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Review Article

Microencapsulation and its application in production of functional textiles

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Currently, the textile sector is moving towards the development of value-added functional textile products based on customer demand. Therefore, several new finishing techniques have been evolved so far to functionalize the textile substrates. In this context, the microencapsulation technique is one of the advanced technologies which has been used to impart functional properties such as antibacterial activity, aroma, mosquito repellency, UV protection, and thermoregulation to textiles. In microencapsulation, the volatile and non-volatile components can be encapsulated within a thin polymeric layer which causes a slow release of the compound, resulting in long-lasting functional effect. Various active materials, like essential oils, enzymes, drugs, pesticides and vitamins, have been successfully entrapped inside microcapsules made from a variety of polymeric materials. There are interesting reports available in the literature dealing with functionalization of textiles using microencapsulated materials. This review paper covers the fundamentals of microencapsulation, its major techniques, materials involved in microencapsulation and the important research reported in this area. The major essential oils used in microencapsulation and the subsequent functional effects are also reviewed. The present and futuristic research trends in this important area of chemical processing of textiles are presented.

Keywords: Controlled release, Essential oils, Functional textiles, Microencapsulation, Polymers

1 Introduction

The utilization of microencapsulated functional materials for functionalization of textiles has facilitated the evolution of value-added textile products with new and improved properties, such as fragrance[1], thermoregulation[2], flame retardancy[3], UV protection[4], antibacterial[5], photochromic effects[6], antioxidant[7], or multifunctional ones by combining two or more functional elements[8,9]. Microencapsulation involves the deposition of thin polymeric layers on the microdroplets of liquids or small particles of solids. It is a packaging technology which packs the substances at a micro-level. Primarily, this technique was introduced in the paper industry as the basis of the carbonless copy paper and is currently explored in other industries, such as agricultural, pharmaceutical, chemical, cosmetics, toiletries, and food processing. Various types of microcapsules loaded with varieties of functional materials are available in the market[10,11]. This technique shows excellent possibilities for improving product and process performance. The improved performance, in terms of efficacy and durability, is offered by excellent stability of the volatile component against oxidation and a resultant controlled release mechanism.

The application of microencapsulated materials in the textile industry has been limited in the initial period because of lack of awareness regarding the scope of this technique and possible value-additions. Even though value-added textiles were prepared and reports regarding this are available in the literature, the practical difficulties faced during scaling-up has made it challenging to commercialize most of the microencapsulated materials. Apart from this, the high costs of processing is another limiting factor[12]. Since the last two decades, the application of microencapsulated materials on textile is gaining momentum; however, the application is restricted to common substrates used in interior textiles and clothing[13]. The vast possibilities of microencapsulation technology have not been entirely exploited, and hence the scope for investigation still exists.

Functionalities can be imparted to textiles by the application of a variety of encapsulated materials, including both natural and synthetic ones. The use of sustainable natural materials for functionalization of textiles is the growing interest and rising demand from modern consumers. In this case, the use of essential oils (EOs) as an encapsulated material can serve the requirements of sustainable chemical finishing of textiles. Apart from the advantage of being natural products, EOs offer high value-added properties because of their interesting

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physicochemical characteristics[14]. EOs have a broad spectrum of antimicrobial and biological activities because of their complex chemical composition, resulting in the presence of several functional properties like antiviral, antifungal, insect and mosquito repellents, antibacterial and pest control[15-17]. In the textile industry, microencapsulated material can be applied on fibres, yarn, and fabrics as a finishing agent using finishing techniques such as pad-dry-cure, exhaustion, screen-printing, impregnation, and spraying[18].

This paper reviews the important aspects of microencapsulation, including the fundamentals of microencapsulation, materials involved, and techniques of preparation. The reported literature dealing with functionalization of textiles using various microcapsules are thoroughly reviewed. Existing technology and futuristic trends are also discussed.

2 Microencapsulation Technology and its Application in Textiles

2.1 Microencapsulation Trends in the Textile Industry

The spray drying technique introduced the concept of microencapsulation and is known from the 1930s. In 1990s, the possibilities of microcapsules in finishing and colouration of textiles were explored, and that was the first application of microencapsulation in the textile industry[19-21].

The commercial use of microencapsulation in the textile industry was there from the beginning of the 21st century, specifically in Japan, Western Europe, and America. The technique was being used to introduce new and interesting properties in textiles, including antibacterial, flame retardant, and medicinal properties[22]. Nowadays, because of the success of microencapsulation in the textiles sector, the manufacturers are very much excited to capture the market opportunities by fulfilling the potential consumer demand[23,24].

2.2 Fundamentals of Microencapsulation

2.2.1 Microencapsulation Process

It is a technique to entrap the core materials (gas compound, solid particles, and liquid droplets) in wall materials (polymers) as shown in Fig. 1. The core compounds are fully covered with a thin film of wall material or embedded in a polymer matrix that forms a micro-size capsule with various important properties[13,25]. The content of microcapsules is described as an internal phase, core, filler or active, and the entrapping material of capsules is known as shell, coating, membrane, wall, or carrier[20-22]. Spears[26] have patented an invention based on encapsulation of hyperbaric gas using sonication method. The prepared microcapsules containing hyperbaric gas were used for providing systemic oxygenation of tissues and in the treatment of infections, atherosclerosis and neoplasms.

