Preparation of Mannitol@Silica Core–Shell Capsules via An Interfacial Polymerization Process from Water-in-Oil Emulsion

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Introduction

Microcapsules with silica shell have drawn tremendous attention in the past years with the advantages of high thermal and chemical stability, environmental friendly, low cost, and low toxicity [1-3]. Silica shells were used to encapsulate organic phase change materials (PCMs) because of their thermal cycle stability and thermal conductivity, showing promising applications in the field of energy storage and thermal shielding.

Mannitol@Silica capsules with thick shell layer have been prepared via hydrolysis and polycondensation of the bicomponentsilicon precursors mixed with tetraethoxysilane (TEOS) and 3-aminopropyl triethoxysilane (APTS) on the interface of W/O emulsion. The mannitol crystal was well sealed inside the silica capsule under an optimized condition and the leakage was successfully avoided. The Mannitol@Silica core-shell capsules own irreversible phase change character in the range of 142.1–166.2◦C with latent heat of 147.4 J/g.

Results and discussion

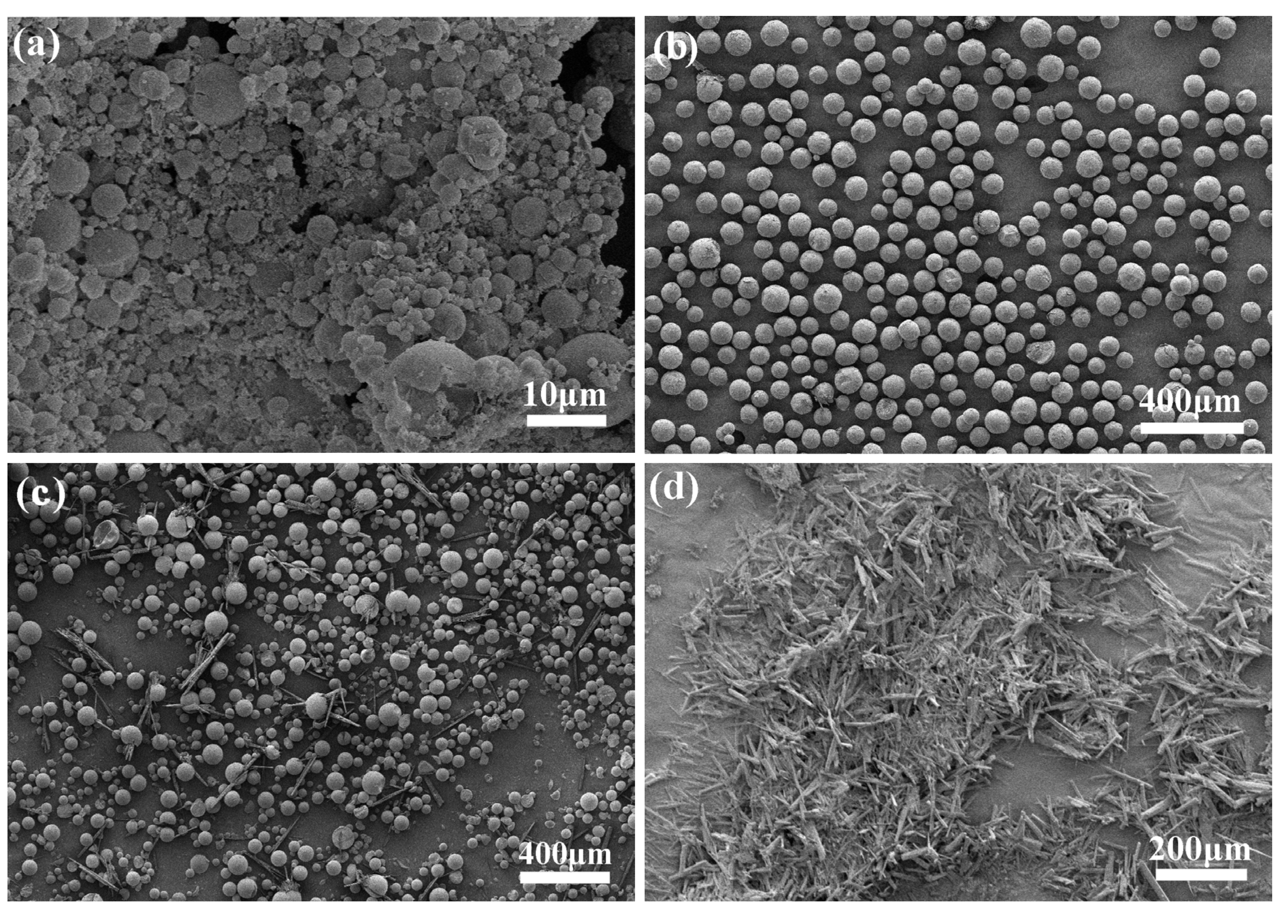


Fig. 1. SEM images of the outcome of the interfacial polymerization of TEOS/APTS bicomponent precursor on the W/O interface with different volume ratio of TEOS to APTS:(a) 20 mL:5 mL, (b) 15 mL:10 mL, (c) 10 mL:15 mL, and (d) 5 mL:20 mL.

