Encapsulation of Peppermint Oil for Dental Care

Jing Xue and Zhibing Zhang*
Centre for Formulation Engineering, Chemical Engineering,
University of Birmingham, Edgbaston, Birmingham B15 2TT, UK
Email: Z.Zhang@bham.ac.uk

Introduction

Encapsulation has found more and more applications in various industrial sectors including paper, pharmaceutical, food, nutraceutical, household and human care, which can be used to stabilize active ingredients, and/or realize their controlled release (Pothakamury and Barbosa-Cánovas, 1995). The active ingredients can be solid powders, aqueous based or oil based materials.

Mint oil is commonly used for medicinal, culinary and cosmetic purposes. Mint is well known for its feature to ease indigestion and upset stomach. The menthol in mint can help to clear sinuses and relieve cold symptoms. In some cases, mint is used to alleviate migraines and fever (McKay and Blumberg,2006; Baratta et al.,1998). Mint oil is one of the most popular and well appreciated flavors, but it also has one of the most persistent odors (Tate, 1997) which potentially can transfer a mint flavor to a material it comes in contact with or can be oxidized by the material such as peroxide. Encapsulating mint oil may help to stabilize the formulations of toothpaste or chewing gum by physically separating the components in order to prevent possible chemical reactions between them. The main aim of this study was to encapsulate mint oil using a food grade carrier which is compatible with mint oil. Moreover, the encapsulation conditions should be mild and doe not require any organic solvent or heat. The formed capsules should have a low permeability, optimum mechanical strength, and the release of the active ingredient may be realized by a mechanical force resulting from a toothbrush or chewing.

Material and Methods

Peppermint oil, gum Arabic and whey protein that were used in experiments were all of reagent grade. All of them have been purchased from Sigma, UK and used as received.

Encapsulation of peppermint oil by coacervation and physical characterization of microcapsules
Whey protein and gum Arabic was dissolved in distilled water respectively. The concentration of whey protein and gum Arabic aqueous solution each was 1% w/v following Weinbreck et al (2004). The volume of whey protein solution was 100ml. 5ml Peppermint oil was added into the whey protein solution and mixeded for 10 minutes using a homogenizer with a rotation speed of 600rpm (L4RT, Silverson, UK).50ml Gum Arabic solution was added into the oil in water emulsion system, which was homogenized for another 30 minutes. The encapsulation process was kept overnight. Microcapsules with peppermint oil as a core material and whey protein/gum Arabic copolymer as a coating material were formed under the conditions of different pH 3, 4 and 7 respectively. The suspension solution was then centrifuged and microcapsules were collected from the top layer of the solution and stored in distilled water at 4^{o}C.
The schematic diagram of encapsulating peppermint oil using gum Arabic and whey protein coacervate is shown in Figure 1.

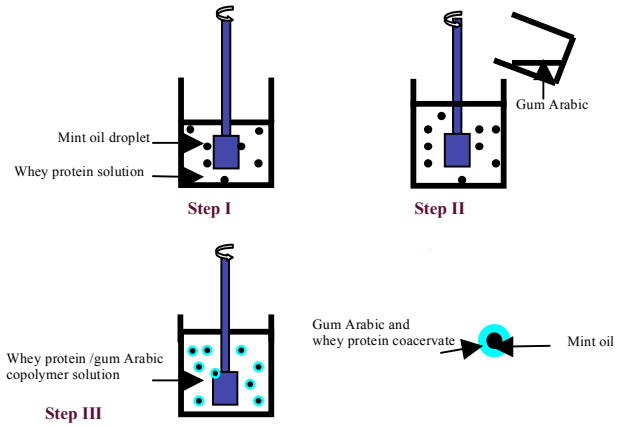

Figure1: Schematic diagram to illustrate preparation of peppermint oil capsules using a coacervation method.

The size distribution of peppermint oil microcapsules was quantified by a Marlvin size analyzer respectively.

Measurement of Encapsulation Efficiency

A known amount of hexane was added to the suspension solution which contained peppermint oil microcapsules. The solution was gently mixed by a glass rod to facilitate complete diffusion of the peppermint oil which had not been encapsulated into the hexane phase. The 2-phase system was allowed to equilibrate for 10 minutes. 4ml of the sample was taken from the hexane phase and was analyzed for its menthol content by UV spectroscopy at the wavelength of 230nm (Sibanda et al, 2004), since menthol is the principal bioactive component of peppermint oil. The Encapsulation Efficiency is defined as follows:

$$\text{Encapsulation Efficiency} = \frac{V_0 - V_H}{V_0} \times 100\%$$

where V_0 is the volume of initial input of mint oil and V_H is the volume of mint oil which hadn't been encapsulated.

FTIR analysis

In order to determine whether the peppermint oil had been encapsulated in the microcapsules or not, Fourier Transform Infra Red Spectroscopy (FTIR) was used to measure the absorption of light by molecular vibrations in samples. Peppermint oil microcapsules that had been washed by hexane were ground in a glass mortar to release the core material. This pulverized mass was placed on a plate in FTIR and scanned. The infrared spectrum is plotted as a graph of the fraction of light absorbed by each sample through a range of light frequencies.

Micromanipulation

A micromanipulation technique has been applied to measure the force resistance of the produced peppermint oil capsules. The basic principle of this technique is to squeeze single capsules in a mixture of water/ethanol medium on a glass slide, by stamping it between a glass probe with a flat end and the slide surface (Mashmoushy et al., 1998; Sun and Zhang, 2001) as shown in Figure 2 below.

