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Application of microencapsulation in textiles

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Abstract

The textile roots of yeast microencapsulation technology was introduced as were the wide range of applications in food and other business sectors. In microencapsulation in general the number of commercial applications in the textile industry continues to grow particularly in the textile industries of Western Europe, Japan and North America. The move by the more developed countries into textiles with new properties and added value, into medical textile and technical textiles for example has encouraged the industry to use microencapsulation processes as a means of imparting finishes and properties on textiles which were not possible or cost-effective using other technology. Textile manufacturers are demonstrating increasing interest in the application of durable fragrances to textile as well as skin softeners. Other potential applications include, insect repellents, dyes, vitamins, antimicrobials, phase change materials and in specific medical applications, antibiotics, hormones and other drugs. Examples of each technology are described. A short summary of a new microencapsulation technology with roots in the textile industry, yeast based microencapsulation, is also described. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Microencapsulation; Yeast; Textiles; Fragrance; Phase-change materials

1. Introduction

My work on microencapsulation in textiles began over 12 years ago when I was asked to produce a durable finish on a, cotton or woolen textile, which combated body odour in products such as shirts and socks. Research turned to microencapsulation and eventually to the development of a low cost-high volume microencapsulation process based on waste yeast cells (see Fig. 1), (*Saccharomyces cerivisiae*) (Bishop et al., 1998; Pannell, 1990; Nelson et al., 1991). At that stage the main objective was not met in textiles, however, the technology has progressed well in other business sectors, primarily in food, but now also in agrochemicals, cosmetics and pharmaceuticals. A company called Fluid Technologies Plc. was set up in September 2000 to bring the technology to market and has now developed a series of essential oil flavour encapsulates for food applications, with many more in the pipeline.

Fluid's yeast-based microencapsulation technology is a unique process utilising the finest quality

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yeast, chosen for its bland taste, light colour and is availability in large volumes. The patented process is a simple mixing process and because of the natural properties of the yeast has many benefits over existing, microencapsulation technology:

- A natural 'preformed' capsule, made up of a strong carbohydrate outer cell wall and a lipid bilayer membrane
- Process uses no additives, only water, yeast and active
- High active loading, up to 70% w/w, 30–40% most common
- Flavour and other actives are released in contact with moist mucous membrane surfaces such as the tongue and nose without braking the cells
- Release properties in many cases allows reduction of flavour in product by as much as 75% without loss of flavour profile
- Due to the natural bioadhesive properties of the yeast, flavour release can also be described as lingering or long-lasting
- The double carbohydrate wall/lipid membrane capsule protects volatile products from evaporation, and from damage from light and oxygen, therefore, providing a highly stable product with extended shelf-life
- The capsules are not thermoplastic and retain and protect most of the capsule contents from heat, during processes such heat extrusion, and cooking processes such as baking, roasting, frying, boiling etc.
- Cost-effective against all known microencapsualtion technology

So returning to the textile industry, in general, overall, the textile industry has been slow to react to the possibilities of microencapsulation, although by the beginning of the 1990s a few commercial applications were appearing with many more at the research and development stage (Nelson, 1991). As the industry moves into the 21st century the number of commercial applications of microencapsulation in the textile industry continues to grow, particularly in Western Europe, Japan and North America. The move by the more developed countries into textiles with new properties and added value, into medical textiles and technical textiles for example, has encouraged the industry to use microencapsulation processes as a means of imparting finishes and properties on textiles that were not possible or cost-effective using other technology.

In textiles the major interest in microencapsulation is currently in the application of durable fragrances and skin softeners. Other applications include insect repellants, dyes, vitamins, antimicrobial agents, phase-change materials and medical applications, such as antibiotics, hormones and other drugs.