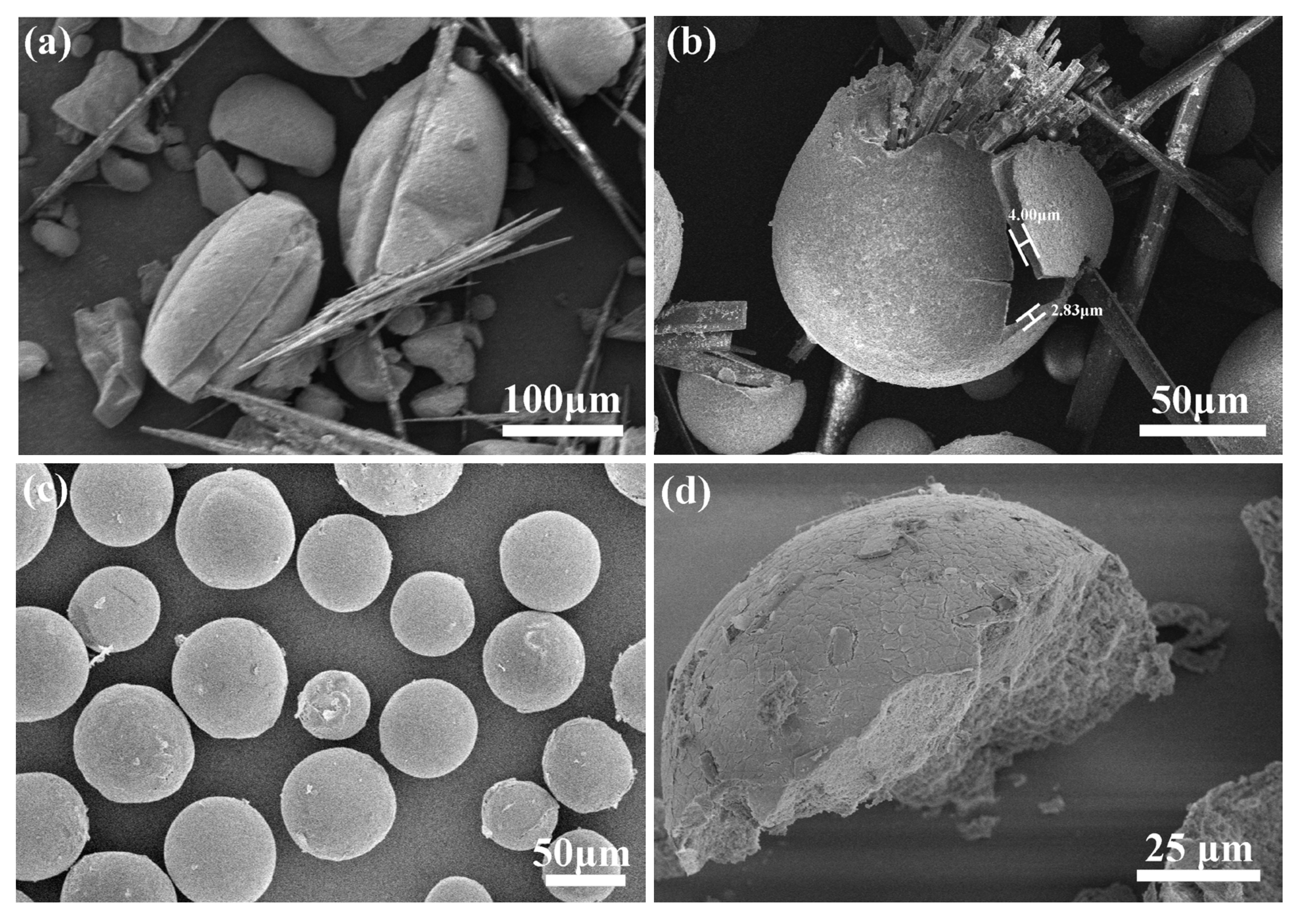
