

Cosmetic textiles with biological benefits: Gelatin microcapsules containing Vitamin C

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Abstract. In recent years, textile materials with special applications in the cosmetic field have been developed. A new sector of cosmetic textiles is opened up and several cosmetic textile products are currently available in the market. Microencapsulation technology is an effective technique to control the release properties of active ingredients that prolong the functionality of cosmetic textiles. This study discusses the development of cosmetic textiles and addresses microencapsulation technology with respect to its historical background, significant advantages, microencapsulation methods and recent applications in the textile industry. Gelatin microcapsules containing vitamin C were prepared using emulsion hardening technique. Both the optical microscopy and scanning electron microscopy demonstrated that the newly developed microcapsules were in the form of core-shell spheres with relatively smooth surface. The particle size of microcapsules ranged from 5.0 to 44.1 μm with the average particle size being 24.6 μm . The gelatin microcapsules were proved to be non-cytotoxic based on the research findings of the toxicity studies conducted on human liver and breast cell lines as well as primary bone marrow culture obtained from patient with non-malignant haematological disorder. The gelatin microcapsules were successfully grafted into textile materials for the development of cosmetic textiles.

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1. Introduction

People have always tried to improve their quality of life with positive developments in hygiene and nutrition as a result of rapid development in medical knowledge. The developments nowadays are focused not only on the solutions to medical problems, but also on the possible ways to improve the comfort of application. Textile systems may be useful in this aspect (1).

The typical benefit obtained from the use of textile materials is protection. However, with the growing demand of enhancing beauty with health, customers request that the apparels and home textiles not only have the original basic characteristics such as maintaining warmth and providing comfort, but also carry extra functions including environmental protection, anti-pollution and the most concerned function like health and beauty care in an attempt to provide a more natural and healthier life (1,2).

The breathing capability makes textile materials become a useful basis for *ex vivo* applications (1). A brainstorming idea has been developed to make textile materials useful in the cosmetic field. As people are getting used to wearing textile materials in their daily life, it is also logical to use textile as the possible basis for cosmetic substance delivery system (1). A new terminology, so called 'cosmetic textiles' has now been introduced to the textile industry and it is very optimistic that these products will open up new target groups and sustainable markets (1,3,4).

2. Cosmetic textiles

On contact with the skin and human body, cosmetic textiles are designed to transfer an active substance for cosmetic purposes. One particular example is the transfer of skin-moisturising substances from the cosmetic textiles to human body. The principle is achieved by simply imparting the cosmetic and pharmaceutical ingredients into the fabric of garment so that with the natural movement of body, the skin is slowly freshened and revitalised. In order to achieve the functional effects, microencapsulation technology appears to

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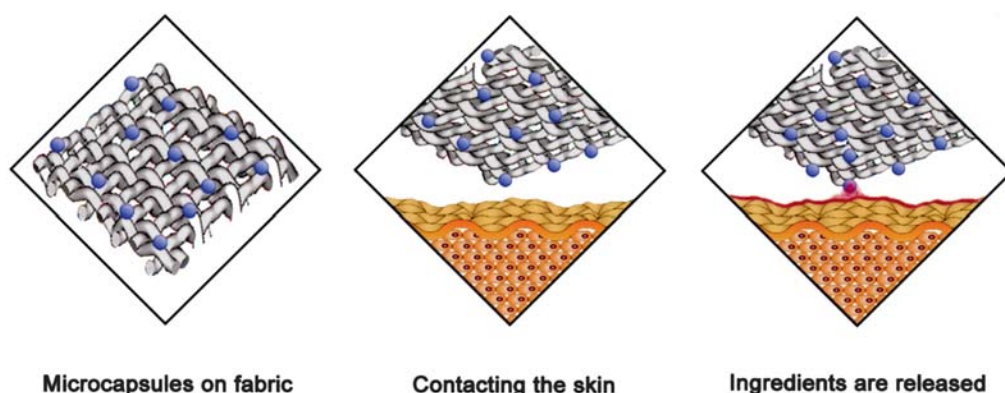


Figure 1. Working mechanism of cosmetic textiles.

