Trends in Microencapsulation Research[†]

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Abstract

A microcapsule is a tiny capsule and its preparation procedure, called microencapsulation, can endow various traits to the core material in order to add secondary functions and/or compensate for shortcomings. This paper first gives a general overview of microencapsulation technology and subsequent sections cover topics of microencapsulation research, including application of microcapsules to information and image technologies for microparticle-based paper-like display systems, a use that has attracted considerable attention recently. The research results of the project conducted by the Japan Chemical Innovation Institute under the commission of NEDO, as one of the projects of the METI Nanotechnology Program to develop microparticle-based paper-like display systems, are also introduced.

1. Introduction

Microencapsulation is a technique to prepare tiny packaged materials called microcapsules that have many interesting features. This technique has been employed in a diverse range of fields from chemicals and pharmaceuticals to cosmetics and printing. For this reason widespread interest has developed in microencapsulation technology.

The first industrial product employing microencapsulation was carbonless copy paper developed by Green and Schleicher in the 1950s. The microcapsules used in it were prepared by complex coacervation of gelatin and gum arabic [1]. To this day, carbonless copy paper is one of the most significant products to utilize microencapsulation technology, and is still produced commercially. The technologies developed for carbonless copy paper have led to the development of various microcapsule products in recent years.

Further function integration in microcapsules is essential to make products with excellent properties. Likewise, strategies should be considered to make smaller microcapsules with thinner membranes. This direction of microencapsulation research is suited to current needs and matches the features of nanotechnology efforts initiated in the United States of America in 2000.

We have developed, so far, microcapsules with functions for separation and purification [2-7], controlled release of drugs [8-20], and the triboelectric property required by toner microparticles [21, 22]. Hollow microcapsules and microcapsules enclosing microorganisms have also been prepared [23, 24]. In this paper, after an introduction to microcapsules and microencapsulation, special attention is paid to research on a type of microparticle-based paper-like display, called the microencapsulated electrophoretic display system [25-27].

2. General features of microcapsules

Microcapsules are tiny microparticles with diameters in the range of nanometers or millimeters that consist of core materials and covering membranes (sometimes also called walls). The most significant feature of microcapsules is their microscopic size that allows for a huge surface or interface area. Through selection of the composition materials (core material and membrane), we can endow microcapsules with a variety of functions. Generally, membrane materials

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are chosen in order to pronounce the effects of microencapsulation. Therefore, not only synthetic and natural polymers but also lipids and inorganic materials are used for the preparation of microcapsules.

Some well-known products prepared using microencapsulation techniques are artificial salmon roe and carbonless copy paper. Microcapsules can be classified into three basic categories according to their morphology as mono-cored, poly-cored, and matrix types, as shown in **Figure 1**. Morphological control is important and much effort has been given to controlling internal structures, which largely depend on the protocol and the microencapsulation methods employed.

Microcapsules have a number of interesting advantages and the main reasons for microencapsulation can be exemplified as (1) controlled release of encapsulating drugs, (2) protection of the encapsulated materials against oxidation or deactivation due to reaction in the environment, (3) masking of odor and/or taste of encapsulating materials, (4) isolation of encapsulating materials from undesirable phenomena, and (5) easy handling as powder-like materials.

More detailed features of microcapsules are summarized in books by Gutcho [28] and Arshady [29], and in review papers by Makino [30], Arshady [31], Kondo [32], Hatate, et al. [33], and Yoshizawa [34-36].

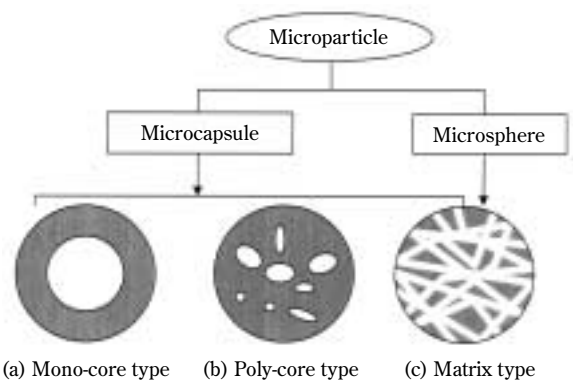


Fig. 1 Classification of microparticles from their morphology

3. Microcapsules in the information and imaging fields

The first successful application of microencapsulation technology in the information and imaging fields was the carbonless copy paper developed by the National Cash Register Company to solve the problems of solvent evaporation and diffusion of ink into paper substrate. Carbonless copy paper has become a $5.5 billion business worldwide.

