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Deformation and Durability Control of Microcapsules for Electrophoretic Display System

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Microcapsules of durability and deformability along with excellent transparency, which was designed for the application of electrophoretic display system, have been synthesized. Microcapsules were synthesized by two-step condensation polymerization using melamine-formaldehyde (MF) resin. In this work, durability and flexibility of MF microcapsules activated by acid catalyst were characterized by compression test accompanied by visual inspection. Microcapsules of MF-AA (using acetic acid as acid catalyst) were rapidly ruptured at force 0.8 mN, while those of MF-MA (using maleic anhydride as acid catalyst) were highly deformed to show a permanent failure at 3.8 mN. Furthermore MF-MA microcapsule showed extremely higher deformation ratio than MF-AA, implying that the maleic anhydride activated system would be much more durable and flexible than the MF system employing AA.

Keywords: deformation; durability; electrophoretic display system; melamine-formaldehyde; microcapsule

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INTRODUCTION

Recently, paper-like display technology (that is, e-paper) has received a great deal of attention, because of its advantages, for example, facility to read and write, thinness and flexibility, high resolution and wide viewing angle, rewritable display of information, wireless adaptability [1–3]. To date, various techniques for the electronic paper application such microencapsulated electrophoretic display, twisting ball display, eletrochromic display and eletrowetting display have been reported [4]. Among them, fabrication of microcapsule-type electrophoretic display has been intensively examined through years because of its interesting properties such as high contrast ratio, lightweight, wide-angle view along with low energy consumption [5–7]. Microcapsules must possess impermeability, transparency, flexibility, durability, chemically inert for electronic paper. It is important that wall materials of the microcapsule should be required not only durable enough to keep its internal phase but also flexible enough to bent easily without breaking. In this study, microcapsules of melamine-formaldehyde (MF) have improved durability and flexibility [8–10]. For the stability during microcapsulation process, the choice of emulsifier is very important, with suitable pH after dissolving and good adsorption into the wall materials.

EXPERIMENTAL

Synthesis of Melamine-Formaldehyde Microcapsules

Microcapsules including dielectric medium and electrophoretic nanoparticles were synthesized using melamine-formaldehyde with acid catalyst. The melamine-formaldehyde (MF) prepolymer with 1:4 molar ratio of melamine (Aldrich) and formaldehyde (37% aqueous solution, Aldrich) were mixed at 70°C, 350 rpm for 30 min. The initial pH of the aqueous phase was adjusted to 8 by adding 10% Na_2CO_3 aqueous solution. After that, MF prepolymer was added into the emulsion including mixture oil. For the polycondensation of MF prepolymer surrounding the emulsion, pH of the emulsion solution was shifted to acid condition ($pH = 4 \sim 5$) using acetic acid (or maleic anhydride). MF microcapsules were further stabilized with polyvinyl alcohols (Mw 100,000, Aldrich) aqueous solution as colloidal stabilizer at 70°C, 350 rpm for 2 hr.

Characterization

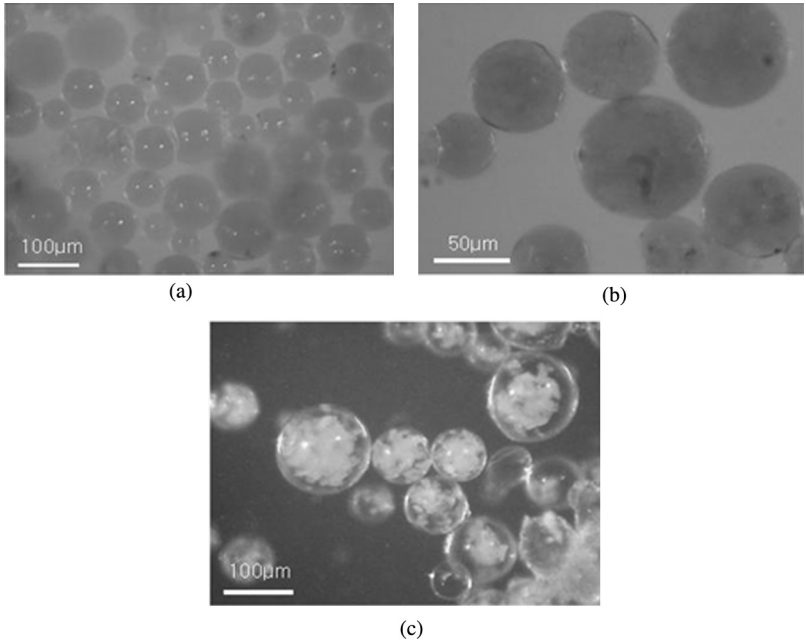
The shape and the wall thickness of MF-AA microcapsules were observed by optical microscope (OM) and scanning electron microscopy

(SEM, Hitachi S-2500C). Samples were sputtered with a thin layer ($\sim$10 nm) of gold- palladium to prevent charging under the electron beam.

Mechanical properties of MF microcapsule were examined on Micro Compression Testing Machine with imaging equipment, which was used with crosshead speed of 2.23 mN/sec. The probe had a diameter of 50 μm and was positioned perpendicular to the bottom of a chamber. The microcapsules were dried in the chamber and observed through side-view cameras. Deformation data from compression test of individual, at least, ten microcapsules were collected and averaged [11,12].

RESULTS AND DISCUSSION

Microcapsules were prepared via conventional *in-situ* polycondensation of MF resin. As found in Figure 1, monodisperse microcapsules of $\sim$100 μm diameter bearing electrophoretic nanoparticles inside were successfully prepared from MF prepolymer and acid catalyst. SEM images in Figure 2 showed that the microcapsule wall was very thin, which is favorable to identify electrophoretic motion of inside



(a) (b) (c)

FIGURE 1 OM images of (a) MF-AA microcapsules, (b) MF-MA microcapsules, (c) MF-MA microcapsules showing electrophoretic nanoparticles inside.