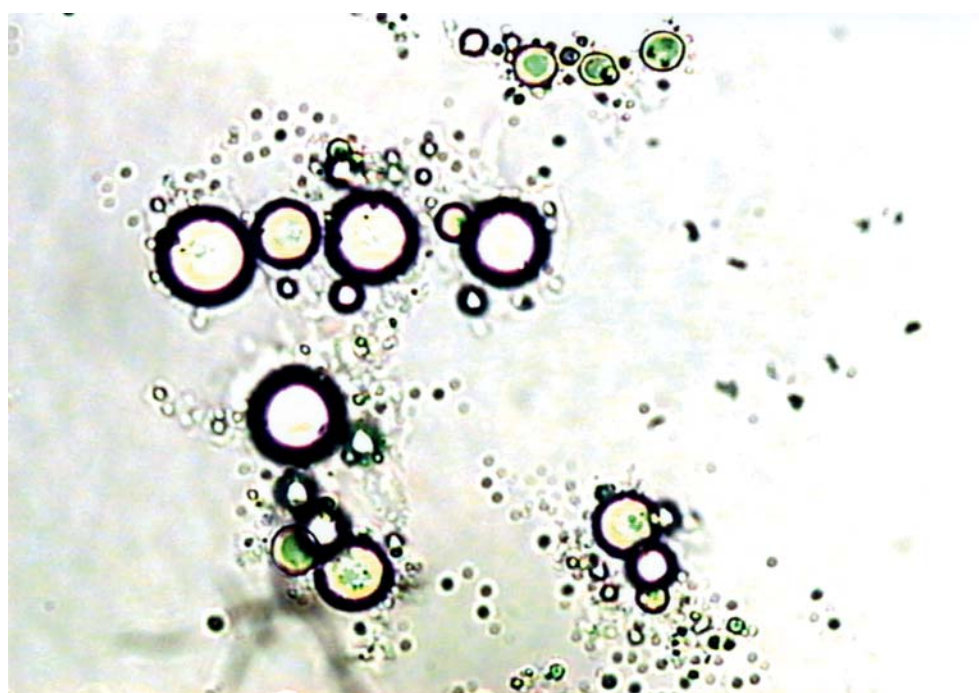


Figure 2. Optical image of chitosan microcapsules containing jojoba oil.

be an alternative way to provide satisfactory performance with increased durability. Fig. 1 illustrates the working mechanism of cosmetic textiles when contacting the skin.

The controlled release properties of the active ingredient with respect to the cosmetic textiles is an important aspect to be studied. Under a slow-release delivery mechanism, the cosmetic substance can be released to human skin in a controlled way to avoid overdose (1,5).

The biocompatibility of the cosmetic substance delivery system is another concern in the development of cosmetic textile. Biocompatibility can be defined as the ability of a material to perform with an appropriate host response in a specific application. The cosmetic substance delivery system of cosmetic textiles should be non-toxic and non-carcinogenic with no interest in the related issues of mutagenicity and teratogenicity. As an *ex vivo* application, the cosmetic textiles should not cause any irritation of skin at the same time of release the active ingredients (1).

In view of the increasing demand in the relevant fields, researchers and textile manufacturers have substantially invested in the cosmetic textiles for research and product development. In general, the events are focused on a) opportunity and limit for cosmetic and wellness applications of textiles, b) possible ways of incorporating active substances in a functional manner with increased durability, c) systematic characterisation model for assessing the cosmetic textiles for safety and d) biological safety and biological benefit to human skin.

3. Microencapsulation

Microencapsulation technology. Microencapsulation technology is now rapidly developing in the field of chemical finishing because of its versatility and flexibility. Microencapsulation is actually a micropackaging technique involving the production of microcapsules which act as



Types of wall material	Examples
Natural	Gelatin, agar, gum, sodium alginate, calcium alginate, dextran, fat and fatty acid starch, chitosan, casemate, stearm, sucrose, and wax
Semi-synthetic	Cellulose acetate, cellulose nitrate, ethylcellulose and hydroxypropylcellulose, methylcellulose, sodium carboxymethylcellulose, hydrogenated tallow, myristyl alcohol, glycerylomon-or dipalmitate, hydrogenated easter oil, glycerylmono-,di, or tristearate, and 12-hydroxystearyl alcohol
Synthetic	Acrylic polymer and copolymer

barrier walls of solids or liquids. The microcapsules are produced by depositing a thin polymer coating on small solid particles or liquid droplets, or on dispersions of solid in liquids. The core contents are released under the controlled conditions to suit a specific purpose. Particles in micro-size prepared by the microencapsulation technique are called microcapsules or microparticles (6-8). Fig. 2 and 3 show the optical image and scanning electron microscope (SEM) image of chitosan microcapsules containing essential oil.

The release mechanism of the core contents vary depending on the selection of wall materials and more importantly, its specific end uses. The core content may be released by friction, pressure, change of temperature, diffusion through the polymer wall, dissolution of the polymer wall coating or by biodegradation (9-11).

Historical background of microencapsulation. The earliest conception of microencapsulation, which carries or holds a core material trapped within a shell material, possibly dates back to 1930s by using spray-drying technique (8). Until 1950s, the first significant application of encapsulation technology was developed by Barrett Green of National Cash Register Company to provide carbonless copy paper using complex coacervation technique. This was employed in a novel printing system which incorporated a colourless dye within the oil phases and coated a second review article sheet with acidic clay (8,12). Since then, the US-based Eurand America company had acquired the rights to subsequently develop and market microencapsulation technology for all new applications (13).

Microencapsulation techniques developed by a number of companies were noted henceforth. This versatile micro-packaging technique has been applied in a wide range of fields including the pharmaceutical, bulk chemical, agricultural, food processing, cosmetic and toiletry industries (6,13). The textile industry has reacted slowly to the possibilities of microencapsulation. It is not until 1990s that a few commercial applications were appearing at the research and development stages (6).

