Microencapsulation at an Affordable Price

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Microencapsulation is the process of enclosing a substance inside a miniature capsule. Extremely tiny droplets, or particles of liquid or solid material, are packaged within a second material or coated with a continuous film of polymeric material for the purpose of shielding the active ingredient from the surrounding environment.

These capsules, which range in size from one micron to seven millimetres, release their contents at a later time by means appropriate to the application. The ingredients to be coated are referred to as core, internal phase (IP), encapsulate or fill, whereas terms applied to the coating of the microcapsules include the wall, shell, external phase or membrane.

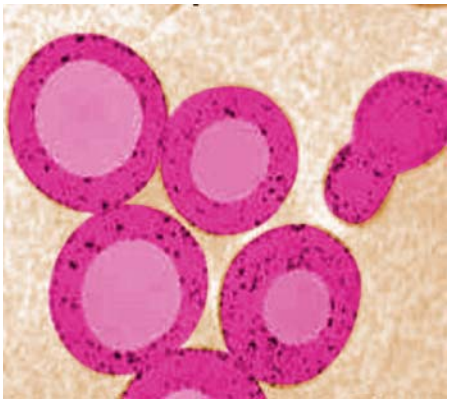
All three states of matter, ie. solid, liquid and gases, may be encapsulated and affect the size and shape of the capsules. If a solid or a crystalline material is used as the core, the resultant capsule may be irregularly shaped. However, if the core material is a liquid, simple spherical capsules, containing a single droplet of the encapsulate, may be formed.

The capsulated particles produce their required effect when their core material is released. There are four typical mechanisms by which the core material is released from a microcapsule:

- Mechanical rupture of the capsule wall
- Dissolution of the wall
- Melting of the wall
- Diffusion through the wall.

The microencapsulation technique was brought to use for the first time in 1940 by B.K. Green, for the production of No Carbon Paper (NCR). Later, in 1942, he ingeniously combined two contemporary techniques for the preparation of solid gelatin spheres and

Figure 1 – Cross-sectional View of a Microcapsule



the inclusion of an oil phase within a gelatin coacervate, and prepared the first gelatin capsules. The technique took nine long years to develop, from the laboratory to commercial use, and the new printing system was triggered by including a colourless dye base, contained within the oil phase, while the second sheet of paper was coated with acidic clay, which would react with the dye base on top to produce a colour.

Advantages of Encapsulation

Encapsulation of active ingredients is carried out for one or more of the following purposes.

1. Rendering liquids into powders, to prevent clumping and improving mixing.
2. Protecting active ingredients from oxidation, heat, acidity, alkalinity, moisture or evaporation.
3. Preventing ingredients from interacting with other compounds in the system, which results in their degradation or polymerisation.
4. Masking the taste of unpleasant flavours or odours.
5. Improving handling of an ingredient before processing.
6. To release active ingredients in a controlled or targeted fashion.
7. Protecting workers or end users from exposure to hazardous substances.

Choice of Encapsulation Technique

The choice of the encapsulation technique depends on factors such as:

1. The functionality the capsule needs to provide in the finished product.
2. The type of coating material, ie. the coating material should not react with either the ingredient to be encapsulated, or the formulation in which the capsulate will be added.
3. The processing conditions the encapsulate must survive before releasing the contents.
4. Concentration of the core material in the microcapsule.
5. The mechanism of release of the active agent from the microcapsule, eg. agitation, pH, pressure, solubility, time, etc.
6. The type of release (targeted, sustained or controlled) of the active ingredient.
7. The particle size, density, and stability requirements for the active ingredient.
8. The cost of the capsules and the cost of the formulation or application into or onto the final product.

Encapsulation Techniques

Many techniques are commercially available for the microencapsulation process and they can be broadly classified as follows:

1. Spray Coating Method

a. Pan coating
b. Fluid bed coating
c. Wurster air suspension coating

2. Wall Deposition from Solution
a. Complex coacervation
b. Organic phase separation coacervation
c. Hydroxypropylcellulose encapsulation
d. Urea-formaldehyde encapsulation (3M)