2. Phase-change materials

Microencapsulation technology was utilised in the early 1980s by the US National Aeronautics and Space Administration (NASA) with the aim of managing the thermal barrier properties of garments, in particular for use in space suits. They encapsulated phase-change materials (PCMs) (e.g. nonadecane) with the hope of reducing the impact of extreme variations in temperature encountered by astronauts during their missions in space. Ultimately the technology was not taken up within the space programme. However, the potential was recognised and after further development the work was licensed by the inventor, the Triangle Research and Development Co. to Outlast Technologies, based in Boulder, Colorado. Outlast has exploited the technology in textile fibres and fabric coatings (see Fig. 2), and PCM capsules are now applied to all manner of materials (Zubkova, 1995a,b; Colvin and Bryant, 1998), particularly

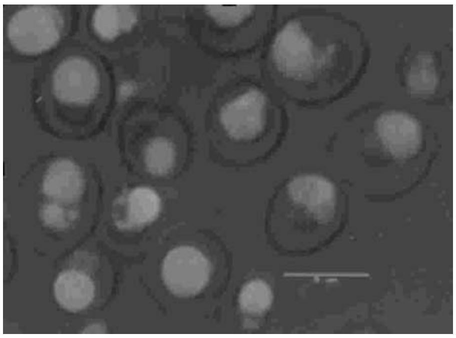

Fig. 1. Yeast microcapsules containing sage oil.

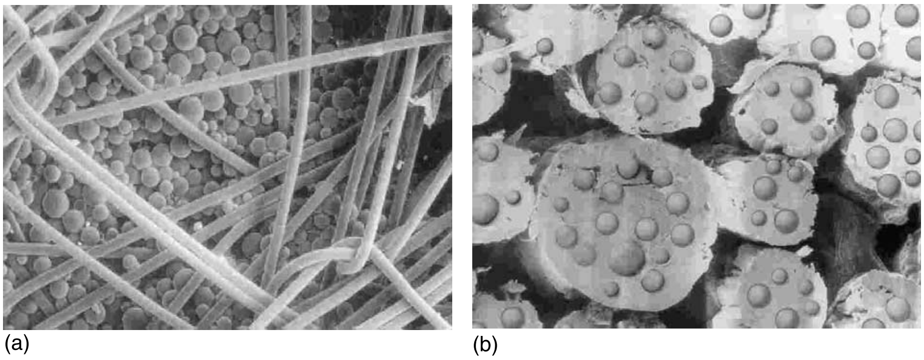

Fig. 2. PCM microcapsules coated on the surface of fabric (a) and embedded within fibre (b).

outdoor wear (parkas, vests, thermals, snowsuits and trousers) and in the house in blankets, duvets, mattresses and pillowcases. As well as being designed to combat cold, textiles containing PCMs also helps to combat overheating, so overall the effect can be described as thermoregulation. The microcapsules have walls less than 1 μm thick and are typically 20–40 μm in diameter, with a PCM loading of 80–85%. The small capsule size provides a relatively large surface area for heat transfer. Thus the rate at which the PCM reacts to an external temperature changes is very rapid (Pause, 2000).

Accordis, formerly Courtaulds Fibres, in Bradford, UK, developed the technology of in-fibre incorporation of the Outlast microcapsules, loading the fibre with 5–10% of microcapsules Cox, 1998. The process utilises late injection technology that was also used to produce the antimicrobial fibre Amicor. In this way the PCM is permanently locked within the fibre; there is no change necessary in subsequent fibre processing (spinning, knitting, dyeing, etc.) and the fibre exhibits its normal properties of drape, softness and strength.

3. Fragrance finishes

The addition of fragrances to textiles has been carried out for many years in the form of fabric conditioners in the wash and during tumble-drying; all are designed to impart a fresh aroma to the textile. However, no matter the quality of the technology used to impart the fragrance, the effect is relatively short-lived. Numerous attempts have been made at adding fragrances directly to fibre and fabrics but all fail to survive one or two wash cycles. Only through microencapsulation are fragrances able to remain on a garment during a significant part of its lifetime. Microencapsulation of essential oil flavours has led to many novelty applications, particularly for children's garments, but it has also allowed exposure at home and in the work place to the beneficial effects of aromatherapy. In future, fashion garments may carry the smell of branded perfumes, particularly as many perfume houses have entered the world of haute couture.

Since 1979 R T Dodge of Dayton, Ohio, has been involved in the development and manufacture of microcapsules for a wide range of industries. In recent years the company has gained much experience in the provision of microcapsules for textiles. The majority of the work has been in microencapsulated 'scratch and sniff' T-shirts and in women's hosiery. The nature of the microcapsules have not been revealed but it is claimed that the shirts survive washing (typically 8–20 cycles), depending on the active agent encapsulated, and the hosiery up to ten washes. The capsules also survive drying in conventional tumble-dryers. Well-established techniques such as in situ and interfacial polymerisation are used to manufacture the capsules.

