Carnauba wax as a carrier for aroma encapsulation

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Introduction

The most significant features of microcapsules are microscopic size and surface or interface area. Through selection of the composition materials (core material and membrane), we can endow microcapsules with a variety of functions (Yoshizawa, 2004).

Waxes are of interest for the preparation of controlled release systems by means of melt granulation or extrusion techniques (for granules and beads) and melt dispersion or spray congealing techniques (for microparticles) which does not require the use of solvents (Passerini et al., 2003). *Carnauba* wax is one of the classic materials used in pharmaceutical formulation work, e.g. as a glossing agent. It has also been suggested as a potential matrix material or even as a solvent for solid dispersions for sustained-release tablets or microspheres manufactured by coacervation spray-congealing or extrusion (Emas, 2000). Waxy materials have major application in sustained release systems and the use of the wax matrix appears to have several advantages such as being a multiple units system, chemical inertness against other materials and ease of manufacturing with high reproducibility that can be obtained without special instrumentation, as well as low production cost. *Carnauba* wax was used to produce lipid matrix granule and tablets of metronidazole (Özyazýcý, 2006). Resent researches have been made on encapsulation of sunscreens into lipid matrices consisting of *Carnauba* wax (Villalobos-Hernandez et al., 2005; 2006a; 2006b, 2007). It was shown that lipophilic carriers such as *Carnauba* wax might be used for delivering highly water-soluble drugs like potassium citrate (Cao et al., 2007). The advantages of waxes include good stability at varying pH and moisture levels, well established safe application in human due to their nonswellable and water insoluble nature, minimal effect on food in the gastrointestinal tract, and no dose damping (Kamble et al., 2004).

Development of new lipophilic carriers has provided impetus to the research in the area of the processing techniques involving molten state (Paradkar, 2003). In this study *Carnauba* wax was used, due to aforementioned advantages, to optimize and to develop experimental procedure for producing microparticles with uniform and narrow size distribution and spherical shape. Melt dispersion technique was applied as a simple non-solvent and low cost method. This method basically involves emulsification of the molten mass in the aqueous phase, followed by its solidification by chilling. Effects of following process variables were investigate on the bases of particle size distribution and visualization: concentration of the internal phase (*Carnauba* wax), stirring time and stirring speed. In the next research stage the effect of surfactants (Tween 20, Span 40, Span 60), under optimized experimental conditions were examined.

Materials and Methods

Feed grade *Carnauba* wax was purchased from Carl Roth GmbH (Germany). Emulsifiers (Tween 20, Span 40, Span 60) were supplied from Sigma Aldrich (Germany).

The size distribution of the particles was evaluated by sieve analysis, using set of five standard sieves in the range 125 – 1190 μm.

Photoimages of the produced microparticles were carried out by means of stereo microscope (Leiter MR6), equipped with Sanyo camera (resolution 800 x 600 pixel, program – Image Pro plus v.6.2). Shape and surface properties of wax beads are observed by scanning electron microscopy. The microspheres were preparing for measurement by Pt-Pd steaming.

Carnauba wax (1 – 8 % w/w) was melted in purified water (264 ml), kept on 95° C by termostated water bath, until wax was completely melted. Melt dispersion was mixed by applying different mixing speeds (1000 – 1500 r/min) and different mixing times (2 – 15 min), by mechanical stirrer with two blade impeller. Solidification of microdroplets was performed by cooling melt dispersion with cold water (2-5 °C). *Carnauba* wax beads obtained after solidification were collected by filtration under reduced pressure, washed and dried at elevated temperature (50 °C).

Results and Discussion

Uniform particle size distribution is critical for many applications, especially in situations where only a very narrow melting window is available. Very large microparticles or microcapsules can cause localized effect such as failure to distribute and eventually form immobile melted masses. Very small particles on other hand will have a large surface area.

Melt dispersion technique involves an emulsification of molten mass. Droplet size may depend on different process variables (concentration of *Carnauba* wax, stirring speed and stirring time). An optimized procedure for wax microspheres was developed on the basis of particle size distribution and visualization.

Following concentrations were examined: 3.6, 5, 6, 8, 10 % w/w. Effects of different concentrations on particle size distribution are presented on Figure 1. Results show that with increasing of internal phase portion (*Carnauba* wax), particles have wider particle size distribution. Reason for larger microparticles might be the coalescence of emulsion droplets into larger entity. With 3.6 % w/w of wax in dispersion, satisfied distribution was obtained, with prevalent fraction in the range 125 – 297 μm (69.6 %).

