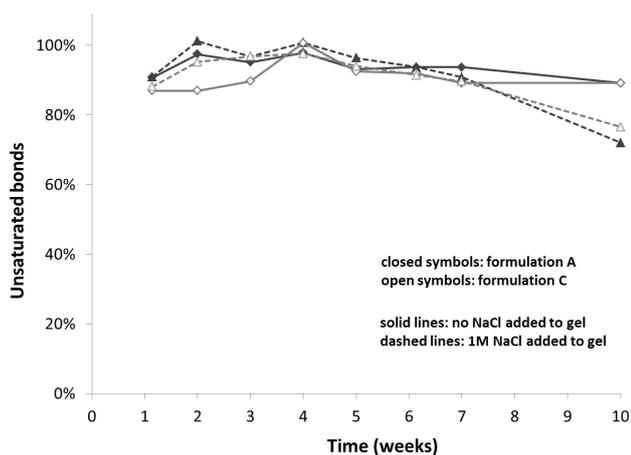
Linseed oil was selected as oxygen-sensitive model ingredient. It was encapsulated in the shell material by spray-drying starting from an emulsion of the oil in ethanol/water mixture, in which the shell material was soluble at elevated temperature.



**Figure 1 : Stability of encapsulated linseed oil in humid air (90% RH) at room temperature**

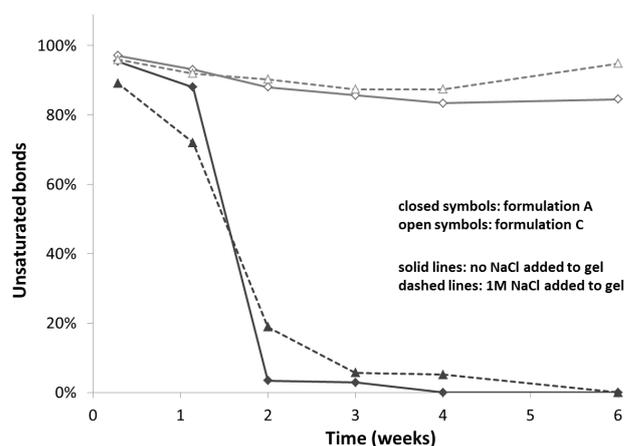
As a reference, linseed oil was also encapsulated in gelatin shell material. The obtained powders were stored in humid air (95% RH) and presence of unsaturated bonds in the linseed oil was monitored by FTIR spectroscopy. It was found that no significant degradation of the linseed oil occurred over a period of 16 weeks for the samples encapsulated in zein/lauric acid without or with calcium citrate added (Figure 1). Linseed oil encapsulated in gelatin was rapidly degraded in the first 3 weeks.

In addition, encapsulated linseed oil was mixed in a hydrogel to simulate a food product with high water content. Two different hydrogels were used, one with low ionic strength and one with high ionic strength (1M NaCl). Again, stability of the linseed oil was monitored by FTIR spectroscopy. It was found that at room temperature no significant degradation of the linseed oil occurred for at least 10 weeks in the gel with low ionic strength and at least 7 weeks in the gel with high ionic strength (Figure 2). The effects were similar for samples with and without calcium citrate in the zein/lauric acid shell material.



**Figure 2 : Stability of encapsulated linseed oil in hydrogel matrix at room temperature**

When storing the hydrogels at elevated temperature, a clear difference was found for the samples without and with calcium citrate in the shell material. Over a period of 6 weeks, good stability of the linseed oil was found for the samples encapsulated in zein/lauric acid with 21 w% dispersed calcium citrate, while samples encapsulated in zein/lauric acid without calcium citrate were fully degraded after 2–3 weeks (Figure 3).



**Figure 3 : Stability of encapsulated linseed oil in hydrogel matrix at 40°C**

## CONCLUSIONS

Linseed oil could sufficiently be protected against oxidative degradation by encapsulation in zein/lauric acid shell material for a period of at least 16 weeks of storage in high relative humidity, or at least 7–10 weeks when present in a hydrogel. Increased stability at elevated temperature was obtained by addition of crystalline calcium citrate particles in the shell material. This approach, based on edible materials, may be a promising approach for protection of oxygen-sensitive ingredients in food products with high water content.

## REFERENCES

- Schrooyen P.M.M. et al. (2001) *Microencapsulation: its application in nutrition*. Proc. Nutr. Soc. 60, 475–479.
- Gouin S. (2004) *Microencapsulation: industrial appraisal of existing technologies and trends*. Trends Food Sci. Tech. 15, 330–347.
- Sagalowicz L. (2010) *Delivery systems for liquid food products*. Curr. Opin. Coll. Int. Sci. 15, 61–72.
- Lin D. et al. (2007) *Innovations in the Development and Application of Edible Coatings for Fresh and Minimally Processed Fruits and Vegetables*. Compr. Rev. Food Sci. F. 6, 60–75.