Celessence International of Hatch End, Middlesex, has been investigating and manufacturing microencapsulated fragrant-smelling compounds for a number of years. In the early days the applications included drawer liners, paper handkerchiefs, gift wrapping, stationary, greeting cards, advertising brochures, books, cartons and labels. The company has now turned its attention to textiles, using its basic technology of encapsulating fragrances in gelatin or synthetic capsules, which protects the contents from evaporation, oxidisation and contamination. The capsules range in size from 1 to 20 μm. In practice, the smaller the capsules the greater the covering of the product and the longer the fragrance will last, as it takes longer for the capsules to be ruptured by physical pressure. Larger capsules release more fragrance when ruptured. Traditionally the 'scratch and sniff' application of microcapsules used screen-printing but now litho and web printing techniques have been adopted, initially in paper products such as bus tickets for promotional activities and now in textiles. In late 1999 Celessence formed an alliance with Brookstone Chemicals (part of the Croda International Group), a textile auxiliary and dyestuff supplier. The technology allows a textile manufacturer to add a fragrance, vitamin, moisturiser or even an insect repellent to all types of textile substrates, including hosiery (Yamada and Yamada, 1999). Depending on application weights and the wash cycle used, up to 30 washes can be achieved without complete loss of fragrance. Celessence TXT capsule systems comprise aqueous dispersions of encapsulates, which can be applied by pad, exhaustion or hydroextraction techniques to a wide variety of textile substrates. Durability to washing and handle (or feel) may be further improved by incorporating suitable formaldehyde-free binders and softeners. All applied products are blended from natural and synthetic materials that conform to legislative guidelines for cosmetic products, and as such pose no health hazards (Yamada and Yamada, 2000). For screen-printed application the encapsulates are simply mixed with water-based, solvent-free inks or binders. The capsule printing must be the last pass under a screen to avoid damage to the walls by further screens. Once printed, the fabric is then cured as with standard textile inks to achieve a good bond to the fibres.

Kanebo Gohsen of Osaka, Japan, has continued its interest in microencapsulated fragrances derived from its perfumery division. The products continue to sell well in Japan, particularly in hosiery, scarves, handkerchiefs and other products. The Matsui Shikiso Chemical Co of Kyoto has also developed a way of fixing aroma compounds to fabric using microcapsules. The fabric is first treated with a nitrogenous cationic compound and the microcapsule wall is manufactured to adhere to this layer. The capsules can range in size from 0.1 to 100 μm and are made using interfacial or in situ polymerisation techniques. Typical compounds encapsulated include perfumes such as musk, civet, ambergris, pine and citrus oils.

LJ Specialities, part of the Itochu Group has also introduced microencapsulated fragrance products for the textile industry. Working with some of the larger textile manufacturers in the UK and elsewhere they have produced fresh-smelling sheets, towels and garments with a wide range of perfumes such as eau de Cologne and fruity smells such as apple and orange (Yamada and Yamada, 2000). More unusually fragrances such as cola and pizza have also been encapsulated and applied to textiles. The contents of the capsules are released with light abrasion as would take place during day-to-day wear. The capsules survive repeated washing and can be applied to fibres such as cotton as a dispersion with a binder, using padding, exhaust or screen-printing techniques. Usually a softener is also required, as unsoftened fabric containing microcapsules can sometimes appear to be stiffened. The capsules are colourless and can be applied over coloured fabric or printed patterns without any adverse visible effects.

In Korea the Eldorado International Co of Seoul and a number of other companies offer new fabrics that emit the natural aroma of flowers, fruit, herbs and perfumes. Emulsified microcapsules containing a natural aroma or essential oil are attached to the fabric after dyeing. The capsules break on movement of the wearer, releasing the aroma. In general the capsules continue to emit aroma for up to 25 wash cycles and on the shelf the finish will remain ready for action for between 3 and 5 years. So far the company has applied the technology to curtains, sofas, cushions and sheets, as well as some toys. Like many of these products, the manufacturers claim aromatherapy effects such as ability to help with insomnia. The most frequently applied aromas include peppermint, lemon, jasmine, pine and orange. Silk ties have also been produced that release fragrant oils during normal wear, and if rubbed they produce a large burst of fragrance. The fragrant effect can last for a year and a half. Gloves and socks are also available that have fragrance-release properties and some antibacterial effects, which the manufacturers claim to last for up to 25 wash cycles.