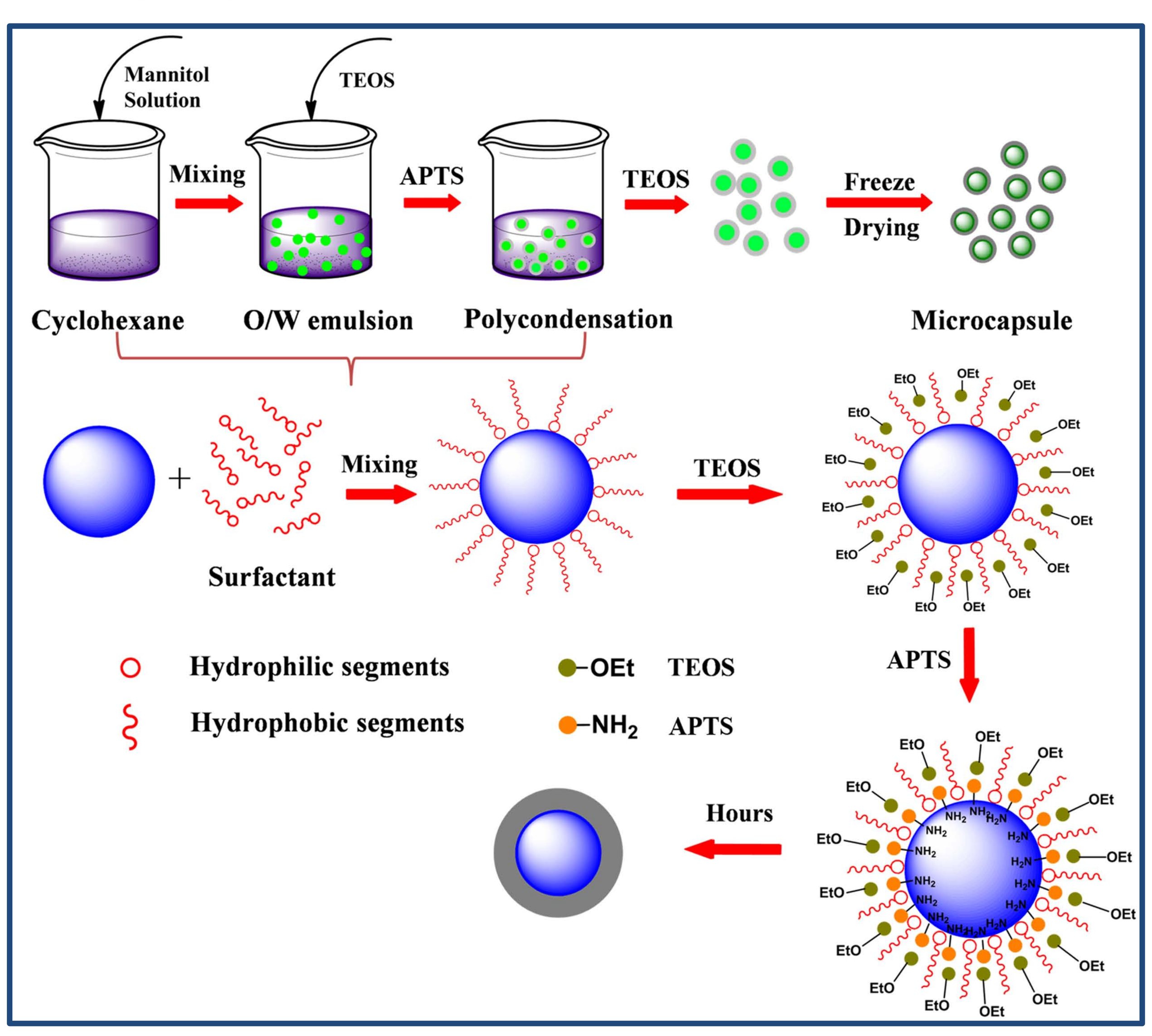