In the 1960s, microencapsulation of cholesteric liquid crystal by complex coacervation of gelatin and acacia was reported to produce a thermosensitive display material. J. L. Fergason developed nematic curvilinear aligned phase (NCAP), a liquid crystal display system using microencapsulation of nematic liquid crystal [37]. Encapsulation technology has provided the enlargement of display areas and wider viewing angles.

The functions endowed by microencapsulation in the development of carbonless copy paper and NCAP can be summarized as the protection of core materials from deterioration such as oxidation and easy handling and treatment as powder-like substances.

4. Why a microparticle-based display is desirable

Creation of a paper-like display with low power consumption has been an ambition of display researchers for many years. To realize this beneficial technology, many researchers have tried to make microparticle-based display systems. An electrophoretic image display system and a rotating bichromal microspheres system are representative of trials to create paper-like display systems [37, 38]. Particle-based display systems are attractive for their optical and electronic properties. These beneficial properties result from the highly scattering and absorbing microparticles that contain pigments like titanium dioxide and carbon black.

Figure 2 shows a schematic illustration of the rotating bichromal microspheres system (a) and the electrophoretic image display system (b). The rotating bichromal system called Gyricon has been developed by Nicholas K. Sheridon at the Xerox Palo Alto Research Center. In the rotating bichromal system, a microsphere with white and black hemispherical surfaces is in each cavity of a silicon polymer film that is placed between a transparent top electrode and a bottom electrode. The principle of the rotating bichromal microspheres system is the rotation of the microspheres to correspond to the addressed external electric field. That is, the positively-charged black hemispherical surface rotates toward the negatively-charged top electrode. In contrast, at the same moment, the negatively-charged white hemispherical surface has an electrostatic affinity to the positively-charged electrode. Thus, the color change is attributable to the

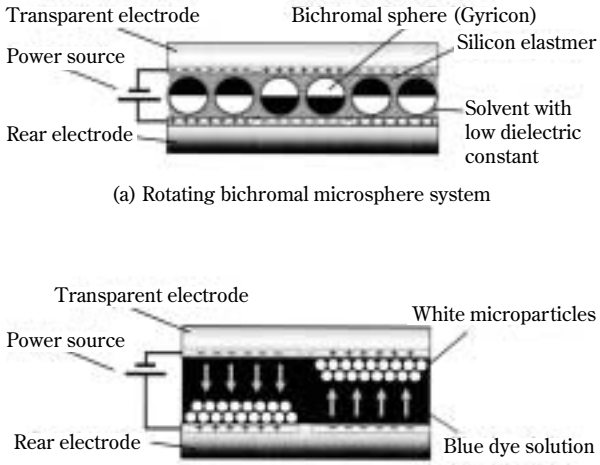


Fig. 2 Schematic illustration of the rotating bichromal microsphere system (a) and the electrophoretic image display system (b)

rotation of bichromal microspheres. On the other hand, the fundamental aspect in the electrophoretic image display systems is caused by electrophoretic migration of charged microparticles in a fluid with a low dielectric constant. This system was presented by Isao Ota of Matsushita Electric Industrial Co., Ltd.

5. Microencapsulated electrophoretic display system

Despite many attractive features, however, the microparticle-based display systems mentioned above suffer from some shortcomings, including difficulty achieving perfect rotation (in the case of the rotating bichromal microspheres system) and short lifetime due to coagulation and agglomeration caused by colloidal instability (in the case of the electrophoretic image display system).

I had a chance to conduct research on paper-like display systems at the Media Laboratory of Massachusetts Institute of Technology with Prof. Joseph Jacobson, a key person in the development of paper-like display systems employing electrophoresis of microparticles. To overcome the previously mentioned shortcomings, we created E-ink, a new display system utilizing microencapsulation techniques with a fusion of chemistry, physics, electronics and other technologies [25]. The schematic illustration is shown in **Figure 3**. As this figure shows, the structure of E-ink is simple and each microcapsule, with about the diameter of a human hair, contains millions of tiny pigment

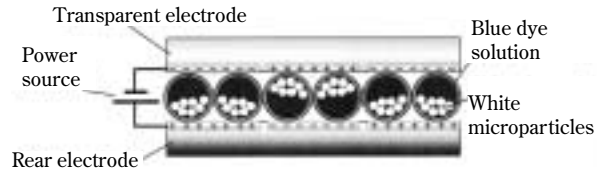


Fig. 3 Schematic illustration of the microencapsulated electrophoretic display system

microparticles that are well dispersed in an organic solution with a low dielectric constant. The microencapsulated electrophoretic display system prototype that we proposed in the journal Nature in 1998 was a one-particle system. Microcapsules contained one type of charged white microparticles dispersed in a blue dye solution. At present, we have developed a two-particle system that uses two kinds of microparticles (one white, one black) enclosed in a microcapsule. In the two-particle system, the dispersion medium in microcapsules is a transparent fluid.

