



## Microencapsulation and its Application in Textile Wet Processing: A Review

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MANY methods for eco-friendly textile processing have been developed by science, including enzymatic textile finishing, plasma technology, natural product finishing, and microencapsulation. Microencapsulation is an innovative technology that has been utilized to give textiles functional features including antibacterial activity, scent, mosquito repellency, UV protection, and thermoregulation. The volatile and non-volatile components can be contained inside a thin polymeric film in microencapsulation, resulting in a delayed release of the chemical and a long-lasting functional impact. This review study focuses on the primary causes for microencapsulation, key microencapsulation techniques, and applications of microencapsulated goods in many fields of science and technology.

**Keywords:** Microencapsulation; pre-treatment; dyeing, finishing.

### Introduction

Environmental demands and concerns, as well as crises for environmentally friendly textile processing, have prompted the development of a slew of new, greener, and cleaner technologies, particularly following the collaboration of textile manufacturers and research laboratories to develop a variety of eco-friendly textile finishes. [1]

Many methods for eco-friendly textile processing have been developed by science, including enzymatic textile finishing, plasma technology, natural product finishing, and microencapsulation. [2-42]

Microencapsulation has become a difficult way to develop novel biotechnological materials. Microencapsulation is the process of forming small “packaging” known as microparticles, microspheres, or microcapsules, which are made up of structures that contain one or more bioactive compounds or are immobilized by one or more polymers.[43]

Encapsulating liquid droplets, solid particles, or gas molecules in an encapsulating agent/matrix/wall material is known as microencapsulation. These chemicals are completely encapsulated in a covering material or integrated into a homogeneous or heterogeneous matrix to generate tiny capsules with a variety of characteristics. The core and the shell/wall of microcapsules are split into two portions. [1, 43, 44]

The microencapsulation technique can protect active chemicals from potentially harmful conditions such as oxidation, heat, acidity, alkalinity, moisture, or evaporation.[45]

### Reasons for microencapsulation

Microencapsulation of materials is used to guarantee that the encapsulated material reaches the action region without being harmed by the environment it travels through. Amongst the principal reasons for encapsulation are:

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1. Reduce the reactivity of the material that is being encapsulated,
2. Prevent evaporation or loss of the enclosed substance into another media,
3. Make the encapsulated material easier to handle, apply, and store,
4. Promote controlled release (sustained or delayed-release),
5. Masking of odor, taste, and activity of encapsulated materials [43, 46]

#### *Morphology of microcapsules*

Microcapsules have two parts: the core and the shell. The core (intrinsic component) contains an active substance (for example, a hardener), whereas the shell (extrinsic part) shields the core from the external environment permanently or temporarily. ( see Figure 1) [47]

#### *Core material*

The core material, which is defined as the exact substance to be coated, might be solid, liquid, or gaseous. The major forms of the core materials are solution, dispersion, and emulsion. Because the liquid core might contain scattered and/or dissolved material, the core material composition can vary. Active ingredients, stabilizers, diluents, excipients, and release-rate retardants or accelerators can all be found in the solid core. The capacity to change the composition of the core materials gives significant flexibility and utilizing this feature frequently enables for effective design and development of desired microcapsule features. [44, 47]

#### *Shell material*

Diffusion, permeability, and controlled-release applications all benefit from shell type and shape. [47] The physical and chemical qualities

of the resulting microcapsules/microspheres are determined by the shell material selected. [44]

#### *Classification of microcapsules*

Microcapsules can be classified based on their size or morphology.

#### *Micro/Nanocapsules*

Microcapsules range in size from a thousandth of a millimeter to a few millimeters. Nanocapsules are microcapsules with a diameter in the nanometer range to emphasize their microscopic size.[48]

#### *Morphology microcapsules*

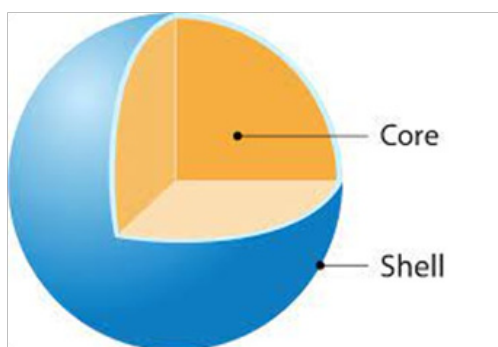
Microcapsules can be classified into three basic categories as given below:

1. Mononuclear microcapsules have a single hollow chamber within the capsule.
2. Polynuclear microcapsules have several different sized chambers within the shell.[48]
3. Matrix type microparticle The active chemicals are embedded in the shell material's matrix. However, the shape of a microparticle's interior structure is primarily determined by the shell materials chosen and the microencapsulation processes used (see Figure49). [2 ]

#### *Methods of microcapsules*

The methods listed below can be used to make microcapsules (see Figure 3):

For the manufacturing of microcapsules, several methods have been proposed. The technique chosen is determined by the core release rate, the permeability of the shell, its thickness, solubility, and physical qualities. Physical, physicochemical, and chemical microencapsulation techniques are grouped into three groups. [51]



**Fig.1. Morphology of Microcapsules.**

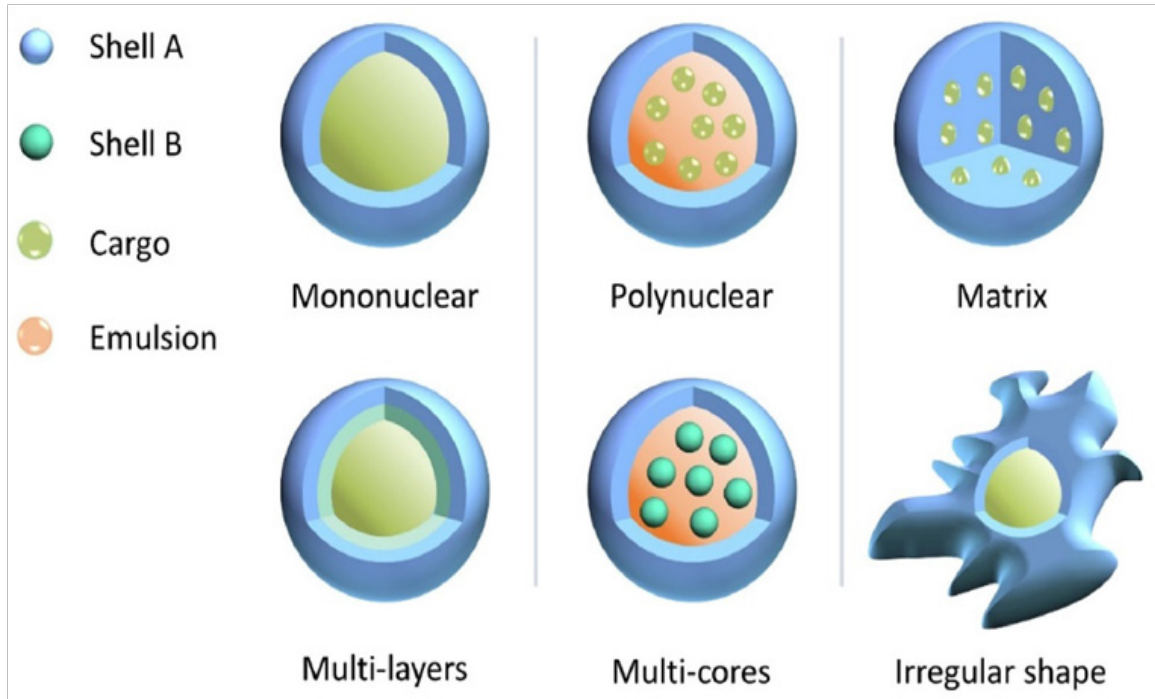


Fig. 2. Morphology of microcapsules [50].

