Physical properties of aminoaldehyde microcapsules

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Introduction

In *in situ* polymerization of aminoaldehyde resins, all materials for the microcapsule wall originate from the continuous (aqueous) phase of the emulsion. Under ideal conditions, by change of pH or temperature, all mass of the wall material precipitates and distributes evenly over the surfaces of droplets in the emulsion to form spherical microcapsules.

Materials and methods

A modified *in situ* polymerisation method by Knez (1995) and Kukovič & Knez (1997) was used for the preparation of microcapsules. Paraffinic phase change materials (PCM) with different melting points were used as core materials, melamine-formaldehyde prepolymers as wall materials, and styrene-maleic acid anhydride copolymers as modifying/emulsifying agents.

Microcapsule diameter and size distribution were measured by Alkatel Cilias Laser Granulometer 715. Olympus microscope BX60 with a Sony CEN50 camera was used for characterization of visual appearance, individual microcapsule size and morphological characteristics of microcapsules. Scanning electron microscopy was performed by a JEOL JSM-6060LV microscope, at accelerating voltage 15 kV, with microcapsule coating C+Au/Pd. The mechanical strength of PCM microcapsules was tested by a smudging colouration test (Figure 1), which was originally designed for pressure-sensitive copying papers. A leuco dye marker was incorporated in microcapsules, prepared by the same procedure as for PCMs, except that a 3% Crystal violet lactone leuco dye in KMC-113 diisopropyl naphthalene was used as a core material.

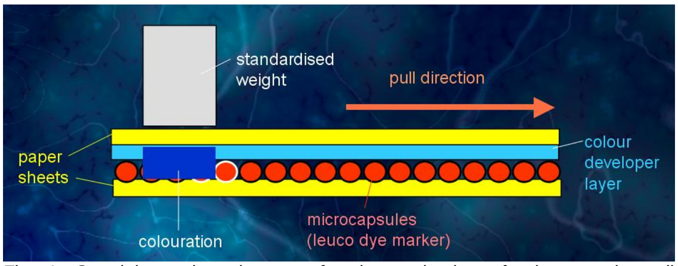

Fig. 1: Smudging colouration test for the evaluation of microcapsule wall resistance to mechanical stress, developed for carbonless copying papers

The relative thermal stability of different batches of microcapsules was evaluated by a modified smudging colouration test at an elevated temperature. A paper sheet with leuco dye marker microcapsules and a colour developer sheet were placed on top of each other, and exposed to 120°C in an oven, under the pressure of standardised weights, for 10, 20 and 30 minutes. In cases of insufficient mechanical or thermal stability of microcapsules, a leuco dye marker diffused into a colour developer layer. Staining was proportional to the microcapsule wall damage (Boh et al., 2005). The melting points of PCM microcapsule cores were determined by differential scanning calorimetry - DSC (Perkin Elmer Pyris-1).

Results and discussion

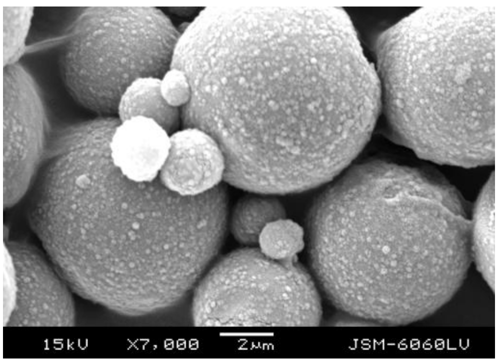

Fig. 2: SEM of microcapsules with PCM as core material produced by *in situ* polymerization

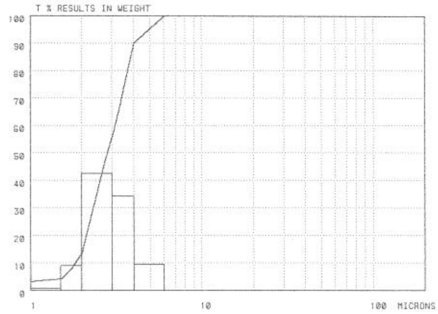

Fig. 3: An example of a size distribution of microcapsules with PCM as core material produced by *in situ* polymerization

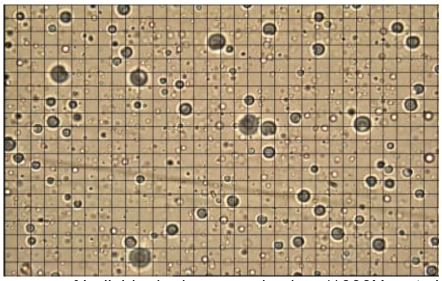

Fig. 4: Measurement of individual microcapsule sizes(1000X; net size 5μmX5μm)

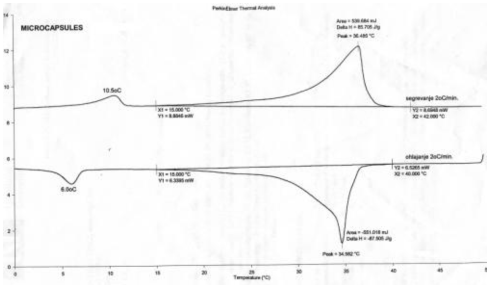

Fig. 5: DSC of microencapsulated PCM

Table 1: Characteristics of PCM microcapsules with aminoaldehyde walls

Parameter	Value
Form and appearance	Aqueous suspension, white
Microcapsule size	1-10 μm
Suspension viscosity	500-1000 mPas
Solid (non-water) content	30 – 35 %
pH value	5,5 – 8,0
Free formaldehyde	0,05 – 0,5 %
Core content in dry microcapsules	700-750 mg/g
Melting points of PCM core material	less than 50°C
Thermal stability	Less than 1% leakage after 1h at 135°C

Conclusions

Microcapsules produced by *in situ* polymerization of melamine-formaldehyde prepolymers can have totally impermeable walls that are durable, resistant to mechanical and thermal stress, and are suitable for microencapsulation of paraffinic PCMs. The described microcapsule characterisation methods are simple, fast, cost effective and therefore appropriate for routine industrial uses.

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References

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