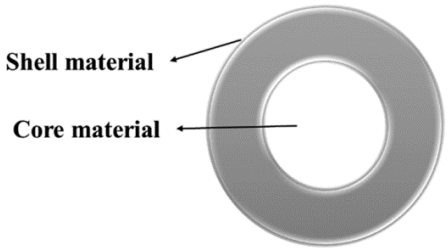

Fig.1 — Description of a microcapsule[13]

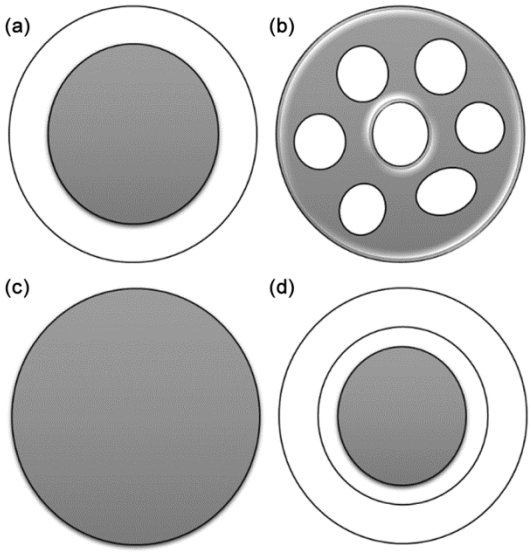

Fig. 2 — Microcapsules morphologies (a) reservoir, (b) polynucleated, (c) matrix and (d) multi-shell[29,30]

Various materials can be encapsulated within macro-package that protect them from the external environment[13]. Various synthetic and natural polymers can be utilized as a shell material. The choice of shell material depends on the physicochemical characteristics of the core material. The hydrophilic core needs a hydrophobic shell, and a lipophilic core is required for hydrophilic shells. The natural polymers, including cellulosic materials, starch, Arabic gum, dextrin, chitosan, alginate, gelatine, and natural gum, have high possibilities of the use as an encapsulating agent[27,28].

2.2.2 Morphology of Microcapsules

The morphology of microcapsules can be classified into four major microcapsules structures, viz reservoir, polynucleated, matrix, and multi-shell structures, as shown in Fig. 2. Morphology of microcapsules is

typically recognized through shape, core material, size, size distribution, and the delivery mechanism[31]. The shape of the microcapsules depends on the type of core material. If the core material is in crystal or solid form, then the resultant microcapsules would be of irregular shape. The liquid core may provide spherical microcapsules. In a matrix structure, many fine core materials are uniformly distributed in the shell material. Moreover, the microcapsules can be formed in the form of multi-core or multi-shell[27,32].

2.2.3 Release Mechanisms

The diffusion rate across the polymer layer depends on the specific characteristics of the polymer network, such as flexibility, plasticization extent, absorbency, chain length, ion extent, or possible interactions between active agent and polymer, and therefore the active agent release[33]. Figure 3 represents the EO-release through the polymeric layer of the microcapsule.

Martin del Valle *et al*.[34] suggested that the controlled release device can be formed when essential oil diffuses from the polymer layer of microcapsule. Generally, the release of core material from microcapsules is based on four mechanisms, viz. mechanical stimuli (during the application of mechanical force in processing), chemical stimuli (the core material slowly dissolves in shell membrane), thermal stimuli (heat), and diffusion[24,35].

2.3 Microencapsulation Techniques

Several techniques have been proposed for the production of microcapsules. The selection of the method depends on the core release rate, shell permeability, thickness, solubility, and physical properties. The microencapsulation techniques are divided into two categories, namely physical and chemical methods. The physical methods are further subdivided into physicomechanical and physicochemical methods. However, only commonly used techniques from each category have been described. The standard microencapsulation techniques, are shown in Table 1. Table 2 shows the advantages and disadvantages of commonly used microencapsulation techniques.

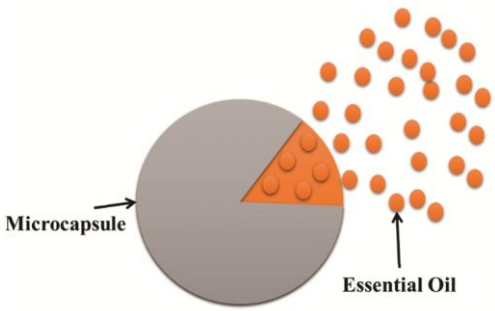

Fig. 3 — Representation of essential oil release from the microcapsule[36]

2.3.1 Spray Drying

Spray drying technique is based on spraying the emulsion of shell and core material into a heating chamber with highly optimized atomization, where rapid evaporation of solvent occurs, thereby producing the capsules. The process flow chart and spray drying equipment are shown in Figs 4 and 5 respectively. The microencapsulation process consists of the following steps:

- Spraying the emulsion into small droplets at a constant feed rate using an atomizer.
- Drying the previously spread droplets by hot gas.
- Collecting and separating the capsules using cyclones and filters.

Generally, the morphology of microcapsules prepared by spray drying is a matrix or polynuclear type[13]. Li *et al*.[49] prepared the chitosan microcapsules loaded with orange oil using spray drying technique and analyzed their stability towards washing with different detergents. They investigated the retention of oils in the microcapsules applied to cotton after certain days. De Oliveira *et al*.[50] attempted to develop a blend of biopolymer for the encapsulation of EO and prepared nanoparticles of alginate/cashew gum using spray-drying. The TGA/DSC and X-ray showed sufficient miscibility of both the polymers. Moreover, a release profile was achieved for over 30 h. Carvalho *et al*.[51] prepared green coffee loaded microcapsules via spray drying technique. The microcapsules displayed enhanced oxidative stability, excellent encapsulation efficiency, and SPF similar to the free green coffee oil.