When an electric field is applied between microcapsules, the microparticles move in the low dielectric constant solution toward the oppositely charged electrode in the phenomenon of electrophoretic migration. If the top transparent electrode is positively charged, white microparticles with negative charges should move toward the top electrode, making the surface appear white at that spot. At the same time, an opposite electric field pulls the black particles to the bottom of the microcapsules. By reversing this process, the black microparticles appear at the top of the microcapsules, which makes the surface become black at that spot. This is how microencapsulated ink (E-ink) forms letters and pictures on the display.

Figure 4 presents microphotographs of the microencapsulated electrophoretic display system at different magnifications, in which the letter 'k' was electronically induced. The prepared microcapsules were dispersed in UV-curable polymer and covered with a transparent electrode. The microcapsules were sieved to have an average diameter of about 40 mm. This diameter allows for a resolution of up to 600 dpi.

The contrast ratio and the reflectance of the prototype were 7:1 and 35%, respectively. Using the same measurement system, a newspaper had 5:1 contrast ratio and 55% reflectance. Furthermore, we observed the microencapsulated electrophoretic display system had bistability (image storage performance) for several months. Bistability is an important performance trait of rewritable paper and paper-like displays,

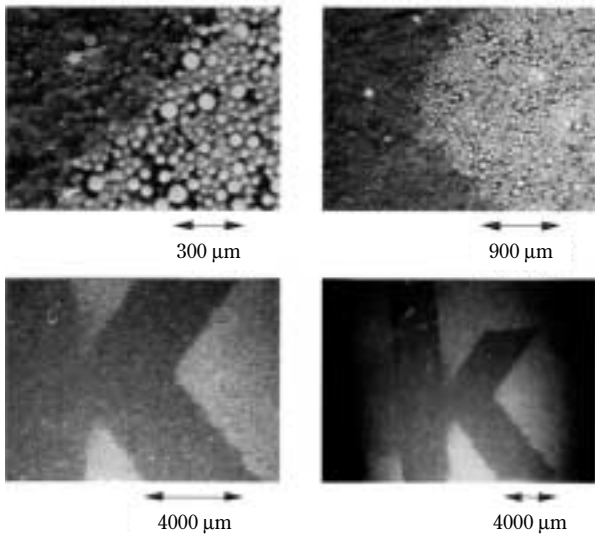


Fig. 4 Photograph of display image of microencapsulated electrophoretic display system

Fig. 5 Photograph of first commercial display with electrophoretic ink by E-ink's Immedia Technology in J.C. Penney stores

because it allows image retention even when the power is turned off. This trait allows for both portability and low-power consumption.

For electrophoretic image display systems, the remaining problems had been particle clustering, agglomeration and lateral migration. The benefit of microencapsulation in this case is the isolation of electrophoretic dispersion in discrete compartments. In other words, agglomeration of electrophoretic dispersion in a microcapsules has no influence on electrophoretic dispersion in neighboring microcapsules.

Prof. Joseph Jacobson and other cofounders established E Ink Corporation in Cambridge, Massachusetts in 1997 to produce a next-generation display which has many of the advantages of paper-like media, including the ability to access information anytime, anywhere [40]. This technology can be said to be the future of paper. E Ink advertises that paper-like displays have the following advantages. (1) *Paper-like readability* means that the display can be read in dim light with the same contrast as in bright sunlight, and at any angle without loss of contrast. (2) *Ultra-low power consumption* results from the performance of bistability, which allows a fixed image to be kept even when the power is turned off. (3) *Exceptional portability* because of the reduced use of both polarizer and glass compared to liquid crystal display systems.

E ink Corporation first demonstrated these systems as store signs under the brand name of Immedia in 1999. They were tested at various places including J. C. Penny retailing stores (**Figure 5**), and by the Arizona Republic newspaper for headline displays [27].