Also in Korea, workers at Pusan National University were able to prepare microcapsules using melamine-formaldehyde systems containing fragrant oil (Hong and Park, 1999). When attached to cotton these capsules were able to survive over 15 wash cycles. Scanning electron microscopy indicated that the smaller of the capsules in the range survived more effectively after laundering. This phenomenon may simply be due to the relative thickness of a capsule within an adhesive film binding the capsules to the textile substrate.

Euracli, a company based in Chasse-sur-Rhône in France, has produced microcapsules containing perfumes or cosmetic moisturisers that can be padded, coated or sprayed onto a textile and held in place using an acrylic or polyurethane binder. Many fibre types have been produced containing microcapsules with many successful products including the Hermes scarf with Caleche perfume, Neyret lingerie, small silk squares by Lancôme perfumed with Poeme to celebrate the Chinese New Year, perfumed dresses for the Olivier Lapidus summer show (July 1998), Playtex bras and Dim moisturising and energising tights, on the market since December 1998.

Paper-like products have been produced containing microencapsulated essential oils such as lavender, sage and rosemary for odour control applications in shoe liners and insoles by Aero of Celje, Slovenia (Boh et al., 1999). Paper and other nonwoven products, no matter the method of manufacture, lend themselves very well to entrapment of microecapsules, producing long-lasting effects.

In Germany Hako–Werke Gmbh has produced a microencapsulated, fragrance-coated floor cloth (Hako-Werle Gmbh, 1997). A fresh fragrance is released during normal use reducing the requirement for aerosol fragrance application.

4. Polychromic and thermochromic microcapsules

Colour-changing technology has been around for a number of years, generally applied to novelty application such as stress testers, forehead thermometers and battery testers. New applications are now beginning to be seen in textiles, such as product labelling, and medical and security applications. In addition there is continued interest in novelty textiles for purposes such as swimwear and T-shirts.

There are two major types of colour-changing systems: thermochromatic which alter colour in response to temperature, and photochromatic which alter colour in response to UV light. Both forms of colour-change material are produced in an encapsulated form as microencapsulation helps to protect these sensitive chemicals from the external environment. Today manufacturers are able to make dyes that change colour at specific temperatures for a given application, e.g. colour changes can be initiated from the heat generated in response to human contact.

Physiochemical and chemical processes such as coacervation and interfacial polymerisation have been used to microencapsulate photochromic and thermo-chromic systems. However, to obtain satisfactory shelf-life and durability on textiles, interfacial polymerisation techniques are nearly always adopted; these are the same techniques used to produce textile fibres and films such as polyester, nylon and polyurethane. The most widely used system for microencapsulation of thermochromic and photochromic inks involves urea or melamine-formaldehyde systems (Aitken et al., 1996). Some efforts are being made to improve the shelf-life of thermochromatic dyes and companies such as Chromatic Technologies Ltd have recently demonstrated methods that substantially improve performance by careful choice of microcapsules and dispersing solvents (Small and Highberger, 1999). Microencapsulated thermochromatic dyes generally survive up to 20 laundering cycles, although excessive drying at elevated temperatures or use of bleach can reduce the longevity of the finish.

5. Fire retardants

Fire retardants have been applied to many textile products, but in certain cases they can affect the overall handle, reducing softness and adversely affecting drape. Microencapsulation has been used to overcome these problems for example in fabrics used in military applications such as tentage (Kover et al., 1997). Others have incorporated the microencapsulated fire retardants during spinning of a polyester fibre for blending with cotton (Zubkova, 1997).