Table 1 — Important microencapsulation techniques[22,30,37-42]

Physical methods		Chemical methods
Physico-mechanical	Physico-chemical	
Pan coating	Ionic gelation	*In-situ* polymerization
Extrusion	Coacervation	Interfacial polymerization
Air-suspension coating	Sol-gel	Polymer-polymer incompatibility
Ultrasonic atomizer	Supercritical Fluids	Polycondensation
Spray drying	Solvent evaporation	Emulsification
Microwave processing	Polyelectrolyte Multilayer	Liposome formation

Table 2 — Advantages and disadvantages of commonly used microencapsulation techniques

Techniques	Advantages	Disadvantages	References
Spray drying	Quicker, scalable, affordable, offers desired size of microcapsules, spherical and small particles with homogeneous distribution, stable microcapsules	Microcapsules agglomeration, uncoated core material, works on moderate viscosity of the emulsion	43,44
Coacervation	High encapsulation efficiency, Easy and affordable	Limited stability of microcapsules in a limited ionic strength and range of pH, works on limited polymer materials and microcapsules agglomeration	45
Extrusion	Tetter stability of oils against oxidation, low surface oil, prolonged shelf-life of essential oils and reduce evaporation rate of essential oils	Expensive process, large particles size	46
In-situ polymerization	Spherical and smooth microcapsules, offers good chemical, storage, and thermal stability and provides high encapsulation efficiency	Sensitive microcapsules to pressure due to transparent wall	47
Emulsification	Relatively easy and low cost	Physical instability (pH, heating, drying, and high mineral concentrations) particles and offers limited control release of essential oil	46

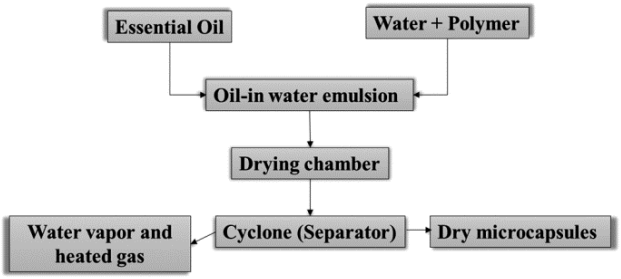

Fig. 4 — Typical process flow of spray drying encapsulation technique[48]

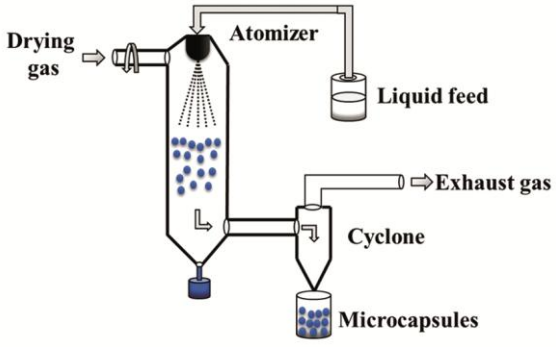

Fig. 5 — Schematic diagram of spray drying equipment[43]

2.3.2 Coacervation

The coacervation process can be either simple or complex. The interaction between the dissolved polymer and the low molecular compound results in the simple coacervation. When the two opposite charges of polymers interact with each other, the process results in the complex coacervation[52]. Figure 6 shows the process flow of complex coacervation technique. Han *et al*.[45] used complex coacervation for encapsulating the patchouli oil. The volatility of the oil was reduced, which stabilized the strong smell of oil and also prevented its oxidation.

2.3.3 In situ Polymerization

In this technique, the polymerization process is responsible for the formation of a capsule shell where a specific pre-polymers or monomers are suspended into a stable emulsion of the immiscible core material and a continuous solution. In this chemical technique, the formation of microcapsules does not require reagents.

The capsules loaded with perfume, carbonless paper inks and other components are extensively produced by *in situ* polymerization technique[53]. The melamine-formaldehyde and urea-formaldehyde encapsulation systems for phase change materials are perfect examples of this method[47,54-56]. Chung *et al*.[57] utilized *in situ* polymerization technique for the development of microcapsules loaded with thyme oil (Fig. 7). The thyme oil loaded microparticles displayed excellent repellency (90%) against *Plodia intercpuntella* for 30 days.

2.4 Materials Involved in Microencapsulation

2.4.1 Shell Materials

The selection of shell materials is crucial as it affects the stability of microcapsules, encapsulation