E ink Corporation recently announced that they have sold their first electronic paper display module for application in Sony's new e-Book reader, LIBRIé (**Figure 6**), produced in cooperation with Toppan Printing, Royal Philips Electronics and Sony Corporation [41-43]. They claim that more than 10,000 pages

Fig. 6 Photograph of Sony Corporation LIBRIé that uses the electrophoretic ink technology of E-ink Corporation

can be read with just four AAA Alkaline batteries. In fact, the LIBRIé is lightweight and displays images with a white/black contrast that is as high as a newspaper. The LIBRIé is reported to have reflectance of 50%, contrast ratio of 15:1, power requirements of 15 V, and a response time of 40 ms.

This milestone in microencapsulated electrophoretic display system development is the culmination of fundamental investigations of microparticle science and engineering, which have been conducted to make microcapsules with homogeneously transparent membranes, and microparticles enclosing pigment with superior dispersion stability, as well as the development of electronic parts.

6. Full color rewritable paper using functional capsules project

As mentioned above, the electrophoretic image display system shown in **Fig. 2** was developed by a company in Japan. A paper-like display system employing microparticle technology has also gathered much attention from companies in Japan in the fields of electronics and information. For example, a toner-type display system [44], an in-plain-type display system [45], a liquid powder display system [46] and a Gyricon-like cylindrical display system [47] have been developed by Fuji Xerox Co., Ltd., Canon Inc., Bridgestone Corporation, and Oji paper Co., Ltd., respectively. Powder engineers have also achieved marvelous results with the development of liquid powder that is 10 times smaller in bulk density (**Figure 7**). This allows for quicker response to polar change with a time of 0.2 ms, which is one-tenth of that of liquid crystal display systems.

Japan is one of the leading countries in microencapsulation technology. Creation of functional materials and achievement of high productivity are the final subjects of technology fusion with microencapsulation fabrication technology. The Japanese Ministry of Economy, Trade and Industry (METI) has created a national project concerning paper-like displays that employ microencapsulation, called the Full Color Rewritable Paper Using Functional Capsules Project. This project is conducted by JCII under the Commission of NEDO, as one of the projects in the METI Nanotechnology Program. In 2004, this project is also included as a special project to reinvigorate the economy in a program called Focus 21.

The paramount objective of this project is the development of a paper-like display prototype. The following three subjects have been identified as requiring research in order to achieve this goal.

(1) Encapsulation technology
(2) Nano-functional particle surface physical-properties control technology
(3) Display material and functional evaluation technology using the encapsulation technology of nano functional particles

The goals of encapsulation technology research are the development of a microencapsulation procedure that achieves high efficiency for functional nanoparticle encapsulation, and the determination of factors that control microcapsule diameter, membrane thickness and membrane properties. Representative results are shown in **Figures 8～10**.

Fig. 8 shows a photograph of polyurea microcapsules that were prepared by interfacial polymerization

Fig. 7 Photograph of typical liquid powder developed by Bridgestone Corporation

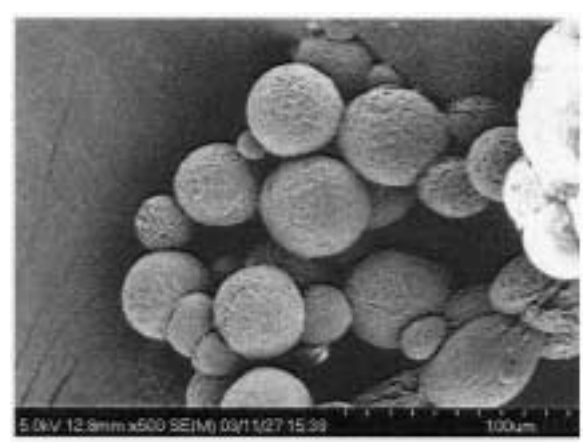


Fig. 8 SEM photograph of microcapsules prepared by interfacial *in situ* polymerization

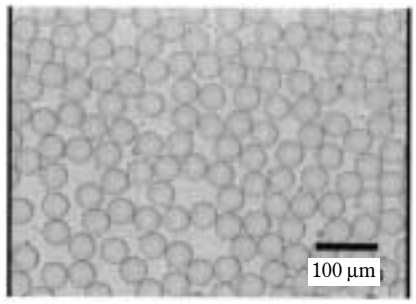
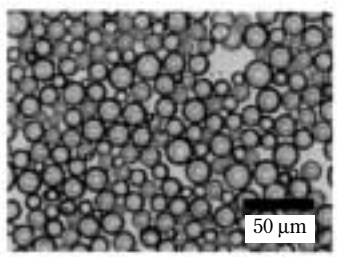
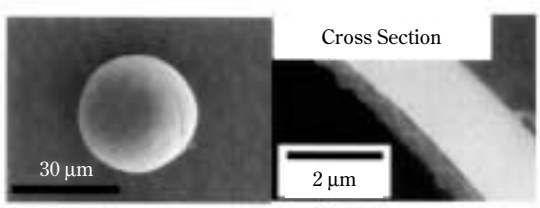