6. Counterfeiting

In high added value textiles, and in branded and designer goods there is great pressure to protect from illegal copying within the marketplace. Microencapsulation can be used to help with this problem by offering a covert yet distinctive marking system. One example of this technology is that developed by Gundjian and Kuruvilla, 1999 of Nocopi Technologies. This system for combating textile counterfeiting utilises microcapsules containing a colour former or an activator applied to, for example, a thread or a label. The microcapsules adhere to the textile and, depending on the type of chemical within the capsules can be detected at a later date to check authenticity. Detection may be achieved directly using UV light or more likely by using a solvent to break open the capsules, releasing the contents and allowing a colour to develop.

7. Liposomes

In recent years liposomes have been examined as a way of delivering dyes to textiles in a cost-effective and environmentally sensitive way (Rocha Gomes et al., 1997; de la Maza et al., 1998; Coderch et al., 1999a,b). The liposomes used (for example, commercially available PC liposomes from Transtechnics SL) were cost-effective, and no specific equipment or skills were required to handle them within the dyehouse. The results were excellent with pure wool and wool blends, and as the temperature of dyeing could be reduced there was less fibre damage. In their studies dyebath exhaustion was shown to greater than 90% at the low temperature (80 °C) used resultling in significant saving in energy costs. The impact of the dyeing process on the environment was also much reduced with chemical oxygen demand (COD) being reduced by about 1000 units.

8. Miscellaneous applications

Working on a range of fabrics, a team of workers in France has encapsulated glycerol stearate and silk protein moisturisers for application on bandages and support hosiery (Dim, 1999). The material maintains comfort and skin quality through extensive medical treatment where textiles are in direct contact with the skin.

The Mitsubishi Paper Mills has produced a polypropylene nonwoven material for application as a cleaning/wiping cloth containing microencapsulated octane, tung oil and paraffin oil as cleaning solvents (Yokato, 1998). The cloths feel good in the hand and have very good cleaning properties.

The application of insecticides and acaricides to textiles to combat dust mites and insects such a mosquitoes has been investigated by many workers. Microencapsulation has been considered as a mechanism of retaining the effect for significant periods without exposing the user to excessive dosages of hazardous chemicals. The use of alternative insecticidal compounds such as those found in many essential oils and other plant extracts has made the production of long-lasting acaricide bed sheets possible (Yamada and Yamada, 1997).

9. Microencapsulation: the future

The 'holy grail' for most textile applications using microcapsules would be a system that is easy to apply, does not effect the existing textile properties and has a shelf-life on a garment that allows normal fabric-care processes to take place. Currently, although capsules can survive 25–30 wash cycles, conventional ironing and other heat-input processes such as tumble-drying can cause a dramatic reduction in the desired effect. The microencapsulation industry must take more notice of the possibilities within the textile industry and specifically design microcapsules that overcome these problems. For the future, the consumer's desire for novel and unique effects will always be present. But more importantly, in an ever-increasing desire for convenience, the consumer will require that fabric properties are inherent in the garment, e.g. fresh odour and softness. Consumers will expect these properties to last the lifetime of the garment, and not involve routine intervention in the form of the never-ending addition of washing aids and fabric conditioners. Microencapsulation may deliver these long-term goals.

The desire for a healthier and more productive lifestyle will continue to generate a market for textiles that promote 'well-being'. Textiles that 'interact' with the consumer, reducing stress, promoting comfort and relaxation, are possible through active delivery from microcapsules. In the last decade the textile industries of Western Europe, Japan and the US have concentrated on developing performance fabrics with added value for sports and outdoor application, as well as novel medical textiles. Microencapsulation can play a part in this continued development, for example by allowing sensing chemicals to be attached to sports clothing and medical products; these will be able to warn of damage or hazard to the wearer. Systems can also be developed that deliver measured dosages of chemicals to combat muscle pain or other more serious injuries. The potential applications of microencapsulation in textiles are as wide as the imagination of textile designers and manufacturers. Early success for some companies in producing microencapsulated finishes for textiles have come about from collaboration and adaptation of technology from other industrial sectors. The textile industry must continue to be outward looking and develop the textiles that consumers desire.

References

Aitken, D., Burkinshaw, S.M., Griffiths, J., Towns, A.D., 1996. Textile applications of thermochromic systems. Rev. Prog. Color 26, 1–8.

Bishop, J.R.P., Nelson, G., Lamb, J., 1998. Microencapsulation in yeast cells. J. Microencapsulation 15 (6), 761–773.