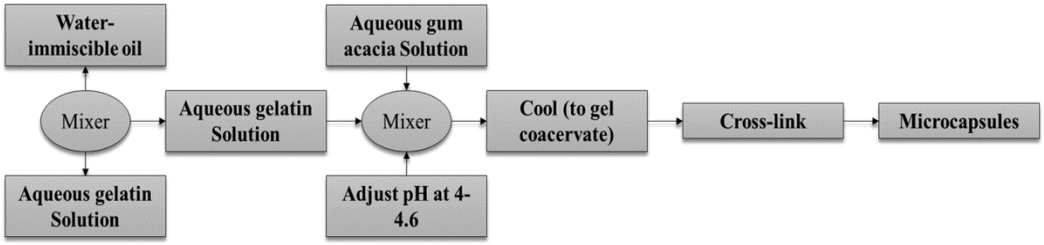

Fig. 6 — Typical process flow of complex coacervation technique

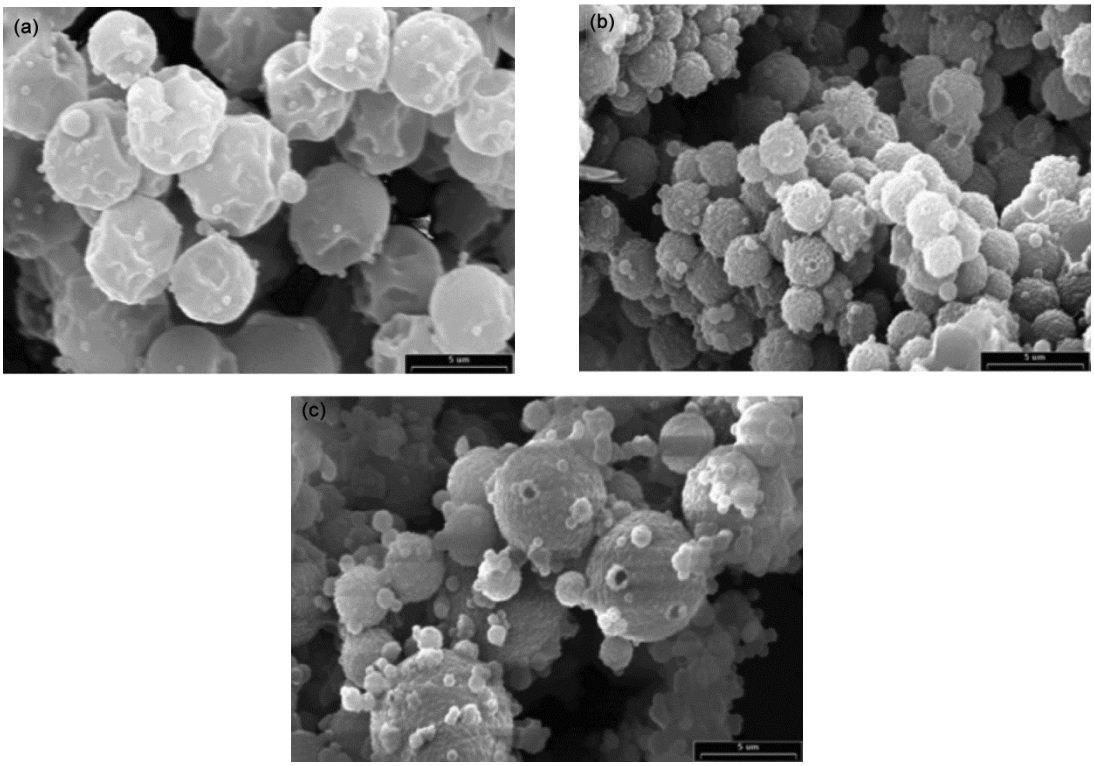

Fig. 7 — SEM images of microcapsules with different emulsifiers (bar is 5μm); (a) Sodium lauryl sulphate, (b) Tween 80 and (c) Pluronic F-127[57]

efficiency, loading capacity, retention rate, and delivery system. The selection criteria for shell material depend on the physic-chemical characteristics of the core material such as solubility and porosity, the properties of the shell material such as mechanical properties, the viscosity of the emulsion/suspension, the compatibility of core-shell materials and the economic factors[58,59]. Sometimes, the compatibility between the two materials becomes crucial because one shell material alone may not have all the required properties. Therefore, two or more polymers can be used to achieve the desired properties[60]. The shell materials can be selected from a broad range of synthetic and natural polymers.

Some commonly used natural polymers (vegetable, animal or bacterial sources) are alginate, pectin, chitosan, carrageenan, Arabic gum, cellulose derivative, starch, gelatine, milk protein, and gellan gum[61.62]. Synthetic polymers that could be used are polyvinyl alcohol (PVA), polyethylene glycol, polycaprolactone, poly(lactic-co-glycolic acid), isocyanate, polyamide, polyurea, polyurethane, and melamine formaldehyde[63]. The most commonly used biopolymers and their advantages/limitations are presented in Table 3.

Traditionally, Arabic gum is considered as one of the most common polymers used as a shell material. However, limited availability is the main issue for this polymer. Alginate is also an excellent polymer to entrap EOs, but it suffers from some problems related to porosity and high processing cost in the industry.

Gelatine can be potential polymer for the encapsulation of EOs for the application on textiles,

Table 3 — Commonly used natural polymers as shell material of microcapsules for textile application[25,59,64,65]

Polymers	Advantages	Limitations
Acetophtalate cellulose	Cellulosic and biodegradable	Slows the absorption of drugs, dissolves only above pH 6, and synthetic polymer
Alginate	Natural product and easy to work on in the laboratory	Very permeable and porous membranes, challenging to scale-up, and costly on an industrial scale
Arabic Gum	Low viscosity, adequate solubility, smooth paste, and gives good emulsifying properties	Costly and availability problems
Chitosan	Derived from chitin, non-toxic, biodegradable, easy to scale-up, affordable and biocompatible, non-allergic and pH-sensitive	NA
Ethyl-cellulose	Insoluble in aqueous medium and derived from glucose	Low mechanical resistance and insoluble in the gastrointestinal system
Gelatine	Biodegradable, non-toxic and low price	Soluble in aqueous systems
k-carrageenan	Natural product	Soluble at temperatures (60–80 ^{O}C) for 2–5% concentration
Malto-dextrin	Low viscosity at high concentrations	
Starch	Abundant, biodegradable, and non-toxic	High viscosity

offering some advantages over other polymers such as biodegradability, availability at a low price, and non-toxicity. Also, it has some limitations like high solubility in an aqueous medium[66,67]. Therefore, such polymers need to be modified or used in combination with other polymers.

Chitosan is a naturally occurring cationic polymer prepared from the alkaline deacetylation of chitin (N-acetylglucosamine polymer). Chitosan is used in many applications because of its excellent chemical properties, biodegradability, and polymer compatibility[68]. In recent years, chitosan showed a great promise for its use in microencapsulation.

Various synthetic polymers have also been utilized to prepare microparticles. Since the last years, aliphatic polyesters, like polylactic acid and glycolic acids and lactic copolymers, have been used as biodegradable wall materials for controlled release devices[69]. Nevertheless, modified celluloses such as hydroxypropyl cellulose and the semi-synthetic cellulose acetate, and butyrate are commonly utilized for the microencapsulation of EOs [70].

2.4.2 Core Materials

EOs are the aromatic materials, usually have a complex composition, acquired from specific plant raw materials, or extracted by a mechanical method. EOs are generally separated from the aqueous phase by a physical method without affecting their original chemical composition[71]. EOs are obtained from various defined plant organs such as fruits (star anise, anis), flowers (rose, jasmine, lavender and violet), leaves (Eucalyptus, salvia, thyme), buds (clove), bark (cinnamon), seeds (cardamom), zest (citrus), roots (ginger) and wood (ginger). These are lipophilic, soluble in organic solvents and insoluble in water due to their hydrophobic nature, and lower density[72].