Fig. 9 Monodispersed microcapsules prepared by interfacial polyaddition after emulsification by the ink-jet method



(a) O/W emulsion



(b) Microcapsule

Fig. 10 Photographs of O/W emulsion and microcapsules prepared by phase-inversion emulsification and the subsequent hardening

between isocyanate groups of the acrylic resin and diamine substances. As the figure shows, the prepared microcapsules were found to be monodispersed satisfactorily because emulsification using an ink-jet apparatus provided fairly evenly monodispersed oil droplets. Membrane thickness can be controlled in a range of up to 800 nm.

Fig. 9 is an SEM photograph of microcapsules prepared by interfacial radical polymerization (interfacial *in situ* polymerization). In conventional *in situ* polymerization, the polymerization reaction occurs in liquid droplets with dissolved monomers and initiators. Therefore, tiny microparticles dispersed in liquid droplets can be embedded in a polymer membrane to make a microencapsulated electrophoretic display system. By modifying the procedure with a reactive surfactant, the reaction site for radical polymerization of acrylonitrile was successfully localized at the O/W interface.

Phase-inversion emulsification was adopted to make monodispersed O/W emulsion. A photograph of a typical O/W emulsion is shown in **Fig. 10a**. At present, we can control monodispersion of prepared O/W emulsions by adjusting process parameters, including the polymer concentration, the degree of neutralization, and the rate of water addition. The prepared microcapsules are shown in **Fig. 10b**. Subsequent hardening with epoxy resin was useful to make the microcapsule membrane.

7. Systematization of capsule technology

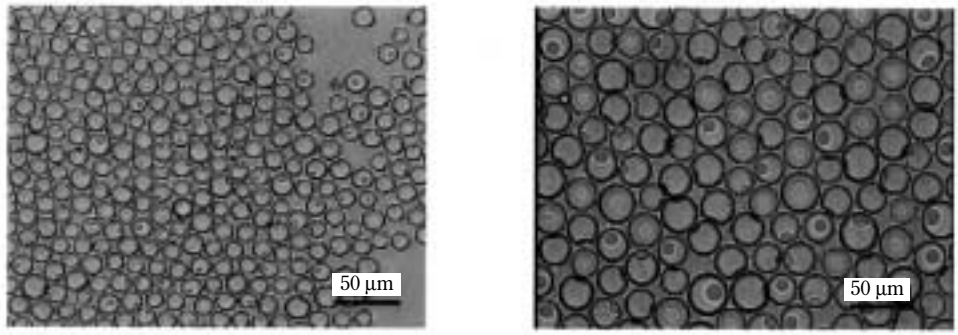
In this project, 6 research groups have joined forces and worked to systematize the knowledge of microencapsulation methods, including phase separation, solvent extraction, coacervation, *in situ* polymerization, and interfacial polymerization.

As an example of the results of these capsule technology systematization efforts, the microencapsulation by phase separation procedure is introduced in some detail below.

One objective was to establish a technique to control properties of crosslinked polyamino resin microcapsules prepared by the phase separation method. In order to develop a technique to control microcapsule diameter, membrane thickness and membrane morphology, the following investigations were carried out: (1) controlling the diameter of a microcapsule using the Shirasu Porous Glass (SPG) membrane emulsification technique, (2) controlling microcapsule membrane thickness with a mixture of two kinds of water-soluble polymeric surfactants.