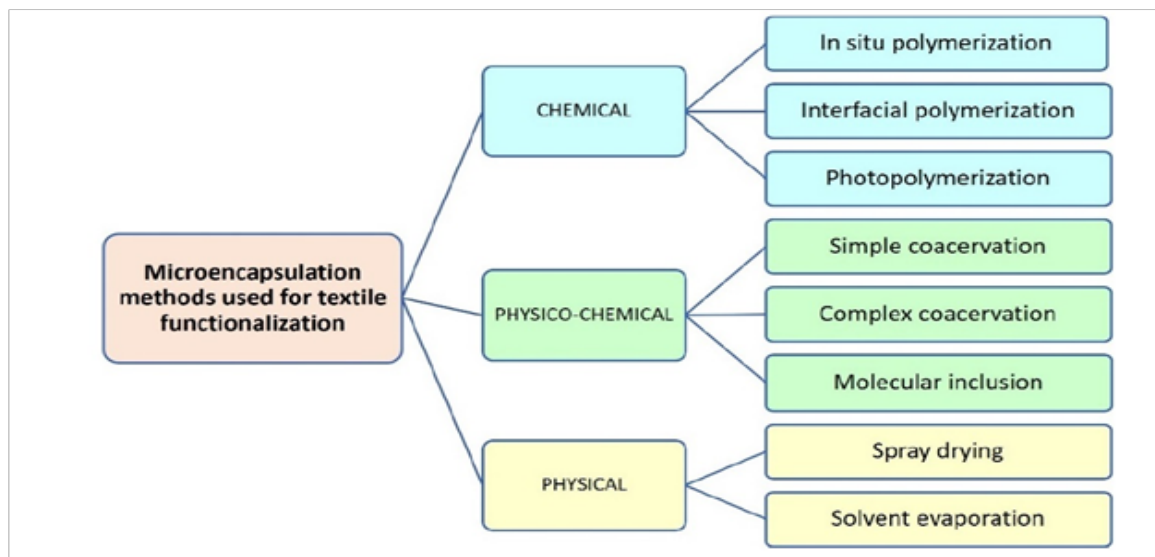


Fig. 3. Microencapsulation methods commonly used in textile [52].

*Physical methods*

*Spray drying*

Spray drying involves spraying an emulsion of shell and core material into a heating chamber with highly tuned atomization, where the solvent evaporates quickly, resulting in capsules. The phases in the microencapsulation process are as follows:

- Using an atomizer to spray the emulsion into minute droplets at a steady rate.

- Using hot gas to dry the previously distributed droplets.

- Using cyclones and filters to collect and separate the capsules. [51]

*Pan Coating*

The core particle is put into the pan, and the polymer is slowly injected within the pan, which rotates at a slower speed to ensure that the core material is properly coated.[44]

*Air Suspension coating*

It entails dispersing the core material in a supporting air stream and spraying the air suspended particles with a coating.[53]

*d. Centrifugal extrusion*

This system uses a concentric feed system with nozzle heads for both the core and shell parts. When the active core in liquid form is deposited in the nozzle's center, and the polymer in melt or solution form is inserted in the nozzle's shell, vibration occurs, followed by curing activity, resulting in the production of microcapsules.[54]

*Solvent evaporation method*

The active core ingredient dissolves slowly in the dissolved polymer volatile solution. The solvent is evaporated from the solution, leaving the microcapsule. [44]

*Physical-chemical methods*

**Coacervation** The macromolecular colloid-rich coacervate droplets produce a viscous microcapsule wall that is solidified using cross-linking agents and surrounds scattered microcapsule cores. This is a phenomenon that occurs in colloid systems. [52]

- i) Simple coacervation: A desolvation agent is used to separate the phases in simple coacervation.
- ii) Complex coacervation: When solutions of two hydrophilic colloids are combined under proper circumstances, complex coacervation refers to the phase separation of a liquid precipitate or phase. The three phases of the solution are formed, and the solvent evaporates, leaving the sheath over the core material. [55]

*Chemical methods*

**Polymerization** The monomers in an emulsion polymerize around the droplets and create a solid polymeric wall. **Polymerization in situ** Only the aqueous phase of the emulsion receives monomers or pre condensates. **Polymerization at the interface** One monomer is dissolved in water, while the other is dissolved in a lipophilic solvent. [55]

*Techniques of microencapsulation**Microencapsulation Process*

It's a method of encasing the core components (gases, solid particles, and liquid droplets) in the wall materials (polymers). The core components are completely covered by a thin layer of wall material or integrated into a polymer matrix, resulting in a micro-size capsule with a variety of

useful features.[56] The interior phase, core, filler, or active microcapsules is referred to as an internal phase, while the encasing material is referred to as a shell, coating, membrane, wall, or carrier. [55]