The application of encapsulated PCMs for functional sportswear and technical garments has become popular among the textile and apparel industries. The microencapsulated PCMs can offer heat and cold absorbing ability which could be utilized to developed thermal regulating textile products. The working principle of PCMs is that it absorbs and release heat when the material changes from solid to liquid and vice versa. Therefore, the microencapsulated PCMs system helps to store the thermal energy efficiently. These materials change their physical state within a specific range of temperature and are different from one another in their heat storage capacity and phase change temperature[21]. Presently, varieties of PCMs are available such as n-hexadecane[73], n-nonadecane, n-eicosane, n-octadecane[74], and n-alkane. Various materials have been continuously used as an active core material for the application of functional textile products. These materials include vitamins, minerals, additives, oxides, aldehydes, thermochromic and photochromic materials, phase change materials and gasses[24,75].

2.5 Methods for Application of Microcapsules on Textiles

Various techniques can be used for stabilization of microcapsules on the fabric are padding, coating,

immersion, spraying and printing. In the padding method, the fabric is transported through the dispersion of microcapsules and then passed through the padding rollers to remove the excess liquor. The immersion method is the same as the padding method, except the fabric is not passed through the squeeze rolls. In the printing method, the printing paste is formed by mixing microcapsules with a binder and applied on the fabric[76]. In the coating method, the uniform layer of microcapsules is deposited on the textiles. In spraying method, the microcapsules are sprayed on the fabric via spray nozzle in a closed chamber, and further curing is carried out to stabilize the microcapsules on the fabric by thermal treatment at a high temperature (130–170 ^{O}C). However, at high temperature, quick evaporation of liquor is taking place, which leads to instability of microcapsules. Therefore, ultra-violet and microwave curing could be the alternative to the high-temperature curing process. In microwave curing, the microwave vibrates and polarises liquor molecules which result in heat generation and stabilization of microcapsules on the fabric. The fabric is exposed to UV light, which polymerizes the resin to form continuous film and stabilizes the microcapsules on the fabric. The benefits of UV curing include lower time consumption, high production efficiency, energy-saving, low-temperature requirement, and lower pollution load. This avoids rupture of microcapsules and evaporation of the core element, which also improves the durability[77,78]. The durability of the finish on the fabric cured with UV was found to be more than 50 washing cycles, which was better than that of thermal curing (25 washes)[79].

2.6 Application of Microcapsules for the Development of Functional Textiles

2.6.1 Aroma/Fragrant Textiles

The aromatherapy textiles offer fragrance of EOs derived from plant-based raw materials, which could boost the emotional and physical scene of the body[20,80,81]**.** During the second world war, Dr Jean Valnet used EOs for the surgeries and verified that they have more significant antiseptic properties than phenol[82]. Microcapsules loaded with EOs showed various applications in the textile and apparel sector[83]. The mechanical pressure on the capsules during actual wear leads to the rupture of microcapsules which results in the release of active component[84,85]. Several research studies have been conducted to develop aroma textiles, as shown in Table 4.

Sharkawy *et al.*[18] utilized the complex coacervation method to prepare chitosan/Arabic gum loaded with limonene and vanillin microcapsules using tannic acid as a solidifying agent. The results showed a sustained release pattern where the cumulative release pattern after seven days at 37 ± 1 °C was 19.4%, 52%, and 75% for the polynuclear vanillin microcapsules, the mononuclear limonene microcapsules and the polynuclear limonene microcapsules, respectively.

Table 4 — Research studies on aroma/fragrant textiles

Core material	Shell material	Method	Fabric	Application method	Results obtained	Reference
Lemon fragrance	NA	NA	Cotton	Pad-dry-cure	UV-curing Durable up to 50 washes	79
Lemon fragrance	NA	NA	Cotton	Pad-dry-cure	Conventional curing Durable up to 25 washes	78
Rose fragrance	Polybutylcyanoacrylate (PBCA)	Anionic polymerization	Cotton	Immersion	Durable up to 20 washes EE was 65.83%	86
Lavender fragrance	Melamine-formaldehyde	NA	Cotton	Pad-dry/ Exhaustion	Padding was found better than exhaustion	87
Lemon oil and rosemary oil	Methylmethacrylate-styrene	Mini-emulsion polymerization	Cotton	Immersion	6.8% cologne maintained after 15 washes EE was 85.4%	88
Jasmine oil	Acrylic-based	Two-stage emulsion polymerization	Cotton	Pad-dry-bake	25.3% residual rate of essential oil after 15 washes EE was 78.76%	89

Li *et al.*[79] utilized UV curing to stabilize the microcapsules on the cotton fabric. The fabric was suspended in a finished bath followed by padding and drying at 100 °C for 1.5 min. The finished fabric was then introduced under different UV lights, such as iron, xenon, and mercury lights, which showed different interaction of resin with the fabric. The excellent durability of finish was achieved when the finished fabric was cured with iron or mercury light.

Li *et al.*[78] applied the microcapsules loaded with lemon fragrance on the cotton fabrics by using an acrylic binder, polyurethane binder, and DMDHEU as a fixing agent. The acrylic binder was found more effective as compared to other fixing agents, as it showed excellent durability up to 25 washing cycles.

Hu *et al.*[86] synthesized the rose fragrance nanocapsules with poly butyl cyanoacrylate (PBCA) via anionic polymerization. The encapsulation efficiency was 65.83% with 0.8% butyl cyanoacrylate (BCA), 0.5% rose fragrance, 2.6% emulsifier (Tween-20) and a pH greater than 2. Nanoencapsulation of the fragrance by PBCA using anionic polymerization is found a suitable method for encapsulation, providing a sustained release pattern.

Bonet *et al.*[87] used exhaustion and padding methods for the application of microcapsules on the fabric surface. The fabric finished with padding methods, displayed more microcapsules on the fabric. Moreover, padding was found better than exhaustion because of the requirement of more chemical to fix the microcapsules on the fabric during exhaustion.