In the 21st century, more commercial applications of microencapsulation in the textile industry can be found, particularly in Western Europe, Japan and North America. The technique is being used to develop textiles with new properties and added value; including climate-control materials, fragrances released fabrics, cosmetic, therapeutic

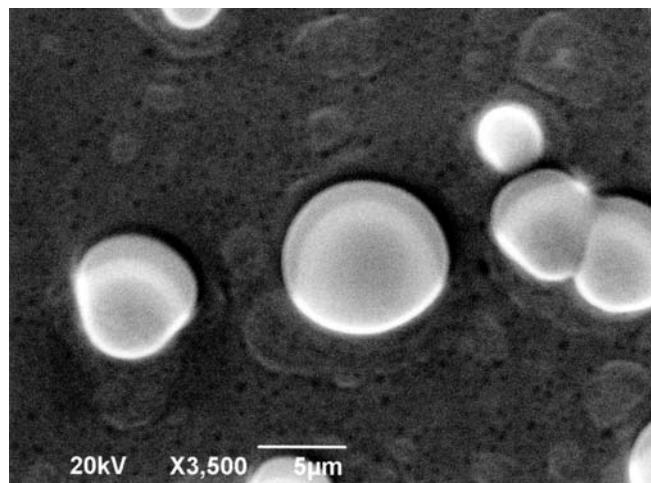


Figure 3. SEM image of chitosan microcapsules containing jojoba oil.

and medical textiles. Textiles and garment manufactures are very optimistic that this novel technology will open up exciting market opportunities and a world of possibilities for consumers (9,14-15).

Structure of microcapsules. Microcapsules are small liquid, solid or aerosol particles surrounded by another substance in order to protect, separate the materials and control the release of core materials. They may be considered as a way of storing materials at the microscopic level (16). Fig. 4 shows the general structure of microcapsule which generally consists of two major components:

i) **Active ingredient.** Active ingredient is the substance that may be in the form of liquid or solid. It is also referred to the core contents, internal phases, active, encapsulate, payload or fill (9).

ii) **Wall shell.** A polymer coating that surrounds the active ingredients which may also be called the wall, shell, external phase, membrane or matrix. It may be natural polymer, semi-synthetic polymer and synthetic polymer (9).

Microcapsules can be made into various forms as shown in Fig. 5, but they are usually in spherical form. Microcapsules of irregular forms are known as a) spherical form with single nucleus, b) special form with multi nucleus, c) indeterminate form and d) indeterminate form with multi nucleus (16).

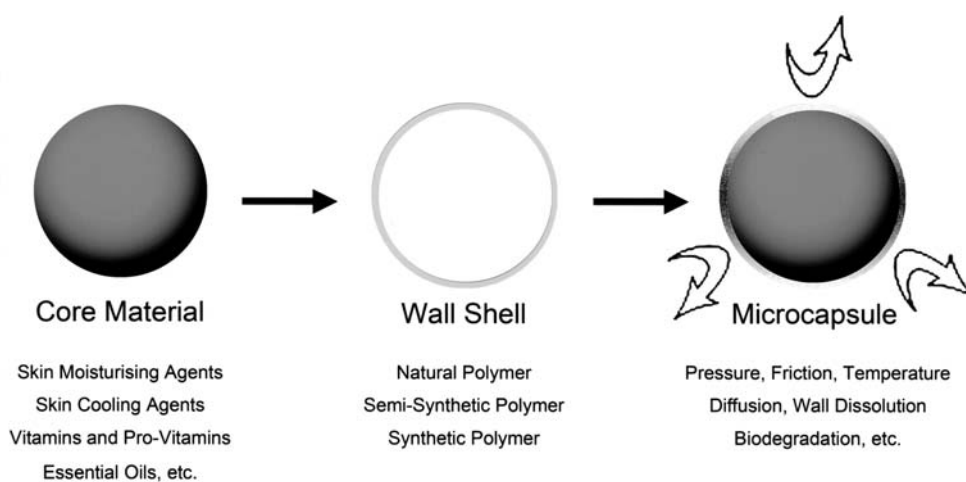


Figure 4. Structure of microcapsule.

Physical appearance of the microcapsule is generally governed by the physical nature of the core material. If solid or crystalline materials are encapsulated, the resulting microcapsule may have an irregular shape which tends to follow the original shape of the core particle. If a liquid is used for the core material, simple spherical microcapsules containing a single liquid droplet may be found (9).

Microcapsules can be produced with non-definite sizes and their typical diameters are in the range of 1-1,000 microns. Theoretically, microcapsules can be produced with diameters between 0.01 and 10,000 microns (9). The thickness of the microcapsule wall is generally in the range of 0.5-150 microns. In most cases, the core content of the microcapsule will represent between 20 and 95% of the total mass (9).

