

# Microcapsules

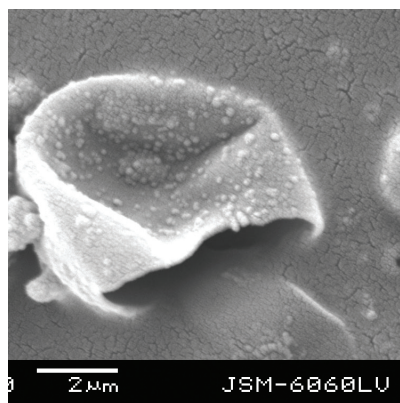
Microcapsules containing a range of useful materials can be found in a number of everyday products, write **Denis Poncelet** and **Bojana Boh**

# deliver

## In the last decade, designer microcapsules containing a variety of materials

have been increasingly used to impart innovative user functions to end products, particularly in cosmetics and functional fabrics. However, this is the 'visible part of the iceberg', according to encapsulation experts. While encapsulation is often key to delivering an innovative process, the microcapsules themselves are rarely apparent in the final products.

The list of applications for microencapsulation technology, however, is expanding quickly. Almost all of the major firms in almost all sectors are developing or using microcapsules (Table 1) and the number of publications is growing rapidly (Fig 1), dominated largely by patents. Most processes can be divided in two main approaches: either involving the entrapment of liquids in an outer membrane or of particles in an exterior coating. These processes in turn each involve three steps: incorporation of the actives inside the core/matrix; dispersion of the core – *ie* droplet formation or particle mixing; and stabilisation of the droplet or the coating.



Small wonder: microcapsule delivery

Encapsulation can serve any of a number of different functions, from isolating the entrapped core material from its surroundings, to reducing its volatility or improving the handling of a particular material. For most industrial applications, the aim is usually not to isolate the core completely, but to control the rate at which it leaves the microcapsule, as, for example, during the release of certain drugs or pesticides.

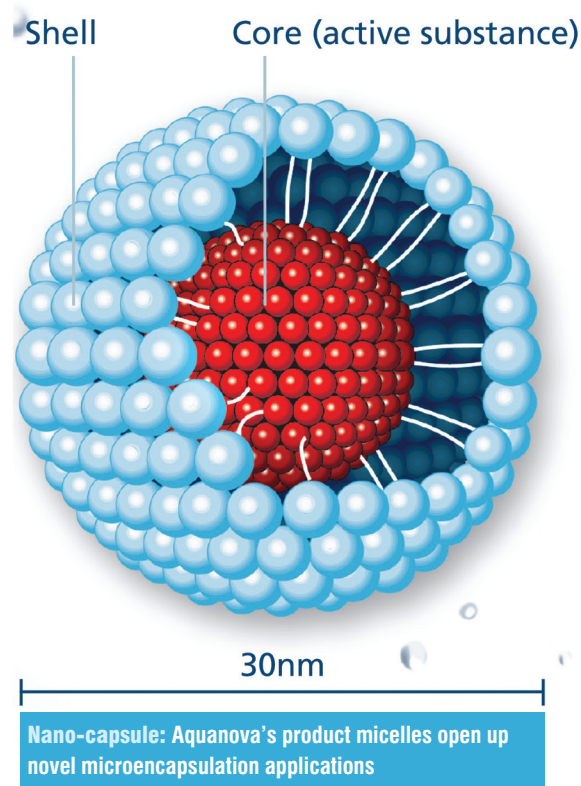
The traditional size of microcapsules is in the range of 200 μm to 800 μm. However, nano-capsules used to deliver active ingredients such as in skin creams and cancer therapies have developed quickly in the past few years. Capsules developed for cosmeo-textile applications – garments designed to release odours or deliver other functions on wearing – must also be smaller than a typical fibre diameter while capsules suspended in clear liquids need to be smaller than 20 μm in order to be 'invisible'.

For usual applications, nanoencapsulation is not always the best solution. The smaller the capsules, the more difficult it is to achieve good protection and release profiles. Particles smaller than 50 μm are often difficult to handle, especially in dry form, and not only present a high risk of inhalation but may also have explosive properties.

In contrast to the 'nano' fashion, another trend is for making millicapsules, from 1 to 3 mm in diameter. These are visible and must therefore be aesthetic and attractive. BASF's Coletica, part of the group's Beauty Care Solutions division, for example, has developed colourful capsules to be integrated in transparent creams, showing the presence of the active ingredi-

## In brief

- The number of applications for microencapsulation technology is growing rapidly
- Encapsulation is generally to control the release of the entrapped constituent
- New technologies to produce microcapsules should broaden the spectrum of possible uses