Various materials can be encased within macro-packages that keep them safe from the elements. As a shell material, a variety of synthetic and natural polymers can be used. The physicochemical properties of the core material influence the shell material selection. A hydrophilic core requires a hydrophobic shell, whereas hydrophilic shells require a lipophilic core. Natural polymers with great potential for usage as encapsulating agents include cellulosic materials, starch, Arabic gum, dextrin, chitosan, alginate, gelatine, and natural gum.[51, 56]

*Release mechanisms*

Encapsulated materials have various release mechanisms that allow for controlled, sustained or targeted release of the core substance. Mechanical rupture in the capsule wall, wall material becoming dissolved in the changing environment, or wall melting due to temperature change are three ways core material gets released from the capsules low wall erosion (ablation) and biodegradation are less well-known processes. Insect repellents, cosmetics, fragrances, deodorants, and medical textiles are some of the applications for these long-term textile materials. The migratory molecular weight can also be used to classify the release process (low or high). [46, 57]

There have been eight distinct release mechanisms reported to achieve such effects in permeability and non-permeable microcapsules.

- a) External pressure: The microcapsules are mechanically broken as a result of this process.
- b) Internal pressure: might potentially cause the microcapsule wall to shatter, for example, if the core shell includes compounds that, under certain conditions (e.g., radiation activation), are transformed into gaseous products, as in the case of the manufacturing of light synthetic leather.[58]
- c) Microcapsule walls abrasion: In the case of scent release, this method is commonly utilized.
- d) Burning: When the temperature rises to a certain level, fire retardants are released.[59]
- e) Radiation: This method can trigger photographic and light-sensitive processes, resulting in changes in the color of these fabrics due to the

- release of microencapsulated dyes.
- f) Temperature Changes: Temperature variations can aid core material release. There are two separate processes for release:
    - Temperature-sensitive: When the crucial temperature is reached, the wall expands and falls.
    - Fusion-activated: The wall dissolves as the temperature rises.[60]
  - g) Chemical reactions: This is the situation with microcapsules containing chemicals added to textile washing or cleaning formulations that are released throughout the wash cycle due to chemical composition or pH changes.
  - h) Enzymatic degradation: This is the case with microcapsules, which are destroyed by enzymes under strict circumstances.[57]

*Methods for Application of Microcapsules on Textiles*

Microcapsules can be mechanically introduced to the fabric at various phases, from the polymer to the fabric stage.

- a) In the polymer, (During the spinning process, microcapsules are inserted into the fibers).[61]
- b) In the padding method, The cloth is transferred by microcapsule dispersion and then pushed through cushioning rollers to remove any surplus liquid.

- c) The immersion method The cloth is not passed through the squeeze rollers as it does with the padding technique.[51]
- d) In the printing method, Microcapsules are mixed with a binder to create the printing paste, which is then applied to the cloth (by Screen, Photographic, Electrostatic, Pressure-Transfer, Thermal Transfer, and Inkjet Printing Techniques).[62]
- e) In the coating method, On the fabrics, a homogenous coating of microcapsules is placed.
- f) In the spraying method, The microcapsules are sprayed on the fabric with a spray nozzle in a closed chamber, and then heat treatment at a high temperature (130-170°C) is used to stabilize the microcapsules on the fabric. [51, 63]

*Application of Microcapsules for Functional Textiles*

Textile product functionality is one of the most important market demands nowadays. Since the mid-twentieth century, The most widely used technology for functionalizing textile materials is microencapsulation.[51] Textiles with novel improved qualities, such as medical textiles, are developed and modified using the microencapsulation process, Antimicrobial Textiles, Aroma/Fragrant Textiles, and Insect/Mosquito Repellent Textiles. (see *Figure 4*) [64]