Fig. 2. SEM images of the Mannitol@Silica core/shell capsules produced with different reaction time (a) 2 h, (b) 3 h, (c) 5 h, and (d) 12 h.



Scheme 1. Schematic diagram of the preparing process for Mannitol@Silica core/shell capsules.

NH_2 of APTS was ionized to NH_3^+ in the water near the interface and a thin ionized water layer coupled with OH− formed, which is suitable for hydrolysis and polycondensation of the precursors. A thin shell emerges at the W/O interface because of quick hydrolysis and polycondensation of APTS. Then, both TEOS and APTS continuously diffuse into the water phase across the interface and the thickness of SiO2shell on the surface of the aqueous droplets increased. Finally, the capsules with aqueous core can be obtained and the water was evaporated by freeze-drying.

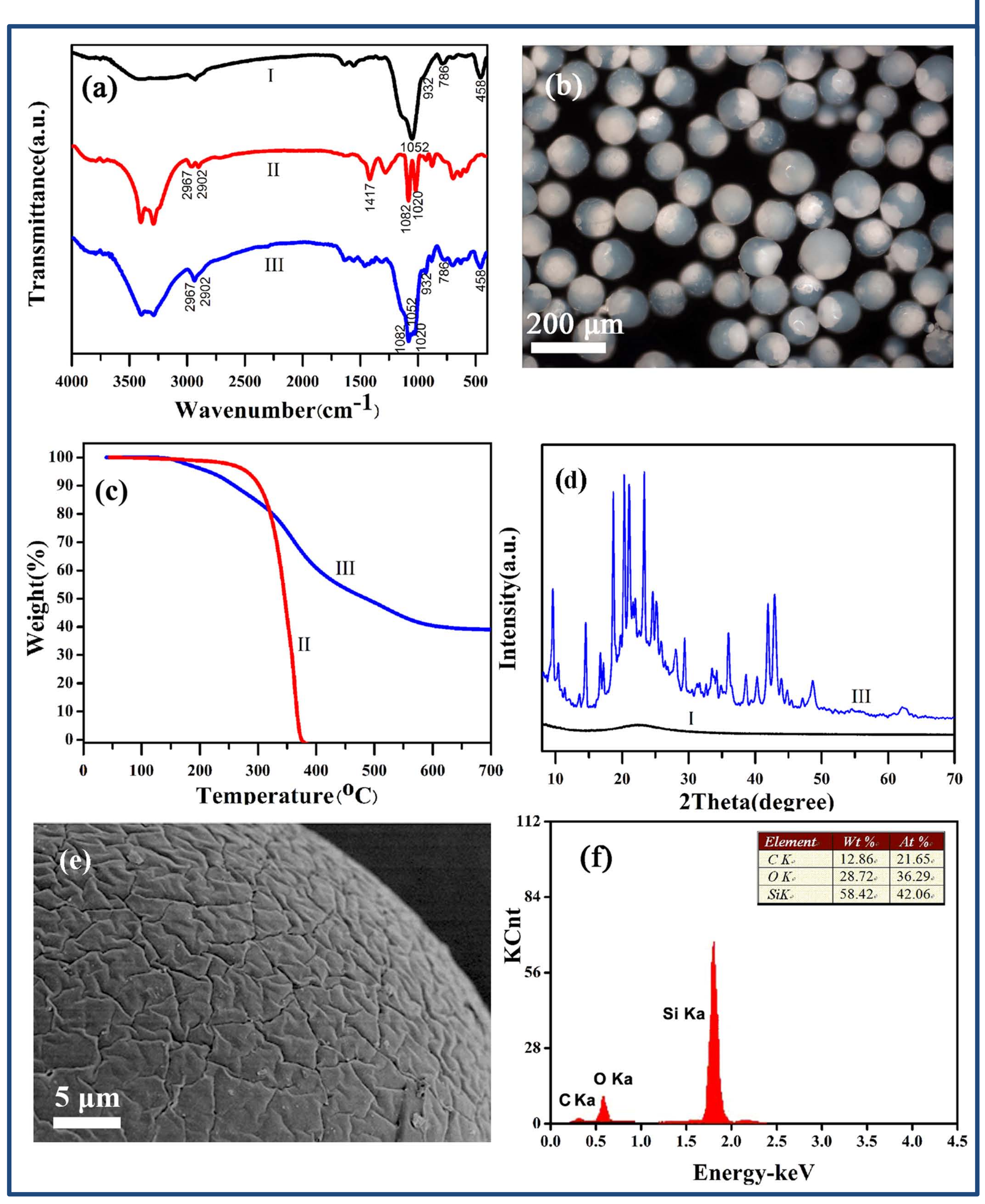


Fig. 3. Characterization of (I) pure SiO_2, (II) Mannitol, (III) Mannitol@Silica capsule: (a) FT-IR spectra; (b) polarizing micrograph photograph; (c) TGA curves; (d) XRD patterns;(e) SEM image of capsule interface; (f) EDS spectra.

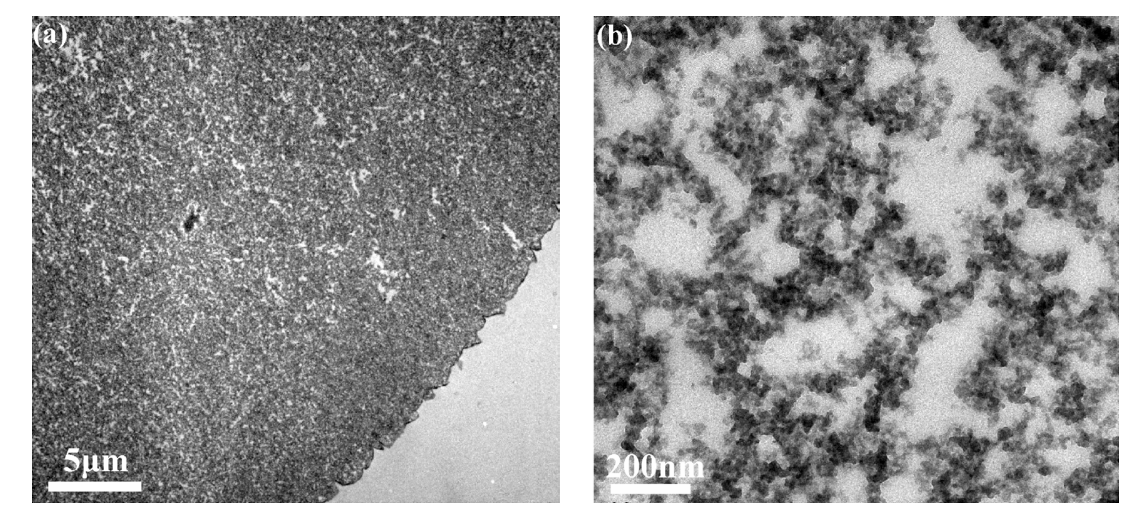
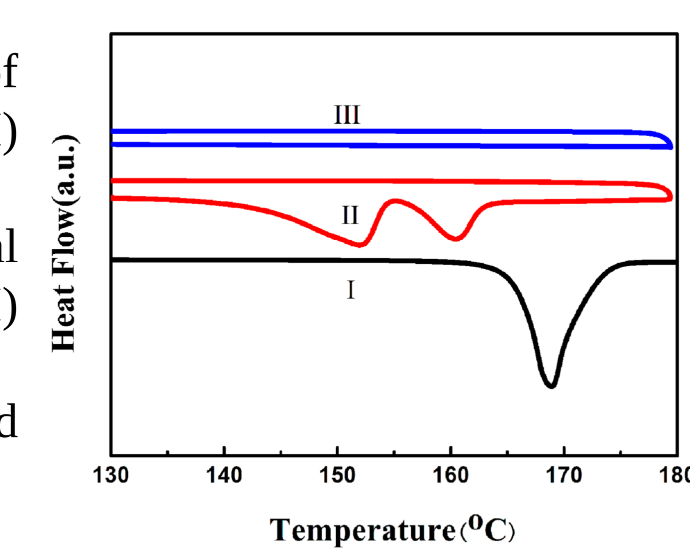
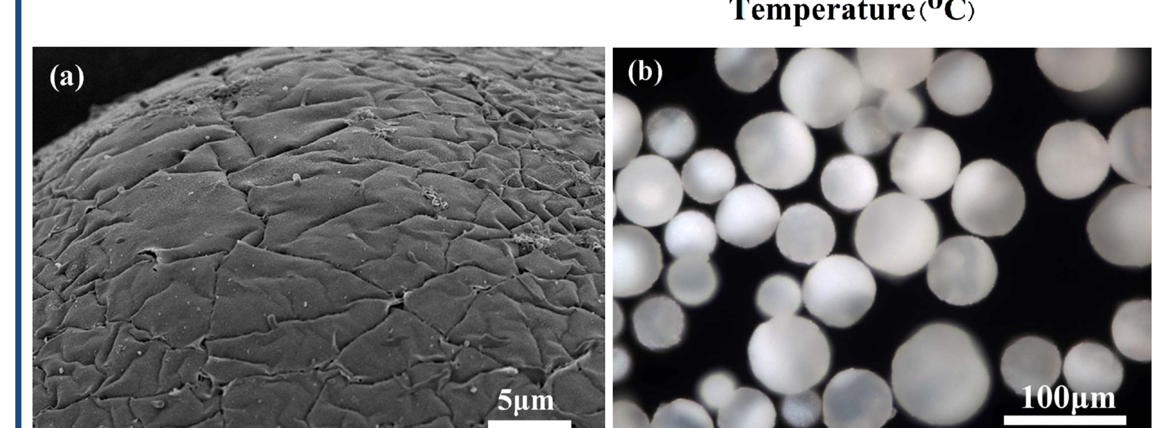