(1) *Investigation on controlling microcapsule diameter*

Monodispersed emulsions having about 10% CV (coefficient of variation) were successfully prepared with the SPG membrane emulsification technique. Diameters of the prepared O/W emulsion were in proportion to the pore diameters of the SPG membrane. The diameters of the emulsion were five times as large as the pore diameters. Preparation of crosslinked polymelamine microcapsules was attempted by using the monodispersed emulsions. Concentration of sodium



Pore diameter of SPG membrane is 2.6 μm　　Pore diameter of SPG membrane is 4.8 μm

Fig. 11 Photographs of crosslinked polymelamine microcapsules prepared with the SPG membrane technique

dodecyl sulfate (SDS), which is a typical emulsifier in SPG membrane emulsification, was found to strongly affect the dispersion stability of the prepared microcapsules. Decreasing the SDS concentration by using the rich top emulsion phase allows monodispersed microcapsules to be prepared. **Figure 11** shows photographs of crosslinked polymelamine microcapsules prepared with the SPG membrane technique. The microcapsule diameter corresponded fairly well with the O/W emulsion diameter. The CV value was also about 10%. Through this investigation, control of microcapsule diameter was achieved in the range of 10−60 μm.

(2) *Investigation on controlling microcapsule membrane thickness*

Control of membrane thickness of crosslinked polyurea microcapsules was investigated by using several kinds of water-soluble polymeric surfactants, which were added in the continuous phase in microcapsule preparation. The relationship between membrane thickness and poly(E-MA) concentration is shown in **Figure 12**. It was found that the membrane thickness is related to the water-soluble polymeric surfactants used. When poly(E-MA), or poly(methyl vinyl ether-alt-maleic anhydride), was used as a water-soluble polymeric surfactant, membrane thickness of the prepared microcapsules were about 100 nm. When poly (acrylic acid) was used, the membrane thickness was about 400 nm. Control of membrane thickness was achieved by using a mixture solution of poly(E-MA) and poly(acrylic acid). **Fig. 12** shows that the membrane thickness could be controlled between 100− 400 nm.

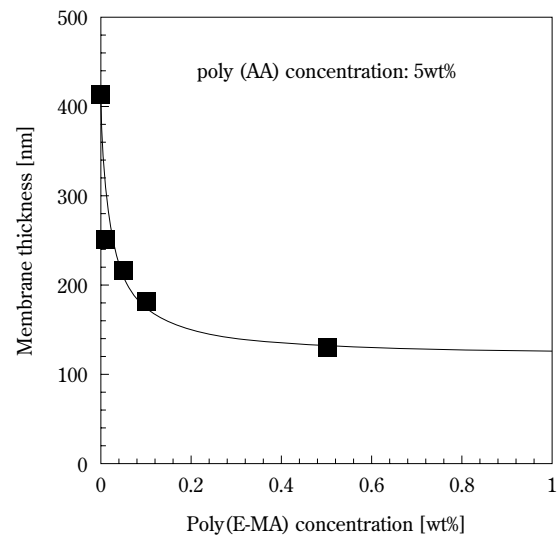


Fig. 12 Relationship between membrane thickness and poly(E-MA) concentration

Future development

This paper has reviewed the recent developments in microencapsulation technology, focusing on the topic of the development of the microencapsulated electrophoretic display system, a microparticle-based paper-like display. Much other research on microencapsulation has been conducted in recent years.

Microcapsules are not raw materials in most cases. Furthermore, microencapsulation is a technique to give raw materials advantageous traits in order to make superior products. This characteristic of the microencapsulation research means that periodically new developments lead to new products worthy of

public notice. The first remarkable product was carbonless copy paper, the second was controlled release of drugs, and at present, paper-like displays are receiving much attention.

In order to make microencapsulation technology more fruitful and to realize the sustainable development of microencapsulation, I believe that fundamental studies and reconsideration of base technologies for various microencapsulation procedures are strongly needed and that cooperation between academic and company researchers should be promoted. Fundamental research should include the elucidation of microcapsule membrane formation mechanisms, quantitative analysis of the effects of process parameters to control microcapsule properties, and the development of new microencapsulation procedures. These investigations would lead to the creation of new products with new functions.

Acknowledgement

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Author's short biography

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Dr. Hidekazu Yoshizawa is a professor of the Department of Environmental Chemistry and Materials at Okayama University. He received his BS (1985) and MS (1987) in Chemical Engineering from Osaka Prefecture University and his PhD (1995) from Kyushu University. He worked at Kagoshima University as Assistant Professor and Associate Professor in the field of microparticle science and technology. In 1997 and 1998, he was a postdoctoral associate at the Media Laboratory, Massachusetts Institute of Technology. He has worked at Okayama University from 1999. He now serves as a sub-leader of the Full Color Rewritable Paper Using Functional Capsules Project. His current research interests are synthesis and characterization of polymer colloids, synthesis and application of biodegradable polymers, and microparticulate drug delivery systems.