Liu *et al.*[88] used mini-emulsion polymerization to synthesize methylmethacrylate-styrene nanocapsules loaded with cologne EOs, and the synthesized nanocapsules were applied on cotton fabric via the immersion method (Fig. 8). The finished fabric displayed the uniform coating of capsules on the fabric and also showed good washing durability up to 15 washing cycle. Liu *et al.*[89] fabricated aromatic nanocapsules via two-stage emulsion polymerization method. The styrene/methyl methacrylate copolymer was used as a core material and modified poly(butyl acrylate) as a shell material which was modified by octamethylcyclotetrasiloxane. The nanocapsules showed excellent washing durability by providing around 25.3% residual content of essential oil after 15 washes.

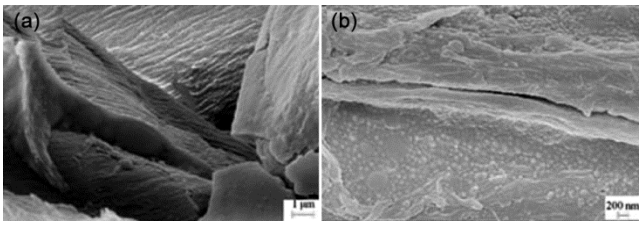

Fig. 8 — SEM photographs of cotton fabric (a) untreated (×1µm) and (b, c) treated with aromatic nanocapsules (×200nm)88 (reproduced with permission)

Rodrigues *et al.*[90] prepared the polyurethane–urea microcapsules loaded with limonene oil through the interfacial polymerization method and utilized it for finishing of the fabric using the padding method. The GC-FID-headspace and SEM analysis confirmed the availability of limonene oil on the fabric. The results showed that the loss of limonene after the first and fifth dry cleaning was 4% and 97% respectively. The loss of limonene during abrasion after 3000 and 9000 cycles was 40% and 60% respectively.

Panisello *et al.*[91] prepared the polysulfone microcapsules containing vanillin. The results showed that microcapsules were stable in the temperature range between 20 and 100 °C, and inhibits the bacterial growth during one week. The results showed moderate washing durability up to 5 washing cycle.

2.6.2 Antimicrobial Textiles

Various antimicrobial compounds such as quaternary ammonium salts, nanosilver and triclosan are available for imparting antimicrobial property to the textiles. However, because of the limitation of such agents like their limited availability, high cost, high toxicity and synthetic nature, manufacturers are moving towards the use of eco-friendly, low-cost antimicrobial chemicals for textile finishing[35].

Natural extracts are widely researched for the possible applications as antimicrobial finish; however, the efficacy of such finishes and the durability always remain a severe limitation. Most of the available EOs show antimicrobial activity and can be utilized for antimicrobial finishing of textiles. The limitations of EOs, especially the volatility can be overcome by encapsulating these in suitable polymer-shells. The various reports regarding the use of microcapsules containing EOs for antimicrobial finishing are available in the literature, as shown in Table 5.

Thilagavathi *et al.*[92] prepared the microcapsules loaded with Mexican daisy and neem extracts using acacia gum as a shell material and applied on the cotton fabric. The finished fabric showed more antimicrobial properties against *S. aureus* as compared to *E. coli*, which were retained even after 15 washing cycles.

Table 5 — Research studies on antimicrobial textiles

Core material	Shell material	Method	Fabric	Application method	Results obtained	Reference
Neem oil + Mexican daisy leaves oil	Gum acacia	Simple coacervation	Cotton	Pad-dry-cure	Antibacterial properties even after 15 washes against *E. coli* and *S. aureus*	92
A mixture of turmeric, tulsi, and neem extracts	Fresh yeast	Plain diffusion method	Cotton and silk	Pad-dry-cure	A very active antimicrobial activity against *Pseudomonas*	93
Ozonated red pepper seed oil	Gelatine and Arabic Gum	Complex coacervation	PP non-woven	Padding	Good antimicrobial activity against *E. coli*, *S. aureus*, and *C. albicans*,	94
Berberine	Chitosan and agar-gelatine	NA	Cotton	Pad-dry-cure	Agar–gelatine microcapsules showed antimicrobial activity even after 20 washes against *S. aureus*	95
Eucalyptus and sandalwood oils	Chitosan	Emulsion	Cotton	Pad-dry-cure	Excellent antibacterial activities against *E. coli*, and *S. aureus*	96
Patchouli oil	Chitosan–gelatine	Complex coacervation	Cotton	Pad-dry-bake	EE was 50.69% Antibacterial activity (65%) against *S. aureus* and *E. coli* even after 25 washes	97
Poly-(n-butyl acrylate)	Chitosan	Surfactant-free emulsion copolymerization (Microcapsules)	Cotton	Pad-dry-cure	Excellent antibacterial effect (99%) against *S. aureus*	98
Poly(n-butyl acrylate)	Chitosan	Surfactant-free emulsion copolymerization (Nanosized particles)	Cotton	Pad-dry-cure	The antibacterial activity > 99% and retained to 90% even after 50 laundering cycles against *S. aureus*	99
Poly(methyl methacrylate) (PMMA)	Chitosan, polyethyleneimine, and chitosan-polyethyleneimine	Emulsifier-free emulsion polymerization	NA	NA	Strong antibacterial properties against *E. coli* and *S. aureus*	100
Tea tree oil	Sodium alginate and a quaternary ammonium salt of chitosan	Complex coacervation	NA	NA	EE was of 66.06% ± 2.53% Excellent inhibition rate against *S. aureus* (89%, and *E. coli* (87%) even after storage for 30 days	101

Saraswathi *et al*.[93] developed the microcapsules loaded with a mixture of turmeric, tulsi, and neem, via plain diffusion method. The prepared microcapsules were applied on silk and cotton fabrics using a commercial binder at *p*H 5.5–6 and the treated fabrics were cured at 120 ^{O}C for 2 min. The treated fabrics showed active antimicrobial properties against the selected bacteria. Ozyildiz *et al*.[94] utilized the complex coacervation method to fabricate the microcapsules containing gelatine and Arabic gum as a shell material and ozonated red pepper seed oil as a core, and then applied on 100% polypropylene non-woven fabric. The finished fabric exhibited antimicrobial activities against *E. coli, C. albicans, and S. aureus*. Lam *et al*.[95] prepared agar-gelatine and chitosan microcapsules loaded with berberine and applied on cotton fabric using acrylic binder to develop the antibacterial fabric. The presence of microcapsules on finished fabrics after 20 washes was confirmed by SEM images. The presence of a few

microcapsules was also reported after 50 washes. The antibacterial activity (against *S. aureus*) of the fabric treated with agar–gelatine microcapsules after 20 washes were better than that of the fabric treated using chitosan microcapsules.