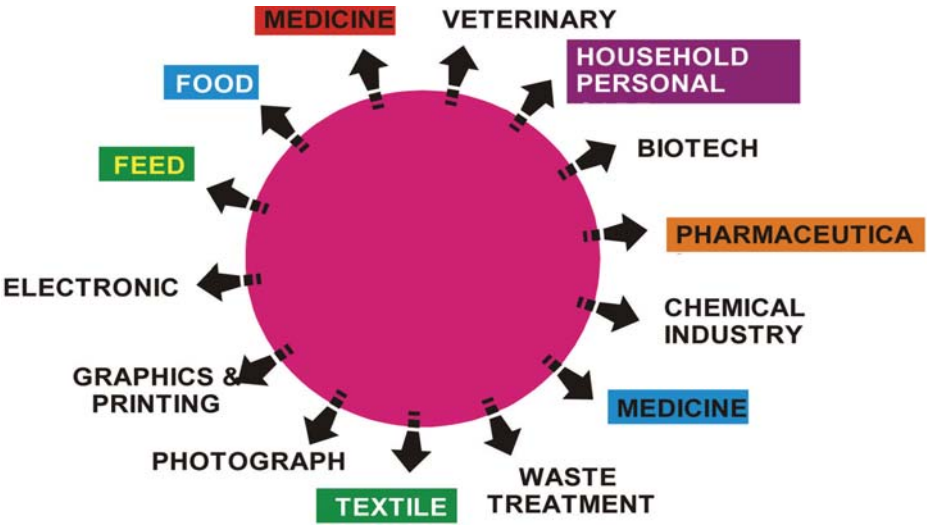
3. Interfacial Reaction
a. Interfacial polycondensation
b. Isocyanate process
c. Parylene free radical condensation (Union Carbide)
d. Alginate polyelectrolyte membrane (Damon)
e. Direct olefin polymerisation (National Lead)
f. Surfactant cross-linking (Champion)
g. Clay-hydroxy complex walls (Ryan)
h. Protein cross-linking (Frippak)

4. Physical Processes
a. Vacuum metallisation
b. Annular – jet encapsulation (SWRI, 3M)
c. Liquid membranes (Exxon)
d. Gas-filled capsules (Materials Technology)
e. Fast-contact process (Washington University)

5. Matrix Solidification
a. Spray drying
b. Spray cooling
c. Emulsified-melt solidification
d. Solvent evaporation (Fuji, Southern RI)
e. Starch-based processes (USDA)
f. Nanoparticle formation (Speiser, Krauter)
g. Cellulose acetate particles (Moleculon)

Spray Coating: This technique, in which particles are coated by dissolved or molten polymers, suspended in an upward-moving air stream in an unstable equilibrium, is used for encapsulation of very fine particles. The process simultaneously applies and hardens the wall materials on to the particles.
Wall Deposition from Solution: The core material is first suspended in a solution of the wall material, and the wall is induced to separate as a viscous liquid phase. This process is also called the coacervation process and is used to encapsulate water-insoluble liquids, solids or dispersions. Coacervation is recognised by the appearance of turbidity, droplet formation, or actual separation of liquid layers.
Interfacial Reaction: The process involves classical interfacial polycondensation polymerisation, which is widely used to produce synthetic fibres and films such as polyester, nylon and polyurethane.

Figure 2 – Applications of Microencapsulation



Physical Process: The process involves the pumping of the fluid core material through a central tube, while liquefied wall material is pumped through a surrounding annular space. A membrane of wall material is formed across a circular orifice at the end of the nozzle, and the core material flows into the membrane, causing extrusion of a rod of material. Droplets break away from the rod and hardening takes place on passage through a heat exchanger.
Matrix Solidification: Microencapsulation is achieved using spray-drying techniques, by atomising a combined solution of core and wall material. The atomised droplets are dried in a hot-air stream. Where the wall material is a melt or fat, the droplets are solidified using a cooled air stream and the process is called spray chilling.

Microencapsulation for Textiles

The textile industry, which was initially slow to exploit the technology of microencapsulation, has now produced a wide variety of innovations that utilise the basic principles of the targeting, slow release and protection of particles of sensitive chemicals, which are then used to give a pronounced effect to the fabric. The properties imparted to the fabric by encapsulation cannot be found in normal fabrics.