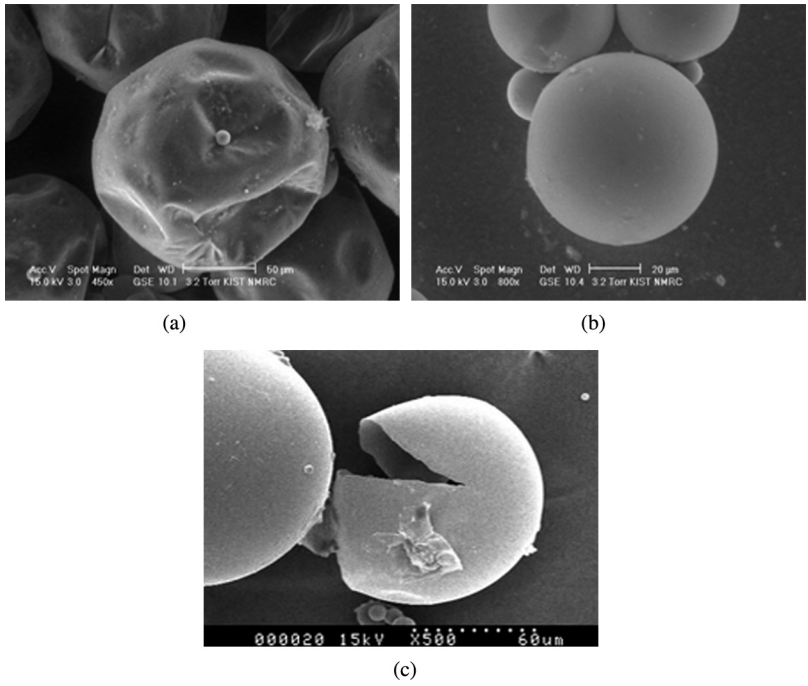


FIGURE 2 SEM images of (a) MF-AA microcapsules and (b) MF-MA microcapsules showing electrophoretic nanoparticles inside, (c) ruptured MF-MA microcapsules.

nanoparticles without loss of incident and scattered light. Since thin walled capsules, however, may happen to easily meet a catastrophic failure, it is essential to examine the mechanical stability of the microcapsules and a compression test under visual inspection was conducted. In the compression test, several tens of microcapsules of comparable diameter were chosen via visual inspection and each individual microcapsule was compressively loaded by a microprobe of MCT. The result of compression test was presented in Figure 3, where A and B are the intersection points of the two tangential lines for MF-AA and MF-MA microcapsules, respectively, which are suggested to represent a rupture point. As shown there, a rupture of MF-AA microcapsule rapidly occurred even under small compressive force and deformation (5% deformation ratio at 0.8 mN force), evidencing that the MF-AA microcapsules are highly brittle. On the other hand, when AA as acid catalyst was replaced by MA, the deformation lasted much longer up to 15% of deformation ratio and the bursting force was five

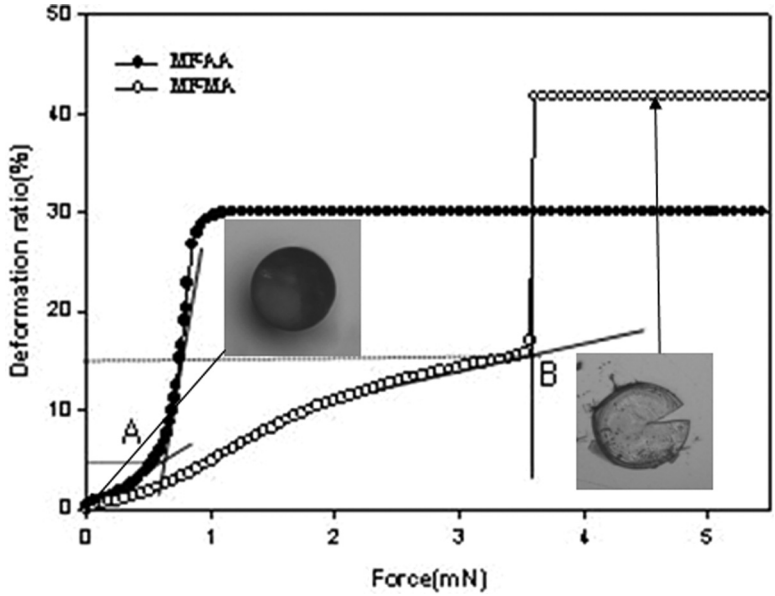


FIGURE 3 Change of deformation ratio of MF-AA and MF-MA microcapsules in a function of compressive force. The insert represents a morphology change of microcapsule before and after rupture by compression.

times as high as that of MF-AA microcapsule, implying the addition of MA induced large increase of deformability and durability. The main role of acid catalyst in the reaction of MF prepolymer is interchain reaction, leading to a network structure. It has been well known that AA induces very rapid crosslinking reaction. Abrupt increase of crosslink density which severely reduce the extent of swelling usually decrease polymer reactivity and limit the maximum conversion by altering reaction medium and diffusion rate within the polymer. Therefore, MF activated by AA resulted in network of low molecular weight/crosslinking density, and a brittle nature was obtained. On the other hand, the interchain reaction of MF prepolymer by MA inevitably occur much sluggish because of lower acidity and amphiphilic nature of MA, resulting in long lasting and high conversion of crosslinking reaction. Therefore, MF network of higher molecular weight along with crosslinking density, induced high durability and deformation. Based on these finding, it can be concluded that acid catalyst is capable of affecting the formation of capsule wall, and the MF-MA microcapsules of large deformability and durability along with excellent transparency are appropriate for electrophoretic display system.

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