Boh, B., Hodzar, D., Knez, E., Kukovic, M., Pipal, V., Voda, K., 1999. Development of microcapsules for textile finishing. In: Glavic, P., Brodjnak-Voncina, D. (Eds.), Zb. Ref. Posvetovanja Slov. Kem. Dnevi, Maribor. University of Maribor, Slovenia, pp. 762–767.

Coderch, L., Manich, A.M., Marti, M., de la Maza, A., Parra, J.L., Serra, S., 1999a. Complementary study of optimizing a wool dyeing process with commercially available liposomes. Text Res. J. 69 (10), 789–790.

Coderch, L., Marti, M., de la Maza, A., Manich, A.M., Parra, J.L., Serra, S., 1999b. Potential of liposomes to aid the quest for cleaner dyeing. Wool Record (Dec), 26.

Colvin D.P., Bryant, G.Y. 1998. Protective clothing containing encapsulated phase change materials. Advances in heat and mass transfer in biotechnology (HTD) New York: ASME, 362, 123–132.

Cox, R., 1998. Dec. Synopsis of the new thermal regulating fiber outlast. Chem. Fibers. Int. 48, 475–479.

Dim, S.A., 1999. Textile Bio-Actif comportant dans ses fibres de la proteine de soie et des microcapsules de produit actif. French Patent, FP 2 780 073 A1.

Gundjian, A., Kuruvilla, A., 1999. Methods of authenticating a textile product and a thread and a woven label usable therewith. United States Patent, USP 5 941 572.

Hako-Werle Gmbh, 1997. Fragrance-containing polishing or cleaning pads for floors. German Patent, DP 1 9545 242.

Hong, K., Park, S., 1999. Melamine resin microcapsules containing fragrant oil:synthesis and characterization. Mat. Chem. Phys. 58, 128–131.

Kover, R.F., Farrel, B., Tiano, T., Levi, R., Harpel, J.W.H. 1997. Microencapsulated Fire Retardants for Flame Resistant Tentage Textiles. In: Proceedings of Eigth Annual BCC Conference on Flame Retardancy, Vol. 8, 88-97.

de la Maza, A., Coderch, L., Manich, A.M., Marti, M., Parra, J.L., 1998. Optimizing a wool dyeing process with an azoic 1:2 metal complex dye using commercially availabe liposomes. Text. Res. J. 68 (9), 635–642.

Nelson, G., Wales D.S., Sagar, B.S., 1991. Treating Materials. European. Patent, EP 0511 258 B1.

Nelson, G., 1991. Microencapsulation in textile coloration and finishing. Rev. Prog. Color 21, 72–85.

Pause, B., 2000. Measuring the thermal barrier function of phase change materials in textiles. Tech. Text Int. 9 (3), 20–21.

Pannell, N.A., 1990. Microencapsulation in microorganisms. European. Patent, EP 0242 135, B1.

Rocha Gomes, J.I.N., Genovez, M.C., Hrdina, R., 1997. Controlling exhaistion of reactive dyes on wool by microencapsulation with liposomes. Text Res. J. 67 (7), 537–541.

Small, L.D., Highberger, G., 1999. Thermochromic ink formulations, nail lacquer and methods of use. Chromatic Technologies United States Patent, USP 5 997 849. 5.

Yokato, Y., 1998. Wiping materials containing encapsulated cleaning agents with good handling and high cleaning properties. Japanese Patent 98 (140), 454.

Yamada, K., Yamada, Y., 1997. Insecticide sheets containing microencapsulated tree extracts. Japanese Patent, JP 96 53 319 A2. 6.

Yamada, K., Yamada, Y., 1999 (Dec). Fragrant Alliance. Int. Dyer, 10.

Yamada, K., Yamada, Y., 2000 (June). A Scent of the Unusual. Int. Dyer, 26.

Zubkova, N.S., 1995a. Thermal Insulation. Knit. Int. 102 (1216), 50.

Zubkova, N.S., 1995b. Phase change technology outlasts lofted fabrics. Tech. Text. Int. 4 (7), 28–29.

Zubkova, N.S., 1997. A highly effective domestic fire retardant for fireproofing fibrous textile materials. Fibre. Chem. 29 (2), 126–129.