It can achieve several effects:
a. Antimicrobial fabrics: These finishes are used for the prevention of microbial attack on the fabric. A biocide is the active ingredient in this case, and is released by the appropriate means to achieve the desired antimicrobial finish.
b. Insect repellent fabric: The insect repellent is the core material and is encapsulated and later applied on the fabric. The fabric repels insects when the microcapsules are broken and the core is released during wear.
c. Fresh fabrics: This imparts a fresher and pleasant touch to the fabric.
d. Cosmeto-textiles: Aloe vera or other such products are encapsulated to give skin moisturising or to render a personal sense of wellbeing.
e. Photochromic textiles: Photochromic dyes, which change colour in response to UV light, are incorporated inside the microcapsules. These find application in product labelling, etc.
f. Thermochromic textiles: The encapsulated dyes change their colour in response to temperature. Currently it is possible to produce colour change formulations in the range of -250°C – 660°C.
We at Sarex have used the microencapsulation technique to impart a mosquito-repellency finish to our fabric. The details of the process and the test results are as follows:

- Material – 100% cotton, polyester/viscose and 100% polyester fabrics.
- Application – Pad-dry method, using laboratory pneumatic padding mangle (80% pickup) and laboratory mini-stenter.
- Recipe –
  - For 100% cotton: Saraguard MOSQ (30 g/l) + Binder (50 g/l) + Saraprint AC (5 g/l)
  - For polyester/viscose: Saraguard MOSQ (50 g/l) + Saraprint AC (5 g/l)
  - For 100% polyester: Saraguard MOSQ (50 g/l)

- Evaluation - Mosquito repellency was evaluated using modified WHO/CTD/WHO/PES/IC/96.1 method. Laboratory-reared strains of the Culex species of mosquito are released in an Excito Repellency chamber, containing finished and unfinished fabrics, to observe any change in behaviour, in the form of moving away from the treated-fabric chamber to the untreated one. The observations were recorded after a period of 10 minutes and after 1/2 hour.
- SEM Analysis - The fabrics were further taken for analysis using Scanning Electron Microscope (Philips XL-30) to actually verify the presence of microcapsules. The SEM machine was operated at 10KV, with the specimen tilt of 45°.

The results are depicted in Table 1 and Figures 3 and 4.

The results obtained clearly indicate a 100% repellency, which is equivalent to the percent protection, imparted by the fabric for 100% cotton fabric, 70% in the case of polyester/viscose fabric and 60% in 100% polyester fabric. The presence of the microcapsules has been further proven by the SEM studies, carried out on treated (Fig. 4) and untreated polyester (Fig. 3). The treated fabric, when viewed under a scanning electron microscope, clearly shows the presence of the microcapsules bonded to the fibre surface.

Fig. 3 – Untreated polyester

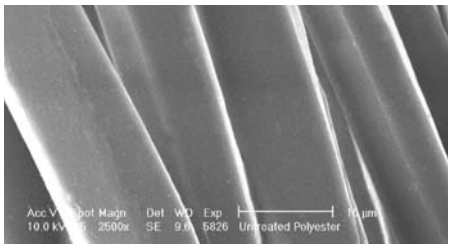
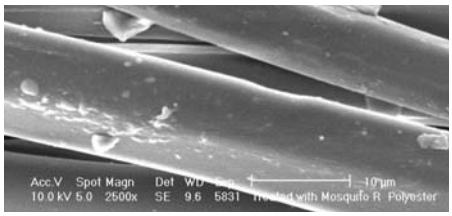


Fig. 4 – Polyester treated with mosquito repellent microcapsules



Further developmental work is being done in our laboratory to encapsulate products like fragrance, antimicrobial agents, flame-retardants and healthcare finishes, for ready and easy application on the fabric during finishing.

These microencapsulation finishes are very interesting but have not been used extensively in the textile industry due to the frightful costs involved. We at Sarex have been able to develop our own homegrown microencapsulation technique and, as a result, it can be offered at less than 50% of the current cost of microencapsulation!!! ❍

Table 1: Results of Modified WHO/CTD/WHO PES/IC/96.1 Test

Sample	No. of Mosquitoes released in treated chamber	No. of Mosquitoes on treated fabric	No. of Mosquitoes on untreated fabric	No of Mosquitoes showing mobility	Percentage Repellency
Cotton Finished (initial)	10	1	4	5	90
Cotton Finished (after 1/2 hour)	10	Nil	8	2	100
P/V Finished (initial)	10	3	5	2	70
P/VFinished (after 1/2 hour)	10	3	6	1	70
Polyester Finished (initial)	10	4	5	1	60
PolyesterFinished (after 1/2 hour)	10	4	6	Nil	60