Fig. 4. TEM images of the ultrathin section of the Mannitol@Silica core/shell. (a) Low magnification image of the cross section of the capsule; (b) high magnification images of the inner part of the capsule, showing a porous structure.

Fig. 5. DSC curve of (I) mannitol,(II) Mannitol@Silica capsule (first thermal cycle), and (III) Mannitol@Silica capsule (second thermal cycle).





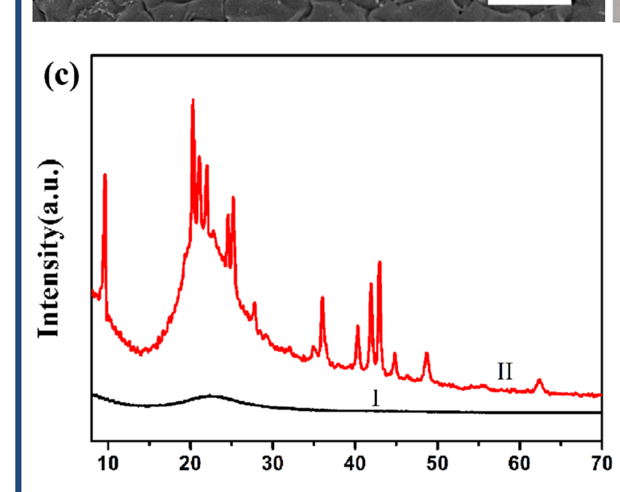


Fig. 6. Characterization of Mannitol@Silica capsule after first thermal cycle: (a) SEM of capsule surface; (b) Polarizing micrograph photograph; (c) XRD patterns for pure SiO2 (I) and Mannitol@Silica capsule (II).

Conclusions

The study provided a feasible route to enwrap organic PCMs with phase change temperature higher than 100◦C into the silica shell tightly, further, it also gave contributions to the understanding of the formation of aqueous core/silica shell microcapsules and helped to explain the formation of the aqueous core capsule with a thick shell.

References

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