# Speciality chemicals

| Domain                  | Company (examples)  | Applications  |
|-------------------------|---|---|
| Food                    | Kraft, Danisco, Unilever, Nestlé, Firmenich, Cargill, Mane, IFF   | Flavour, essential oils, probiotics, vitamins, ferrous supplementation, sweeteners, acidulants, salts, baking powder, antioxidants, preservatives, stabilisers, dyes, enzymes for cheese ripening |
| Feed                    | Pancosma, ErboSpraytec, Frippak Feeds   | Essential oils, vitamins, special diets for aquacultures  |
| Pharmacy                | Merk, Abbot, Sanofi-Aventis, Boehringer-Ingelheim, Bristol-Myers-Squibb, Eli Lilly, Pfizer, Bayer             | Controlled drug delivery, gastric juice protection, diagnostics, contrasting agents   |
| Personal and house care | Johnson & Johnson, Unilever, Genencor, Procter & Gamble   | Enzymes, fragrances, chelating agents, bleaching agents, softeners, antistatics, anti-foaming agents  |
| Chemistry               | BASF, Solvay, Total, Dow Chemicals, Dow Corning, ISP, Bayer, Penwalt, Fuji, Xerox, Canon, Appleton Papers, 3M | Catalysts, enzymes, dyes and pigments, adhesives, corrosion inhibitors, fire retardants, blowing agents, UV-protecting agents, fragrances, biocides   |
| Cosmetics               | L'Oreal, Vichy, LVMH, Symrise, Givaudan   | Perfumes, enzymes, moisturising agents, antioxidants, plant extracts, protein hydrolyzates, vitamins, oils, antiseptics, sun-tanning agents   |
| Textiles                | Outlast, Frisby, Triangle, Microteck, Lytess  | Phase change materials, fire retardants, dyes for special effects, fragrances, biocides   |

**Table 1: Most major companies have microencapsulation interests**

ents. Such capsules have been used in shampoos, cleansers and other cosmetics.

In terms of capsule manufacture, narrow size distribution is a must, allowing better control of properties such as mechanical resistance, release profile and appearance. Large-scale plants producing hundred of tonnes of product generally use emulsion or spray

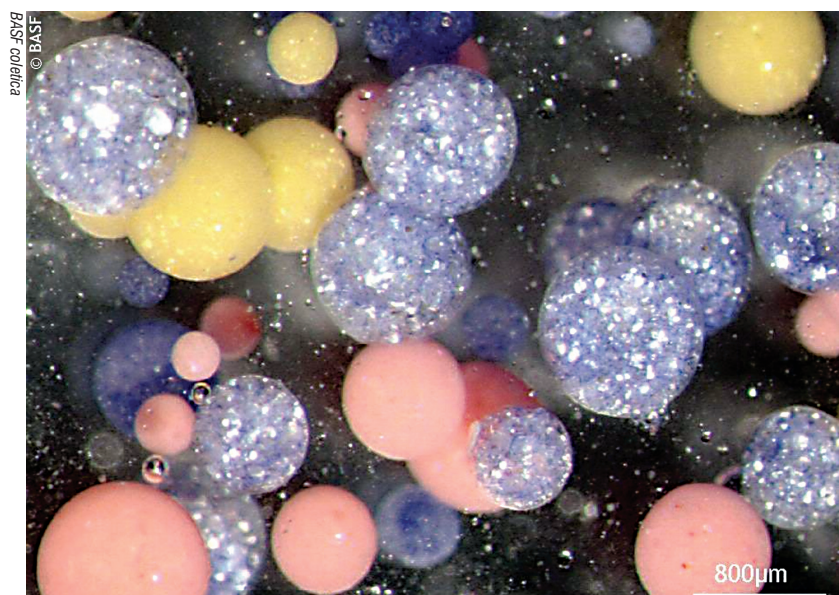
technologies, leading to a large spread of capsule diameters. In the last decade, however, new technologies, such as nozzle resonance technology or liquid jet cutting, can generate tonnes of quasi monodispersed capsules of a few hundred microns in diameter. These newer technologies will develop industrially in the next few years, and produce capsules suitable for biomedical appli-

cations, such as bio-artificial organs as well as biotechnology, cosmetics or personal and home care uses.

Achieving the best release profile for a particular microcapsule depends on the application. Often, a zero order release is needed, as in the case of drug delivery or agrochemical release. This may be related to a specific trigger like pH – as for certain drugs released in the body's intestines – or temperature – say, of spices on de-frozen pizza. Alternatively, the delivery may be linked to mechanical forces such as occur on using so-called 'scratch 'n' sniff' fragrance patches or shampoos. Speciality chemicals firm Cognis is the firm behind several such cosmetic and personal care products currently on the marketplace, for example.

Occasionally, the release is done stepwise, as in the case of US firm Salvo's *MultiSal* system, a family of microspheres containing one or more functional ingredients encapsulated in different compartments which can be adjusted to release the ingredients consecutively. The release can be triggered by water, pH change or various other factors.

Protecting and releasing even a single component may be a challenge. The first developed method, still often used,



**Looking good: colourful capsules for personal care**

involves using external pressure to break the microcapsule wall and release the liquid from the core. This method is applied in pressure-sensitive copying papers (pressure of the pen-ball or typewriter head), multi-component adhesives (activated in a press), deodorants and fungicides for shoes (release on mechanical pressure caused by walking), polishing pastes (rubbing) and aromas and sweeteners in chewing gums (chewing).