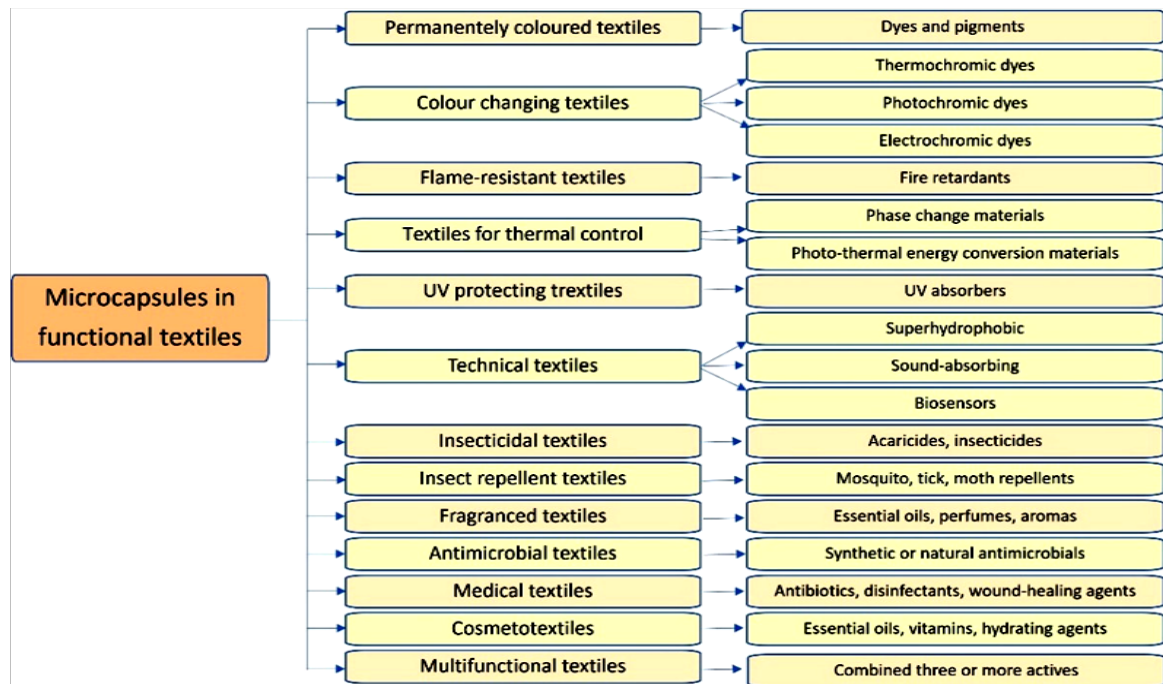


Fig. 4. Functional textiles based on microencapsulated actives [52].

