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Note

Production of aqueous shellac dispersions by high pressure homogenisation[☆]

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Abstract

The use of shellac for coating purposes in pharmacy, food and nutriceutical industry generally involves the use of organic solvents. An aqueous shellac dispersion has been developed by applying high pressure homogenisation. Shellac was dissolved in alkaline solution, precipitated by HCl addition and the obtained suspension homogenised at 1500 bar applying 20 cycles. The volume diameter 50% was 2 μm, a distinct fraction of particles was in the nanometer range. This surfactant-free shellac dispersion with 20% solid content proved to be physically stable and showed good film forming properties. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Shellac; Zeta potential; High pressure homogenisation; Surfactant-free

Shellac is widely used in food industry, to some extent still in pharmaceutical industry and—a market of growing interest—in nutritional supplements, health supplements and nutriceuticals. The latter market being of special commercial interest compared to the food market because the prices of the goods are higher. On the other hand, the production volume in food industry is much higher. Shellac can be used as a moisture barrier in food and nutritional/health supplements. A product with similar application is Sepifilm LP from Sepic. Further applications of shellac are enteric coating for food, retard coating for food (for example vitamins) and it can be used as sub-coat or gloss coat (Specht et al., 1998). Shellac is of special interest because it is one of the few excipients allowed for these coating purposes in food. In addition, it is a natural material from renewable resources (extraction product of Tachardia lacca, a plant louse).

However natural materials show a distinct disadvantage, namely undesired batch-to-batch variation. Further, the major problem associated with

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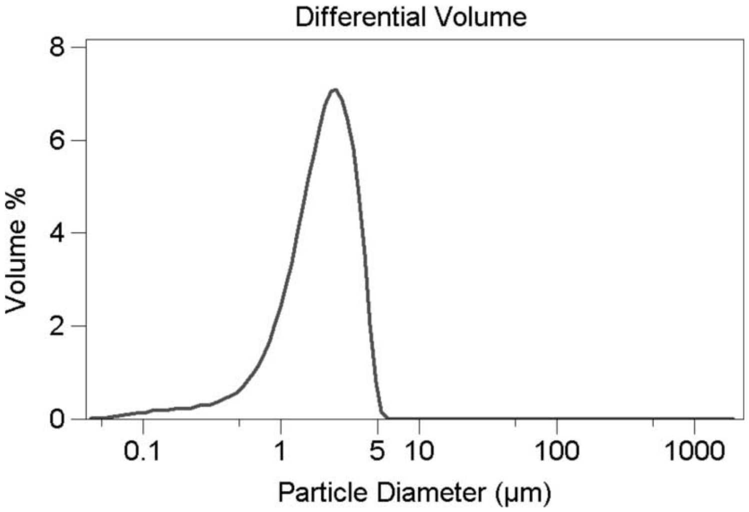

Fig. 1. Particle size distribution of a 20% (w/w) surfactant-free aqueous shellac dispersion.

shellac is the need of organic solutions for processing purposes. Difficulties may arise regarding solvent removal in an environmentally acceptable way (recovery), in addition solvent residues need to be considered. An aqueous shellac dispersion would remove these problems. This paper describes the development of an aqueous shellac dispersion based on particles in the nanometer range up to ~5 μm, its characterisation and stability including film forming properties.

Shellac was obtained from Stroever GmbH (Bremen, Germany) and used without further purification. Tween 80 was used as surfactant, purchased from Sigma Aldrich (Deisenhofen, Germany). Production was performed as described for pseudo latex particles (Specht, 1995). The shellac was dissolved in sodium hydroxide solution at pH 8–9 then hydrochloric acid was added to precipitate the shellac while the solution was stirred using an Ultra Turrax (Jahnke & Kunkel GmbH, Staufen, Germany) at 9500 rpm. The obtained suspension was passed through a high pressure homogeniser Micron LAB 60 (APV Deutschland GmbH, Lübeck, Germany). Production was performed in the continuous mode for 30 min applying a pressure of 500 bar. Particle size analysis was performed by laser diffractometry (LD) (Coulter LS230, Coulter Electronics, Krefeld, Germany). Laser diffractometry yields a volume distribution which weighs large volume particles more intensively. Therefore size data are generally higher compared to data, e.g. from photon correlation spectroscopy (PCS). The diameters 50, 90 and 95% (D50, D90, D95%) were used as characterisation parameters.