Investigated stirring times were: 2, 4, 7, 10, 15 min. Distribution of particle size given for different time conditions is shown on Figure 2. Results of particle size distribution indicate that particles obtained in the experiments with mixing time from 4 to 15 minutes have narrow distribution. Among them, due to the highest contribution of fraction 125 – 297 μm (70.7 %) stirring time of 4 minutes is considered as the most suitable. Mixing time of 2 min was insufficient for forming droplets with appropriate size distribution (microparticles in the range 297 – 420 μm have unsatisfied contribution).

The electronic microscope image of wax microparticles shown on Figure 3 presents high uniformity and sphericity of produced microbeads. SEM image on high magnification of microbeads shown on Figure 4 does not indicate any changes on particles surface caused by emulgents.

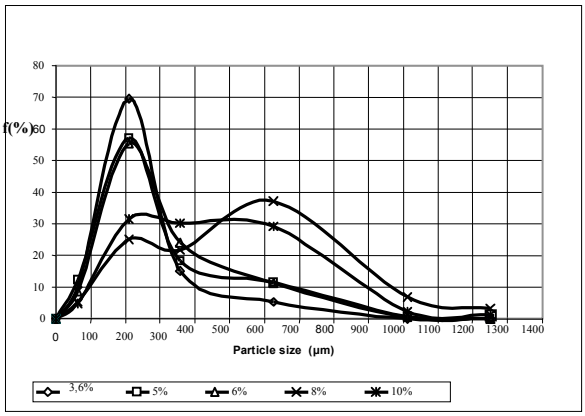

Figure 1. Particles size distribution curves obtained with different concentration of *Carnauba* wax (f – frequency of distribution)

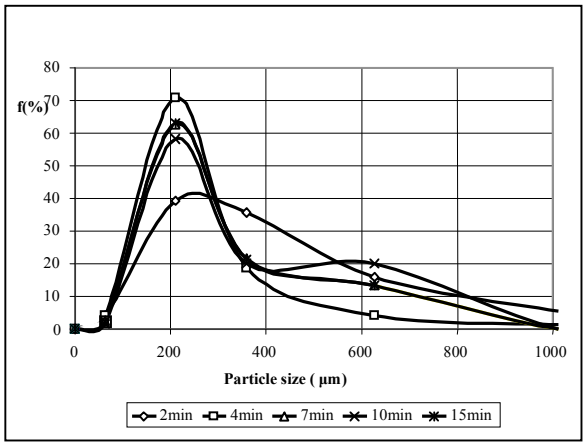

Figure 2. Particles size distribution curves obtained with different stirring times (f – frequency of distribution)

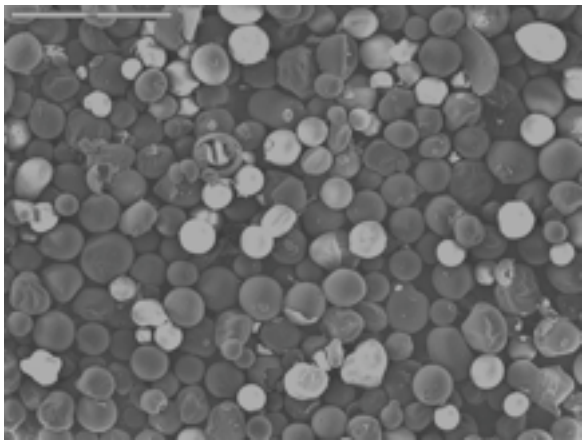

Figure 3. Wax microbeads produced by melt dispersion technique

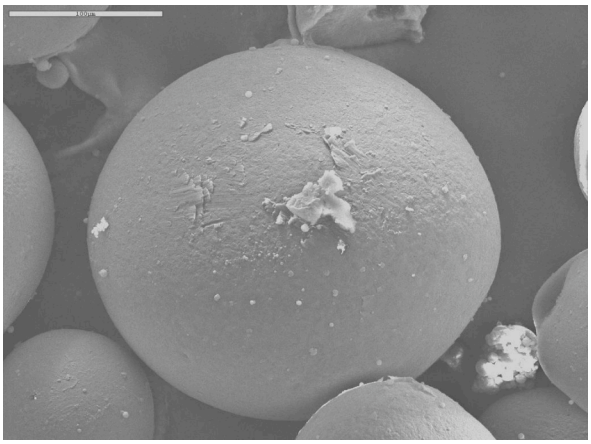

Figure 4. SEM image of a bead made from *Carnauba* wax

Conclusions

Microparticles of *Carnauba* wax with narrow size distribution can be produced by melt dispersion technique. More uniform size distribution was achieved by using emulsifiers. With mixture of emulsifiers Tween 20 and Span 40 significantly high contribution of prevalent fraction in the range 125 – 297 μm was achieved (79 %). It was not observed significant influence of emulsifiers on surface properties and shape of produced wax-particles.

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