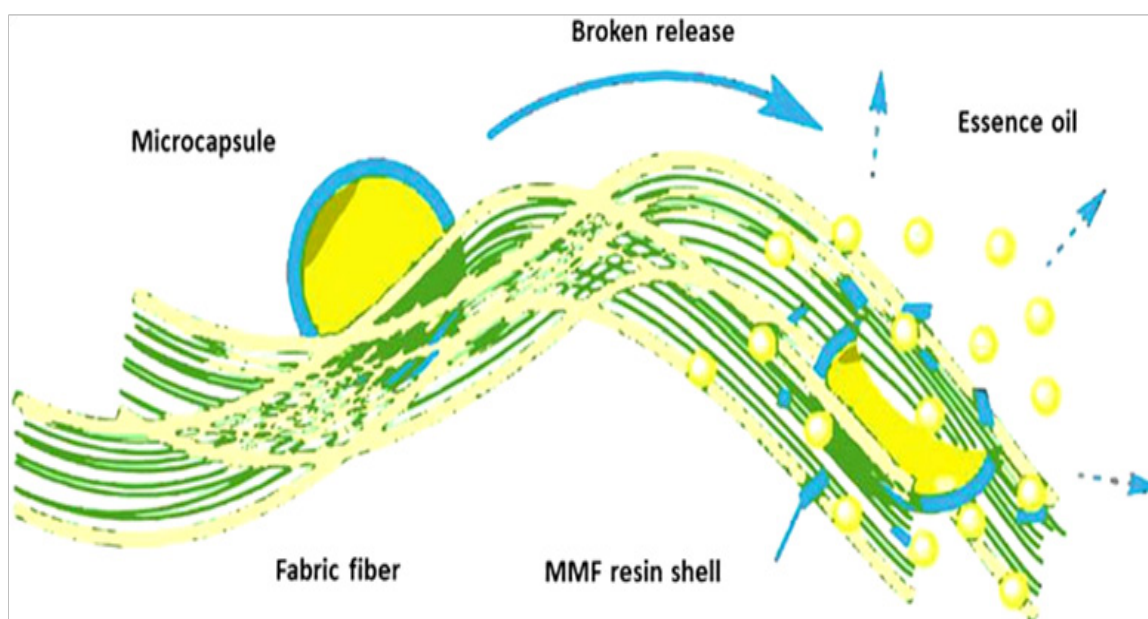
### *Aroma/Fragrant Textiles*

Fragrance/Scent finishing is one such growing sector that adds value and utility to textile products by incorporating aroma into them. The perfumes produced from plant sources, i.e. essential oils, not only have a nice smell but have also been used as antiseptics, anti-inflammatory, antimicrobials, and emotional soothing.[65, 66] Aromatherapy textiles include essential oils generated from plant-based raw materials, which can help to improve the body's mental and physical state. Microcapsules burst as a consequence of mechanical pressure on the capsules during wear, releasing the active component (see Figure 5). To create scent textiles, several research studies have been conducted.

Sharkawy *et al.* used chitosan/gum arabic to make limonene and vanillin microcapsules via a sophisticated coacervation technique. Tannic acid was used as a hardening agent for the shell material. Two different emulsifiers, polyglycerol polyricinoleate (PGPR) and Span 85, were studied for their encapsulation effectiveness, microcapsule morphology, and release profile. The size of the prepared microcapsule was between 10.4 and 39.0  $\mu\text{m}$ . The use of Span 85 resulted in mononuclear morphology regardless of the essential oil used, whereas the use of PGPR resulted in polynuclear morphology. An esterification process with citric acid was used to graft the microcapsules onto the cotton fabric, which was then heat-fixed and cured. Antibacterial characteristics were detected in the completed cloth. [67].

K. J. Sannapamma *et al.* prepared Vetiver oil microcapsule for application on organic cotton knit fabric. The interfacial polymerization process was utilized to make microcapsules with vetiver essential oil, and the pad dry cure method was employed to apply microcapsules to the organic cotton knit fabric. In comparison to knitwear completed by the exhaust, organic cotton knits finished with microcapsules by pad dry cure technique demonstrated the greatest zone of inhibition. Multiple washing cycles were used to test the antimicrobial activity of the completed samples, which revealed that knits finished with the pad dry cure technique had a zone of inhibition even after the 20th wash and had greater scent retention than knits finished with the exhaust way of application. The vetiver microencapsulated organic cotton knits are chemical-free and have multi-functional qualities that make them ideal for medical and healthcare textiles.[68]

Interfacial polymerization was used to microencapsulate methyl central ketone (MCK) with polyurethane urea (PUU) polymers by Coskun Mertgen *et al.* The primary component of the capsule shell, PUU (isocyanate/PEG-400/polyamine) mole ratios (5.4/3.0–5.5/1.5–3.9), was synthesized in 240 minutes at 80 °C. The encapsulation efficiency of the microcapsules generated, CMK-PUU microcapsule release at normal temperature, microcapsule shell deterioration at high temperature, and adhesion durability, when fixed on cotton, polyester, silk, and non-woven fabric, were all tested.[69]



**Fig. 5.** Release of aroma microcapsules under external force.

Tekin et al. used interfacial polymerization to create polyurethane-urea microcapsules containing fabric softener scents as active agents, extending the release rate and increasing the fragrance's durability. It was also discovered that raising the emulsion's stirring pace caused microcapsule breaks, which might lead to the scent being released more quickly. During the emulsion process, the agglomeration rate rose as well. Optical and SEM data indicated that raising the stirring rate during the emulsion stage resulted in lower particle size microcapsules, however, it has an undesirable effect on their shape at a certain point.[70]

#### *Antimicrobial Textiles*

Microbial development on textiles can result in unpleasant odors, skin discomfort, discoloration, and other issues. Microbes thrive in environments with high moisture content and cellulose content, such as cotton fabrics. Antimicrobial finishing is a chemical treatment that kills or stops microscopic organisms from growing.[71-73]

Juliana Oliveira Fiedler et al. created Aloe Vera microencapsulation with cornstarch in cotton nonwoven fabric utilizing the basic coacervation procedure and butane tetracarboxylic acid (BTCA) as a binding agent. The microcapsules, in combination with a simple coacervation process, perfectly accomplished the goals. Micrography, thermograms, o.w.f., and Tw indexes are proofs of this assertion and show that the finishing adheres to the substrate.[74]