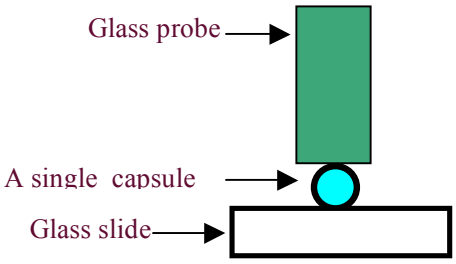

Figure2: Schematic graph to illustrate compression a single capsule between a glass probe and a glass slide by using the micromanipulation technique.

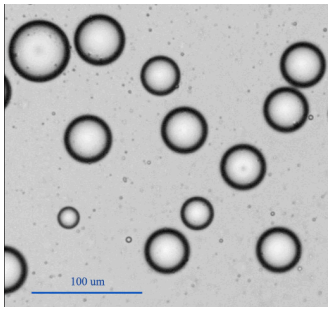

Figure3. Peppermint oil capsules in aqueous solution

Results and Discussion

Figure 3 shows an image of the produced microcapsules under a microscope. They look very spherical although there is a variation in size. The size distribution of the microcapsules prepared at different pH values is illustrated in Figure 4. It can be seen from Figure 4 that the size distributions of peppermint oil capsules produced at pH =3, pH = 4 or pH=7 respectively are very similar. This is expected since the size was dominated by the rotation speed of the homogeniser.

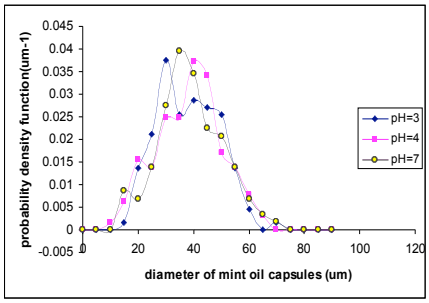

Figure 4. Size distribution of peppermint oil-whey protein/gum Arabic capsules produced at different pH values just after the encapsulation process.

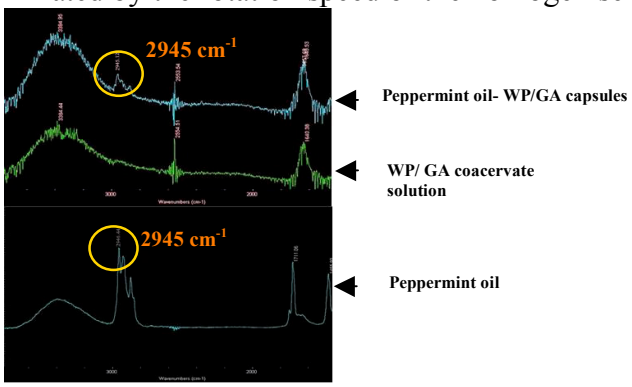

Figure 5: FTIR spectra of peppermint oil, whey protein/ gum Arabic coacervate and peppermint oil capsules with whey protein and gum Arabic as a coating material.

The FTIR spectra of peppermint oil, whey protein/ gum Arabic coacervate and peppermint oil capsules with whey protein and gum Arabic as a coating material are presented in Figure 5. By comparing the FTIR spectra of whey protein/gum Arabic coacervate and the pulverized mass of peppermint oil capsules, it can be seen that the function group of peppermint oil, C-H stretch group, which would have the absorption at the light frequency of 2950 cm^{-1} to 2840 cm^{-1} has been obtained in the pulverized mass of peppermint oil capsules, but not in the spectra of whey protein/gum Arabic coacervates. This proves that peppermint oil has been encapsulated in the whey protein/gum Arabic capsules. The encapsulation efficiency based on freely made microcapsules and those stored in water for 7 days are given in Table 1. Basically, there were losses of the peppermint oil from the microcapsules to a different extent probably due to the oil leaking out with time when they were stored in water. It should be pointed out that the microcapsules produced at pH = 4 was relatively stable and there was only a marginal drop in the encapsulation efficiency.

Encapsulation Efficiency	pH=3	pH=4	pH=7
Fresh capsules	85%	85%	80%
Capsules after 7day	70%	80%	20%

Table1. Encapsulation Efficiency of peppermint oil capsules produced under different pH conditions.

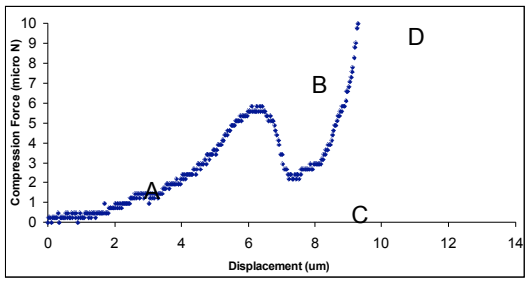

Figure 6.Typical force versus displacement for compressing a single peppermint oil-whey protein/ gum Arabic capsule to rupture.

A typical force versus probe displacement for compressing a single microcapsule to rupture is presented in Figure 6. Curve AB corresponds to the compression, B the rupture of the capsule and CD compression of the ruptured debris. The force at point B is called the rupture force of the microcapsule, which is approximately 6 μN. This force is significantly smaller than those of melamine formaldehyde microcapsules, and the latter is in the range of 100-1000μN (Sun and Zhang, 2001). Measurement of the rupture force of a large number of peppermint oil microcapsules seemed to be difficult because they were lighted than the aqueous solution, floated on the surface of the liquid and tended to collapse after they were dried in air. More work to make stronger peppermint oil capsules is required in future.

Conclusions

Peppermint oil has been successfully encapsulated using whey protein/gum Arabic as a coating material. The encapsulation efficiency was 80~85%. The storage stability of the microcapsules depended on the pH value at which they were prepared, and those at pH 4 seemed to be the most stable. However the prepared capsules seemed to be weak and more work is required to improve their mechanical strength and storage stability.

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