The choice of wall materials depends upon a number of factors including expected product objectives, requirement, nature of the core material, the process of encapsulation and economics (17). The wall material may be natural, semi-synthetic or synthetic. Table I illustrates the typical wall materials used for microencapsulation (12).

Microencapsulation methods. Many different manufacturing approaches have been adopted for microencapsulation. The most commonly used microencapsulation processes include a) complex coacervation, b) polymer-polymer incompatibility, c) interfacial polymerisation and *in situ* polymerisation, d) spray drying, e) centrifugal extrusion, f) air suspension coating, g) pan coating and h) emulsion hardening process (8,12,18,19).

Advantages of microencapsulation. One major advantage of using microencapsulation technology is its ability to protect active ingredients from hazardous environment, i.e. oxidation, heat, acidity, alkalinity, moisture or evaporation. It also protects ingredients from interacting with other compounds in the system, which may result in degradation or polymerisation. At the same time, the use of microencapsulation technology also protects the microcapsule end users from exposure to hazardous substances such as pesticides. Another important advantage of this versatile technology is its controlled release properties that seem to be

the best choice for increasing the efficiency and minimisation of environmental damage. Other advantages of microencapsulation technology include a) its ability to render liquids into powder form in order to prevent clumping and improve mixing, b) disguising the unpleasant flavours or odours such as pharmaceuticals as well as c) improving the handling of an ingredient before processing (6,9,18-20).

Microencapsulation applications in cosmetic textiles. In cosmetic textiles, the major interest in microencapsulation is currently in the application of vitamins, essential oils, skin moisturising agents, skin cooling agents and anti-ageing agents etc. A few studies regarding the techniques of producing microcapsules containing cosmetic substances have been found in literature (21-28) (Table II).

Yamato *et al* prepared microcapsules comprising the active substances used to improve the physiological conditions of human skin. The microcapsule will not be broken during production, but is gradually released when the textiles structure is subjected to light pressure created by the movement of human body (21).

Bishop *et al* introduced the use of waste yeast cells in microencapsulation process. After encapsulating the core material, the yeast cells were attached to both cotton and wool fibre by using crosslinking agents and binders. The processes of filling the yeast cells were very simple and the use of yeast cells as wall material generally provided several advantages such as high loading, inthermoplastic, protection from light, oxygen and hazardous environment, and cost effectiveness (22).

Copete Vidal *et al* introduced the invention of chitosan-based microcapsules containing various active components and investigated the durability performance with the mixture of microcapsules and binding agent. With the effect of finishing using microcapsules and binder, the active ingredients were released and washed out less quickly, and a high degree of hydration was achieved as well (23).

Korean researchers prepared melamine resin microcapsules containing Migrin oil by *in situ* polymerisation method. The structure, mean particle size and size distribution, morphologies, thermal properties and released behaviour



Types of core material	Examples
Solvents	Benzene, cyclohexane, chlorinated phenyls, paraffins, esters, ethers, alcohols and water
Plasticiser	Phthalate, adipate and pjosphate-type, silicones and chlorinated hydrocarbons
Acids and Bases	Boric acid, caustic alkali and amines
Catalysts	Curing agents, oxidants, free radical initiators and reducing agents
Colourants	Pigments and dyes
Adhesives	Polysulphides, cyanoacrylates and isocyanates
Fragrances	Menthol, essences and speciality compositions
Foods	Oils, fats, spices and flavours
Agricultural Chemicals	Herbicides, insecticides and pesticides
Pharmaceuticals	Aspirin, vitamins and amino acids
Recording Material	Reprographic toners, coupling agents, developers, silver halides, fixing agents, photochromatic compound, liquid crystals
Rust Inhibitors	Zinc chromate and other compounds
Others	Detergents, bleaches and fore retardant

A wide range of core materials including solvents, plasticisers, acids, bases and catalysts, etc can be encapsulated. Table II show some typical core materials to be encapsulated (12).

were characterised and discussed (24). They also prepared poly (L-lactide) microcapsules for fragrant fibre by interfacial precipitation method through solvent evaporation from water-in-oil-in-water emulsion. The microcapsules were then uniformly printed on the cotton fabrics and the resulted fabric could withstand 15 cycles of washing durability (25).

Jarfari *et al* have also tried to prepare microcapsules with polymethyl methacrylate (PMMA) as wall shell and Rose fragrance as core materials (26).

Many researchers have made efforts to improve the durability of microencapsulated functions. This is relatively the most difficult task in preparing the cosmetic textiles. Li *et al* investigated the effect of UV curing for the encapsulated aroma finish on cotton. The aroma function was prolonged to 50 wash cycles whereas the traditional curing method could withstand 25 wash cycles only (27). Chang integrated the processing procedure of fabric treatment with low temperature plasma, natural oil essence microencapsulation technique and fabric coating technique to improve the adhesion of microcapsules with fabrics (28).