Javid *et al*.[96] prepared the microcapsules loaded with sandalwood oils, and eucalyptus via emulsion method. The modified dihydroxy ethylene urea was used to graft the microcapsules on the cotton fabric. The antibacterial property of the finished fabric was evaluated against *S. aureus* and *E. coli* using the turbidity method, which shows higher antibacterial properties with the presence of the higher amount of chitosan and essential oils.

Liu *et al*.[97] developed chitosan-gelatin microcapsules loaded with patchouli oil using a complex coacervation method. The prepared microcapsules showed encapsulation efficiency of 50.69% and loading capacity of 30.31%. The treated fabric displayed good antibacterial activity (65%) against *E. coli*, and *S. aureus*, which was durable even after 25 washing cycle. The results were encouraging, and the treated fabrics were projected for the possible applications in the fields like health-care clothes, bacteriostatic sheet, and antibacterial mask. Ye *et al*.[98] fabricated chitosan microcapsules loaded with poly-(n-butyl acrylate) through surfactant-free emulsion copolymerization methods. The cotton fabrics finished with such microcapsules displayed antibacterial property (99%) against *S. aureus*, and the unique core-shell structure showed antibacterial durability over repeated washing. Ye *et al*.[99] used surfactant-free emulsion copolymerization to prepared nanosized particles in aqueous chitosan. The fabric treated using such capsules displayed an excellent antibacterial property (> 99%), and the 90% antibacterial activity was retained even after 50 washes. Inphonlek *et al*.[100] fabricated nanoparticles loaded with poly(methyl methacrylate) using with chitosan, polyethyleneimine, and chitosan-polyethyleneimine as shell materials through emulsifier-free emulsion polymerization. The nanoparticles displayed excellent antibacterial properties against *S. aureus, and E. coli.* Chen *et al.*[101] prepared the microcapsules containing tea tree oil (TTO) using sodium alginate and a quaternary ammonium salt of chitosan as a wall material. The prepared microcapsules displayed significant encapsulation efficiency 66.06% ± 2.53%). The microcapsules showed excellent antibacterial activities, (98.66%) against *E. Coli* and 100% activities against S. *aureus* after storage for five days. The TTO-loaded microcapsules also demonstrated an excellent inhibition rate against *E. coli (87%)* and *S. aureus* (89%) even after storage for 30 days. Lee *et al*.[102] prepared the melamine-formaldehyde microcapsules loaded with C. unshiu fruit extract via *in situ* polymerization and applied it on the cotton fabric using an acrylic binder. The SEM confirmed the presence of microcapsules on the fabric. Qi *et al*.[103] utilized the ionic gelation method to prepare nanoparticles of chitosan containing tripolyphosphate anions. The nanoparticles showed higher antibacterial activity against *E. coli, S. choleraesuis, S. aureus*, and *S. typhimurium* as compared to that using chitosan alone because the nanoparticles easily led to a disruption of cell membranes and the leakage of bacteria cytoplasm.

2.6.3 Insect/Mosquito Repellent Textiles

The textile product which offers mosquito repellency has been recently invented, and the method of preparation involves the application of mosquito repellent agents on to the textile materials. The N, N-diethyl-3 methyl benzamide (DEET) is the most commonly used insect/mosquito repellent. While providing effectiveness against most of the insects, DEET presents some limitations such as higher toxicity along with allergic and synthetic nature. Thus, recent research on mosquito repellent textiles focuses on the use of EOs, which is a safer and more effective way of achieving insect repellency[104]. The examples of such EOs include cedar, pine, thyme, peppermint, citronella, geranium, cinnamon, garlic, and basil. Several research studies have been conducted to develop mosquito repellent textiles, as shown in Table. 6.

Kim and Sharma[105] prepared the gelatine microcapsules containing thyme and clove bud oil via coacervation process. The prepared microcapsules were applied on 100% acrylic fabric through vacuum drying. The finished fabrics acaricidal activities were investigated using AATCC test method 194-2007. The fabrics treated with microcapsules containing clove bud oil showed higher antioxidant and acaricidal activities against *Dermatophagoides farina* as compared to red thyme oil which might be because of the higher number of hydroxyls presence in clove bud oil. Ramya and Maheshwari[106] synthesized andrographolide loaded sodium alginate microcapsules via ionic gelation method, and the microcapsules were applied on the bamboo/cotton fabric by exhaustion method. The microcapsule-finished fabrics displayed 94% mosquito repellency as compared to 96% repellency in case of directly finished fabrics. The repellency was gradually decreased to 52% for

Table 6 — Research studies on insect/mosquito repellent textiles

Core material	Shell material	Microencapsulation method	Fabric	Application method	Results obtained	Reference
Thyme and clove bud oil	Gelatine	Coacervation	Acrylic	Pad-Vacuum drying	The antioxidant and acaricidal activities of clove bud against *Dermatophagoides farina* were higher than that of red thyme oil	105
Andrographolide	Sodium alginate	Ionic gelation	Bamboo/ cotton blend	Exhaustion	Displayed 94% mosquito repellency and gradually decreased to 52% after 30 washes	106
Vitex negundo leaf extract	Alginate	Ionic gelation	Cotton	Pad-dry-cure	Showed 100% mosquito repellency and retained 70 % repellency till 15 washes	107
A mixture of grapefruit oil, cypress oil, and thyme oil	Acacia arabica, sodium alginate, and *Moringa oleifera* gum	Ionic gelation	Bamboo/ Tencel blend	Exhaustion	*Moringa oleifera* gum microcapsules retained 60% mosquito repellency even after 30 washes	108

microcapsule-finished fabrics and 40% for directly finished fabrics after 30 washes. Ramasamy *et al*.[107] synthesized nanoparticle-containing *V. negundo* leaf extract through ionic gelation method to impart mosquito repellency to textile materials. The finished fabrics showed 100% mosquito repellency and retained 70 % repellency after 15 washes.