In other applications, the microcapsule wall breaks because of inner pressure, as, for example, for blowing agents in the production of light plastic materials and synthetic leather. Dissolution at selected pH value, meanwhile, is useful for microencapsulated catalysts and pharmaceuticals. Drugs, vitamins, minerals, essential amino acids, fatty acids, or even whole diets, can be released into the gastrointestinal tract by enzymatic degradation of digestible microcapsules.

Abrasion of the microcapsule wall is yet another mechanism, used for example to deliver anti-statics and fragrances for textiles.

In many applications, core materials are released by heat. Heat-sensitive recording papers, temperature indicators for frozen food, heat-sensitive adhesives, textile softeners and aromas for tea and baking, all deliver their effects via the melting of microcapsule wall. Microencapsulated fire retardants or extinguishers, released by burning, are used in fire-proof materials, including wallpaper, carpets, curtains, fire-protecting clothes, and coatings for electric devices and wires.

Microcapsules in special photographic emulsions, light-sensitive papers and toners for photocopiers, are decomposed, or hardened, by light. If the wall is permeable, it slowly releases the content of the core. This mechanism can be applied in hormonal drugs, aromas, fragrances, insecticides and fertilisers.

A further example are microencapsulated phase-change materials for active accumulation and release of heat in textiles, shoes and building insulation materials. To remain functional over numerous phase transition cycles, they have to remain encapsulated within the microcapsule wall over the whole product life.

The success of an encapsulation process is often linked to a know-how of the formulation or of the chemistry to achieve stabilisation. This is especially

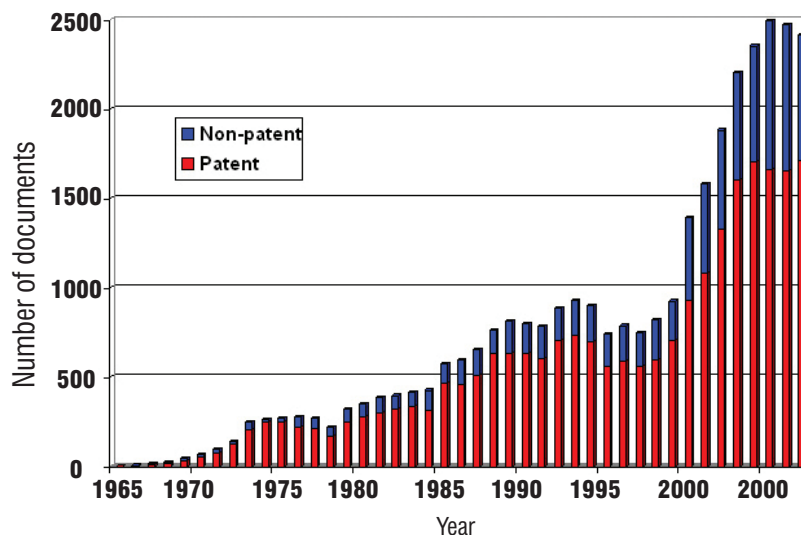


Fig 1: The number of encapsulation-related publications is increasing rapidly

the case in the food industry where the number of acceptable materials is very limited. Swiss firm MCC, for example, has developed expertise in making capsules coated with melted food-grade materials, used in the production of hundreds of tonnes of coated mineral food supplements by German company Dr Paul Lohmann.

To be commercially successful, an encapsulation process must be either small production but high value, in the case of cosmetics or biomedical applications, or large volume but low cost, such as in the food or detergent areas.

and using vegetable oil to replace the traditional system carried out at high pHs and in organic solvents.

Japanese company Shin-Etsu has also developed a dry powder coating process to form the coating. It works without solvent, including water, and speeds by up to five-fold the process of coating production. Initially limited to expensive materials suitable only for pharmacy, we have recently extended the system to make a polysaccharide coating, which should open up applications in the food domain.

New technologies to produce micro-

*'In terms of capsule manufacture, narrow size distribution is a must, allowing better control of properties such as mechanical resistance'*

One way to improve the efficiency of microencapsulation processes is to move to continuous systems such as those pioneered by French bioencapsulation and coating company Capsulae and German firm Glatt International.

Such processes not only promise to be quicker and cheaper, with very low energy consumption, compared with traditional batch processes, but would also reduce the processing cost three-fold, according to our evaluation.

One of the other concerns of researchers is to develop environmentally clean processes, often replacing organic solvents by water. In France, one of the author's research groups has developed an alternative approach to produce polymer microcapsules by interfacial reaction, working at mainly neutral pH

capsules will undoubtedly broaden the spectrum of possible applications for this versatile microencapsulation technology.

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The Bioencapsulation Research Group (<http://bioencapsulation.net>) is a worldwide platform promoting collaboration in the bio- & microencapsulation domain. It organises industrial workshops, technology trade fairs and international conference on encapsulation.