4. Synthesis of gelatin/vitamin C microcapsules and its application on textile materials

To obtain a better understanding of the development of cosmetic textiles, gelatin microcapsules containing vitamin C were prepared in this study using the emulsion hardening technique. The gelatin microcapsules were subsequently

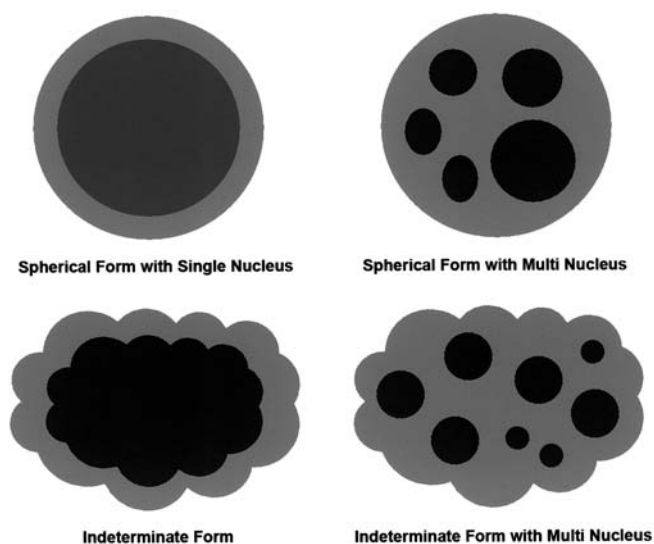


Figure 5. Forms of microcapsules.

grafted into fibrous materials for the production of cosmetic textiles.

Synthesis of gelatin microcapsules containing vitamin C. Gelatin (Ph Eur, Sigma Aldrich, St. Louis, MO) and vitamin C (Puriss. p.a, Sigma Aldrich) as shown in Fig. 6A and B, were dissolved in deionised water at the temperature of 40-60°C