Senthilkumar Boominathan et al. used the Box Behnken experimental design technique to improve the combination of antibacterial and mosquito repellency treatment on woven cotton fabric using microencapsulated *Coleus aromaticus*. Gum acacia was used as the shell material and *Coleus aromaticus* leaf extract as the core material for the microcapsules. Pad-batch approach was used to apply these capsules to the Cotton fabric. Microcapsule concentration (g/l), BTCA concentration, and curing temperature are three process factors that have been optimized. The treated textiles were tested for antibacterial activities as well as mosquito repellency. Following that, a polynomial equation for both antibacterial activity and mosquito repellency behavior was created. The effects of each process variable on the response were investigated using RSM analysis. The optimal process parameters were a microcapsule concentration of 40 g/l,

a BTCA concentration of 67.5 g/l, and a curing temperature of 116°C. The combined desirability coefficient was discovered to be 0.792.n[72]

SJ Kudligi et al. prepared microcapsules from Palmarosa oil using an interfacial polymerization approach and completed them on organic knitted fabric using exhaust and pad dry curing procedures. The effect of laundry on antibacterial effectiveness and scent intensity was evaluated in the final organic knit. Palmarosa microcapsules had a superior TGA with less weight loss at higher temperatures than pure oil, however, the shape of the microcapsules was very irregular owing to preparation circumstances. The antibacterial activity of the final samples was tested repeatedly, and results showed that knits finished with the pad dry cure technique had a zone of inhibition even after the 20th wash and better scent retention than knits finished with the exhaust method of application. As a result, the Palmarosa microencapsulated organic cotton knits are devoid of harmful chemicals and have multi-functional features that make them suited for medical and healthcare textiles.[75]

Dorra Dridi et al. imparted antimicrobial characteristics to cellulosic fibers by microencapsulation using citric acid as a green binding agent. Coacervation was used to encapsulate an essential oil combination utilizing chitosan as a wall material and sodium hydroxide as a hardening agent. The dimension of the microcapsules formed ranges between 12 and 48  $\mu$ m. Attenuated Total Reflectance-Fourier Transformed Infrared (ATR-FTIR) spectroscopy, optical microscopy, and scanning electron microscopy (SEM) examination were used to demonstrate the attachment of the generated microcapsules to the surface of cotton textiles. The dimension of the microcapsules formed ranges between 12 and 48  $\mu$ m. Attenuated Total Reflectance-Fourier Transformed Infrared (ATR-FTIR) spectroscopy, optical microscopy, and scanning electron microscopy (SEM) examination were used to demonstrate the attachment of the generated microcapsules to the surface of cotton textiles. [76]

In other studies, the complicated coacervation process was used to create LO microcapsules with chitosan and gum arabic wall components. The sophisticated coacervation process using gum Arabic and chitosan wall components were used to successfully manufacture lime oil microcapsules. The antioxidant and antibacterial properties of

lime oil were retained after microencapsulation. The lime oil microcapsules demonstrated antibacterial efficacy against all four bacterial species tested. Furthermore, microencapsulation masks the cytotoxic effect of unencapsulated lime oil. Succinic acid was used as a binder to successfully insert the manufactured lime oil microcapsules into the cotton fabric. Following the mechanical crushing of its microcapsules, the lime oil-infused cotton fabric demonstrated strong antibacterial activity. Even after a gentle washing, the cloth with lime oil microcapsules preserved its microcapsules and antibacterial action.[64]

#### *Insect/Mosquito Repellent Textiles*

The mosquito repellent textile product was recently created, and the process of preparation comprises the application of mosquito repellent compounds to the textile materials.[51] The mosquito is one of the most significant arthropod vectors of illnesses including malaria, yellow fever, Rift Valley fever, dengue fever, and arboviral encephalitis. These illnesses represent a danger to human and animal health, resulting in substantial mortality worldwide.[77, 78]

Repellents serve a vital part in several anti-mosquito goods, and while they are not widely utilized, they are necessary raw materials for the production of various anti-mosquito products. Insect repellents such as N, N-diethyl-metoluamide (DEET) can be applied directly to the skin or in conjunction with permethrin-containing textiles to provide mosquito protection.