The produced formulation contained 20% (w/w) shellac and water, that means it was surfactant free. The LD size distribution revealed that almost 100% of the particles were < 5 μm and almost 20% were below 1 μm in the nanometer

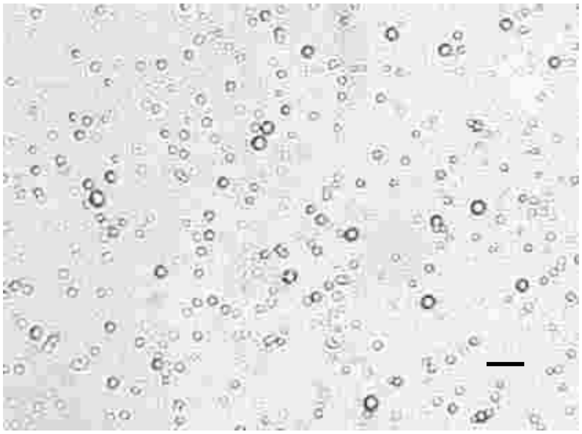

Fig. 2. Light micrograph of the shellac dispersion after 30 min of production (bar = 5 μm).

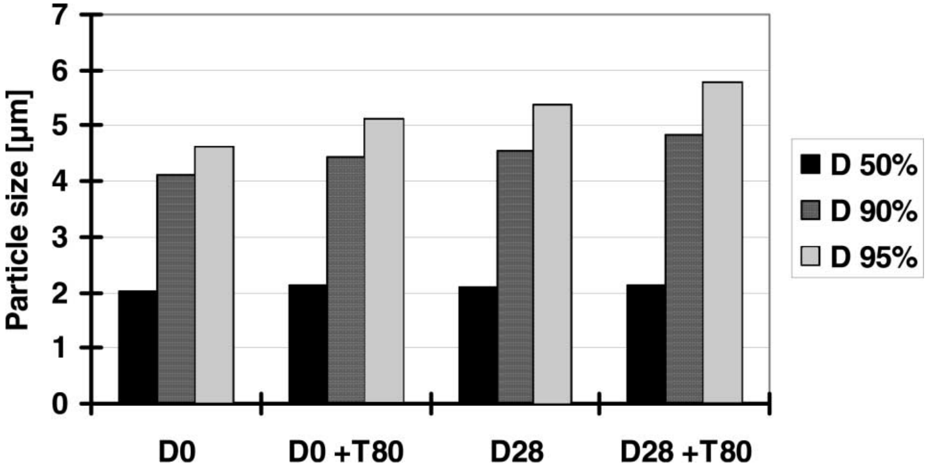

Fig. 3. Diameters 50, 90 and 95% of the surfactant-free shellac dispersion and the additionally Tween 80 stabilised (+T80) shellac dispersion as a function of time (D0 = day of production, D28 = day 28).

range (Figs. 1 and 2). The diameter 50% was 2.12 µm, D90% 3.65 µm and D95% 4.06 µm.

The zeta potential of the particles was determined by laser Doppler anemometry (LDA) (Zetasizer 4, Malvern Instruments, Malvern, UK). Determination was performed at 20 V/cm, Helmholtz–Smoluchowski was used to calculate the zeta potential. The zeta potential was found to be −52.0 mV in distilled water and −57.3 mV in distilled water with a conductivity adjusted to 50 µS/cm by NaCl addition (Müller, 1996). Such a high zeta and therefore related Nernst potential promotes physical stability of dispersions even in the presence of medium electrolyte concentrations. This fact is of special interest regarding, e.g. food processing.

Optimal stability can be achieved by combining electrostatic repulsion with steric stabilisation. To investigate the influence of a steric stabiliser, Tween 80 was added in a concentration of 0.1%. Tween 80 is a well tolerated surfactant, even registered for intravenous formulations in pharmaceutical products. Tween 80 was added after production of the surfactant-free shellac dispersion. The physical stability of the shellac dispersions with and without added Tween 80 was monitored by laser diffractometry up to 4 weeks. There was a slight increase in the characterisation diameters after the day of production—maybe caused by some ripening effects—but little change during the observation period of 4 weeks (Fig. 3). During the storage period limited sedimentation occurred due to the particle fraction >3 µm because of the density of shellac being ~1.07 g/cm^3. This size was estimated by calculations based on the Stoke sedimentation law. The sediment could be redispersed by manual shaking.

A prerequisite for the application of the aqueous shellac dispersion for coating is a good film formation. To test film forming properties, the aqueous shellac dispersion was poured in a thin layer onto a petri dish. The water evaporated at room temperature leading to a fine, dense and elastic film with good optical appearance. A dense membrane was formed.

To summarise: High pressure homogenisation is a suitable method to produce solvent-free, aqueous shellac dispersions. Physically stable surfactant-free dispersions with a high Nernst potential can be produced, the diameter 50% being 2 µm with a distinct fraction of particles in the nanometer range. The production costs of the aqueous shellac dispersions are low because high pressure homogenisation is a technique widely used in industry, available on large scale and the equipment can be bought off the shelf, i.e. it is low-cost equipment. The availability of water-based shellac dispersions can also increase the use of shellac in pharmaceutical industry because the major disadvantage compared to aqueous polymethylmethacrylate dispersions—the need for an organic solvent—has been removed.

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