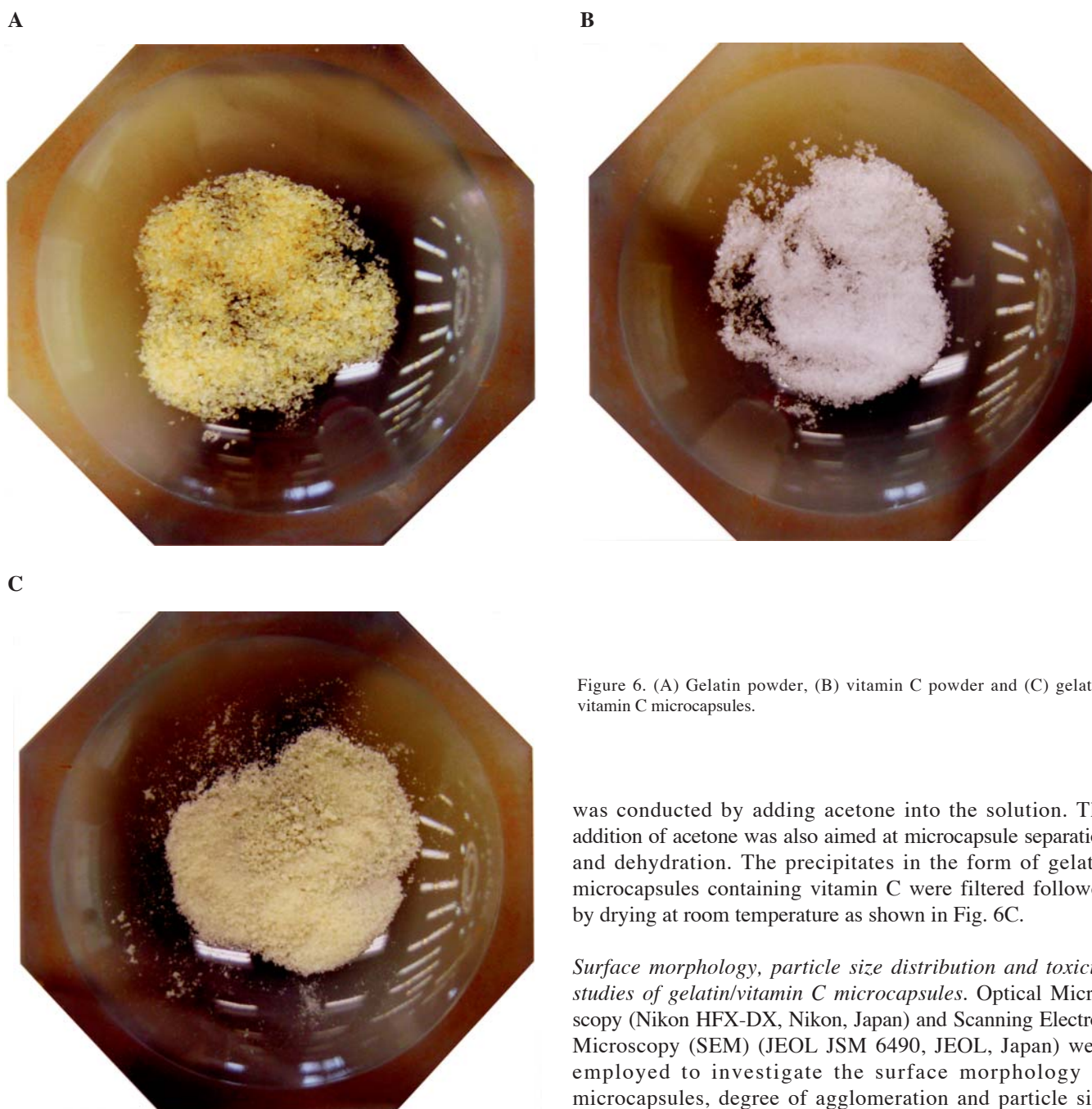


Figure 6. (A) Gelatin powder, (B) vitamin C powder and (C) gelatin/vitamin C microcapsules.

for 15 min. The solution was then poured into 100% pure canola oil to form the water in oil emulsion with the aid of Span 80 surface-active agent. The mixture was intermixed for 5 min using a magnetic stir plate, Heidolph MR3001 (Heidolph, Germany), at a speed of 1200 rpm to form a stable water in oil emulsion. The emulsion was then further mixed using an ultrasonic processor (Vibra Cell™ VCX 750, Sonics and Materials Inc., USA) at 300 Watt with the ultrasonic amplitude at 80% for 1 min to break down the emulsion into smaller droplets.

The water in oil emulsion was then stirred at 20°C while adding formaldehyde (ACS Reagent, Sigma Aldrich) to crosslink the microcapsules for 2 h at 1200 rpm. The addition of formaldehyde should be as slow as possible in order to keep the stability of system. Afterwards, precipitation process

was conducted by adding acetone into the solution. The addition of acetone was also aimed at microcapsule separation and dehydration. The precipitates in the form of gelatin microcapsules containing vitamin C were filtered followed by drying at room temperature as shown in Fig. 6C.

Surface morphology, particle size distribution and toxicity studies of gelatin/vitamin C microcapsules. Optical Microscopy (Nikon HFX-DX, Nikon, Japan) and Scanning Electron Microscopy (SEM) (JEOL JSM 6490, JEOL, Japan) were employed to investigate the surface morphology of microcapsules, degree of agglomeration and particle size distribution. The mean particle size of microcapsules was evaluated using a Zetasizer 3000 HAS (Malvern Instruments, UK). In view of formaldehyde being used as the cross-linking agent in the microencapsulation system, the toxicity studies of gelatin microcapsules was conducted on human liver and breast cell line as well as the primary culture obtained from the patient with non-malignant haematological disorder.

Surface morphology and particle size distribution. Fig. 7 shows the optical micrograph of gelatin microcapsules containing vitamin C. The gelatin microcapsules were in spherical form with a certain degree of agglomeration. Fig. 8 shows the SEM image of gelatin microcapsules at the magnification of x350. In agreement with the observation obtained from the optical micrograph, the gelatin microcapsules appeared to be round in shape with relatively smooth surface and the particle size of microcapsules was

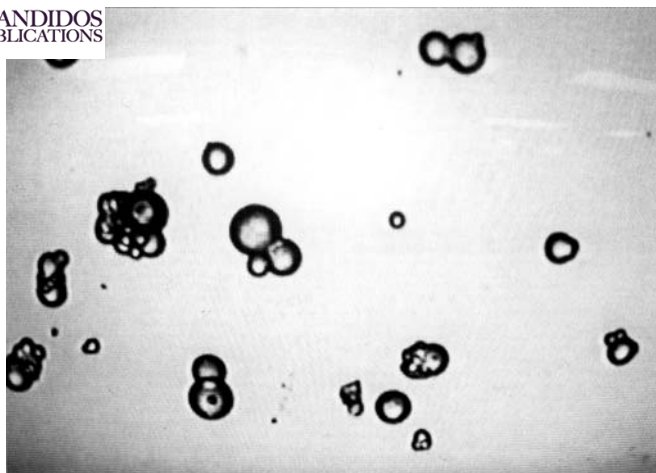


Figure 7. Optical image of gelatin microcapsules containing vitamin C.

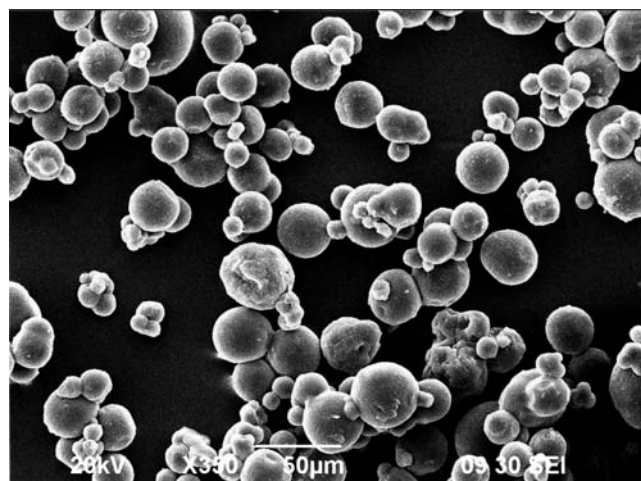


Figure 8. SEM image of gelatin microcapsules containing vitamin C.