Ramya et al. used an ionic gelation process to create andrographolide-loaded sodium alginate microcapsules, which were then exhausted onto a bamboo/cotton fabric. The mosquito repellency of the microcapsule-finished textiles was 94 percent, compared to 96 percent for the directly finished materials. After 30 washes, the repellency of microcapsule-finished textiles was reduced to 52% and 40% for directly finished fabrics, respectively.[79]

Geethadevi et al. used the ionic gelation process to create mosquito repellent microcapsules using three wall materials: Acacia arabica, sodium alginate, and Moringa oleifera gum, as well as a mixture of three core materials: grapefruit oil, cypress oil, and thyme oil. Even after 30 washing cycles, the microcapsules made with Moringa oleifera gum retained 60% mosquito repellency.[80]

#### *Medical Textiles*

Medical textiles are used for a variety of purposes, including first aid, clinical, pharmaceutical, and sanitary purposes. Medical textiles include bandages, gauzes, and tissue culture media, as well as body implants such as skin, artificial heart, blood artery, and heart valve.[51]

Hanaa Mohamed ElRafie et al. use eco-friendly algal volatile organic components to impart wound healing properties to cotton fibers, resulting in a bandage (VOCs). VOCs isolated from Egyptian maritime macroalgae *Digenea simplex*, *Lurencea papillosa*, *Galaxurea oblongata*, and *Turbenaria decurrens* were 0.52, 0.9, 0.87, and 0.62 % (v/w), respectively. Using sodium alginate (SA) as a shell wall material, these VOCs and their microencapsulated (VOM) forms were completed onto cotton textiles using a traditional pad-dry curing procedure. Gas chromatography coupled with mass spectrometry (GC-MS) was used to extract and analyze the VOCs of each alga. The findings reveal the diversity and striking variations in volatile composition among the four algae, in addition to the discovery of 125 volatile compounds. The completed textiles' wound-healing abilities were assessed. Textiles finished with VOCs microcapsules (VOMF) were more effective than fabrics finished with VOCs (VOF) and were almost similar to fabrics finished with mebo-ointment (standard medication) (MoF). The four algae have different types and amounts of volatiles, which might explain the discrepancies in VOC efficiency. As a result, obtaining encapsulated VOCs for use in textile wound healing is a low-cost, easy, repeatable, and scalable process.[1]

EOs such as chamomile blue, tea tree, lavender, lemongrass, peppermint, elicriso italic, lemon oils, cinnamon, and eucalyptus oils were encapsulated in a film of sodium alginate with glycerol as a plasticizer by Liakos et al. The composite wound dressing film revealed that the majority of EOs inhibit *C. Albicans* development. Peppermint oils, lemongrass oils, and cinnamon oils, on the other hand, have antibacterial activity against *E. coli*.[81]

#### *Multifunctional Textiles*

Textiles with two or more useful qualities are known as multifunctional textiles. The functional



qualities of microcapsules are obtained from the functional shell and active core components. These microcapsules have one or more functional features, such as antibacterial activity, antifungal activity, flame retardancy, thermochromic performance, electrical conduction, photoluminescence, and thermal stability, in addition to the principal functional property.[82]

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## الكبسلة الدقيقة وتطبيقاتها في المعالجات الرطبة للمنسوجات

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الكبسلة الدقيقة هي تقنية مبتكرة تم استخدامها لمنح المنسوجات ميزات وظيفية بما في ذلك النشاط المضاد للبكتيريا والرائحة وطرد البعوض والحماية من الأشعة فوق البنفسجية والتنظيم الحراري. يمكن احتواء المكونات المتطايرة وغير المتطايرة داخل فيلم بوليمر رقيق في كبسولة دقيقة ، مما يؤدي إلى تأخر إطلاق المادة الكيميائية وتأثير وظيفي طويل الأمد. تركز دراسة المراجعة هذه على الأسباب الرئيسية للتغليف الدقيق ، وتقنيات الكبسلة الدقيقة الرئيسية ، وتطبيقات السلع المغلفة في العديد من مجالات العلوم والتكنولوجيا. الكلمات الرئيسية: الكبسلة الدقيقة، المعالجة الأولية، الصباغة والتجهيز.