ranging from 5.0 to 44.1 μm . The result of average particle size was 24.6 μm as obtained from the particle size analyser. However, a certain degree of agglomeration of microcapsules was noted as shown in Fig. 7. The agglomeration of microcapsules was largely affected by the stirring speed during the cross-linking process. When the stirring speed was too slow, the uncrosslinked microcapsules could not be entirely separated and it would be very difficult to separate the microcapsules once they combined. Hence, high stirring speed during the crosslinking process should be maintained in order to form the separated microcapsules.

Toxicity studies. Formaldehyde is known to be toxic, allergenic and carcinogenic. It is classified as a probable human carcinogen by the US Environmental Protection Agency. Melamine-formaldehyde microcapsules have been studied by a number of researchers over the past decade to encapsulate different essential oils for specific purposes (24,29-32). However, the related cytotoxicity studies of the microcapsules are always missing. Since formaldehyde was used as the crosslinking agent in the gelatin-based microencapsulation system, an *in vitro* toxicity test of gelatin microcapsules was conducted on the human liver Hep3B and human breast MDAMB-231 cell lines as well as the primary bone marrow culture obtained from patient with non-malignant haematological disorder. It was observed that these microcapsules exerted no significant toxicological effect on those human cells at a maximum concentration of 100 $\mu\text{g}/\text{ml}$ after 48 h of incubation as shown in Fig. 9A-F, respectively (33-38). The gelatin microcapsules containing vitamin C were therefore proved to be non-cytotoxic to human cells under *in vitro* condition.

Application of gelatin/vitamin C microcapsules on textile materials. Cotton (100%) fabric samples were cut into many pieces of 20x20 cm. Gelatin microcapsules containing vitamin C were firstly diluted with deionised water at the liquor ratio of 1:10. The solution was stirred for 10 min using a magnetic stir plate at a speed of 1200 rpm to obtain a better dispersion of microcapsules. Textile binder (Devan Chemicals) was used to enhance the fixation of gelatin microcapsules

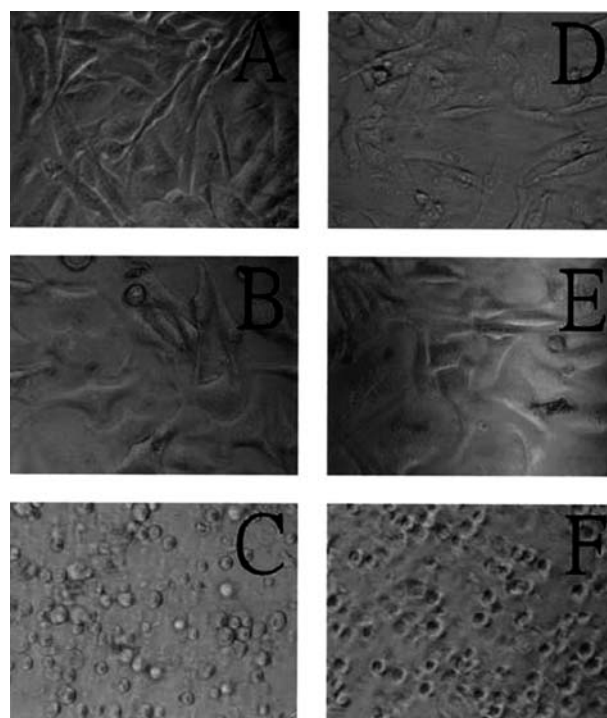


Figure 9. (A) Control liver Hep3B cell line. (B) Control breast MDAMB-231 cell line. (C) Control primary culture obtained from patient with non-malignant haematological disorder. (D) Liver Hep3B cell line treated with gelatin/vitamin C microcapsules. (E) Breast MDAMB-231 cell line treated with gelatin/vitamin C microcapsules. (F) Primary culture obtained from patient with non-malignant haematological disorder treated with gelatin/vitamin C microcapsules.

onto the cotton fabric. The fabric samples were then immersed into the microcapsule solution followed by padding using a vertical padder at a constant pressure of 1.5 kg/cm^2 and a speed of 7.5 rpm. The wet uptake level of fabric samples was $70 \pm 5\%$ followed by drying the fabric at room temperature.

Fig. 10A shows the SEM image of cotton fibres exhibiting the fibrous structure. Fig. 10B shows the SEM image of cotton fibres embedded with the gelatin microcapsules. The SEM results confirmed that microcapsules

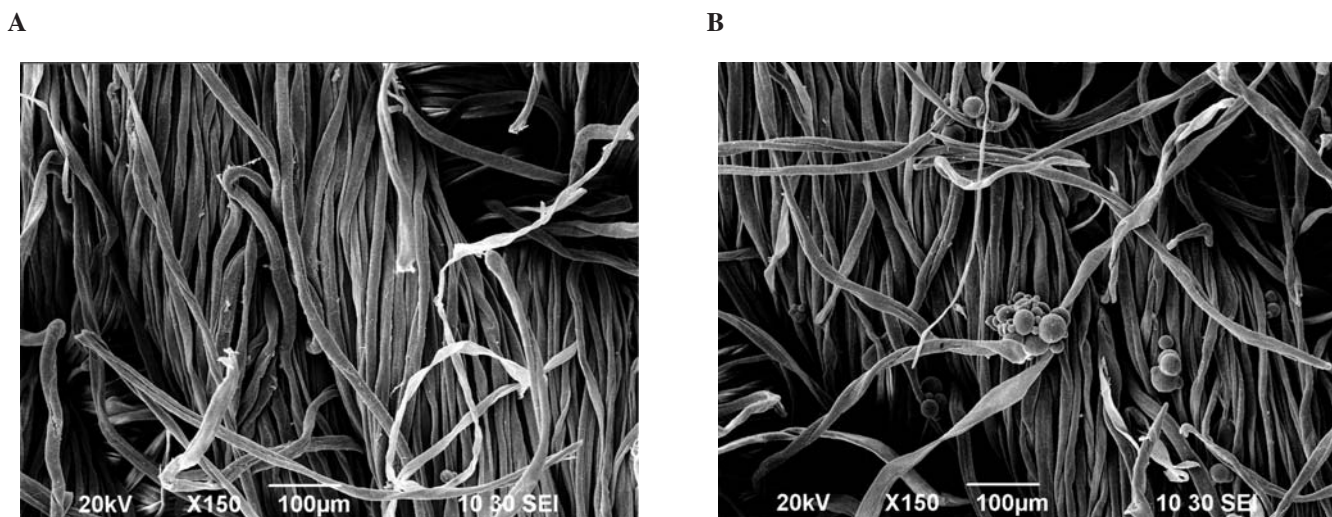


Figure 10. (A) Control cotton fibre and (B) cotton fibre embedded with gelatin/vitamin C microcapsules.

were successfully padded onto the fibre surface without obvious breakage during the padding process. The particle size of the gelatin microcapsules ranged from 6.7 to 36.7 μm which was similar to that of the previous SEM evaluation. The use of vitamin C is known to be beneficial to skin such as promoting the synthesis of collagen and restraining the decomposition of collagen, while it is effective in preventing wrinkles on the skin. The vitamin C substance is released from the gelatin wall shell in the presence of humidity due to its sensitivity to moisture. A relatively small amount of water is sufficient to achieve the release, such as the water and sweat present on the surface of the skin. Hence, the released vitamin C may be directly be adsorbed by the skin.

5. Conclusion

Cosmetic textiles are increasingly popular and expanding considerably in the textile industry as the market message becomes more widely appreciated. The innovative development of cosmetic textiles can be as wide as imagination. In this study, the gelatin microcapsules containing vitamin C were prepared using the emulsion hardening technique and successfully grafted into fibrous materials. This provides a practical example for explaining the development of cosmetic textiles with biological benefits. The controlled release mechanism of cosmetic textiles with respect to human skin can be further studied.

The integration of aromatherapy in textile application is a novel and user-friendly idea that enables an alternative basis for essential substance delivery systems. At present, the application of aromatherapy in textiles is commonly concentrated on skin caring benefit and stress management. More innovative ideas can be as wide as imagination such as hair care and treatment, body slimming and even medical applications etc. Microencapsulation technology is an effective technique to achieve satisfactory performance; although it is still relatively new to the textile and apparel industry. The wide range of benefits of aromatherapy and the controlled release of essential oils or active ingredients are expected to be appreciated by consumers.

The application of cosmetic textiles can also be expanded to the bio-medical field. Traditionally, people with adverse skin conditions are treated with either oral or topical drugs, or a combination of both. The traditional problems associated with the use of biological agents on skin are usually related to inappropriate, delayed or missing doses. The development of bio-medical textiles with the microencapsulated agents will definitely offer a more reliable and durable protocols for providing skin-care solutions and treating skin diseases. The events will be focused on encapsulating drugs to provide a controlled release of an active ingredient to solve the skin-related problems by absorption through the skin. As for the skin-care purposes, the encapsulated components can include i) skin moisturisers, e.g. elastin and keratin, ii) UV blocking agents, e.g. zinc oxide and titanium oxide and iii) growth factors for stimulating hair growth. In the case of treating skin associated disorders, the encapsulated substances can include anti-allergic agents, e.g. anti-histamine, or immuno-suppressing agents.

As a whole, it is anticipated that the development of cosmetic textiles will continue to grow and explore completely new possibilities for providing bioactive body care functions to wearer in the near future. It is really a challenging and exciting time for both the textile and medical industries. The textile industry collaborated with medical profession should continue to explore and develop functional textiles that fit the consumer needs and behaviour.

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