Geethadevi and Maheshwari[108] prepared mosquito repellent microcapsules by ionic gelation method using three wall materials such as acacia arabica, sodium alginate, and Moringa oleifera gum and the combination of three core materials namely grapefruit oil, cypress oil, and thyme oil. The microcapsules prepared using *Moringa oleifera* gum retained 60% mosquito repellency even after 30 washing cycles. Specos *et al*.[109] used a complex coacervation method to fabricate the microcapsules loaded with citronella essential oil. The gelatine and Arabic gum were used as a wall polymer, and glutardialdehyde was used as a hardening agent. The cotton fabrics finished using such microcapsules showed more than 90% repellent activity for three weeks.

2.6.4 Medical Textiles

The medical textiles are covering a wide range of application such as first aid, clinical, medicinal and hygienic purpose. Medical textiles are ranging from bandages wound dressing, gauzes and tissue-culturing to body implants like a skin, artificial heart, blood vessel, and heart valve. These types of textiles have been developed by following various approaches. However, microencapsulation of EOs into a non-toxic, biocompatible, biodegradable polymers, and applying on the textile can be a possible approach to introduce an alternative of synthetic chemicals[110].

Gouveia[111] investigated the development of microspheres using a combination of n-dodecane, bovine serum albumin (BSA), and L-Cysteine (L-Cys) and applied these microspheres as a coating on the fibrous material. The coated fibrous material showed excellent antibacterial activity along with high stability. The results suggest that the new coating could be an alternative to develop fibrous bioactive delivery systems. Gong *et al*.[112] utilized solid dispersion method to encapsulate the curcumin into poly(ethylene glycol)-poly(e-caprolactone) copolymer and loaded it with a thermosensitive hydrogel composite namely poly(ethylene glycol)-poly(e-caprolactone)-poly(ethylene glycol). The prepared wound dressing showed strong tissue-adhesiveness with an extended time of curcumin release, which results in suitable granulation, high collagen content, and excellent wound healing ability. Liakos *et al*.[113] encapsulated different EOs such as chamomile blue, tea tree, lavender, lemongrass, peppermint, elicriso italic, lemon oils, cinnamon, and eucalyptus oils in a film of sodium alginate and glycerol as a plasticizer. The composite wound dressing film showed that

most of the EOs prevent the growth of *C. albicans*. However, peppermint oils, lemongrass, and cinnamon oils displayed antibacterial properties against *E. coli*.

2.6.5 Cosmetic Textiles

The microencapsulation is also used to prepare cosmetic textiles by applying cosmetic agents such as anti-ageing agents, essential oils, skin cooling agents, vitamins, and skin moisturizing agents. The microcapsules and their application should be based on the principles of good manufacturing practices. Moreover, the combination of cosmetic and textile materials offers an advance and unique cosmetic textile product. These kinds of product can offer other properties like antioxidant, odour-reducing, antimicrobial and mosquito repellence[21]. The durability of a cosmetic ingredient on the textile is essential. Several researchers have studied various techniques for enhancing the durability of functional properties offered by microcapsules; however, this is comparatively the most challenging work in the production of cosmetic textiles[22]. Cheng *et al.* [24] developed a vitamin C loaded gelatin microcapsules using emulsion hardening technique. The gelatin microcapsules were found to be non-cytotoxic based on the toxicity studies conducted on human liver. The prepared microcapsules were successfully grafted into textile materials.

2.6.6 Multifunctional Textiles

Multifunctional textiles offer two or more functional properties. The functional properties are derived from the functional shell and active core materials of microcapsules. In addition to the main functional property, these microcapsules possess one or more functional properties like antibacterial activity, antifungal activity, flame retardancy, thermochromic performance, electrical conduction, photoluminescence and thermal stability[21,85,114].

Wang *et al.*[115] developed multifunctional cotton with efficient ultraviolet protection, thermal insulation and superhydrophobic and aromatic performance using an aluminum-doped zinc oxide -embedded lemon microcapsule and SiO_2 dual-layer coating. Janarthanan and Kumar[116] synthesized microcapsules containing brown algae extracts. The finished fabric with microencapsulated material showed excellent antioxidant and antimicrobial properties. Scacchetti *et al.*[117] prepared multifunctional microcapsules using monochlorotriazinyl-β-cyclodextrin conjugated with thyme oil. The microcapsules provided antibacterial, antifungal and thermoregulation properties. Li *et al.*[75] prepared microcapsules with a high latent heat and a high thermal reliability using CuO-doped polyurea as a shell and an n-eicosane as a core through a one-step interfacial polymerization. Cotton fabric coated with the prepared microcapsules showed high phase change enthalpy of 36.8 J/g, an effective thermoregulation capability, and a large contact angle.

3 Recent Patents on Application of Microencapsulated Materials on Textiles

Recently, textile industry is exploring the potential of microencapsulation for developing innovative functional clothing/apparel. Patents on the functionalization of textiles using microcapsules are reported Table 7.

Table 7 — Summary of recent patents on application of microcapsules on textiles

Title	Year	References
Anti-mosquito finishing process of textile fabric	2012	118
Isatis root oil microcapsule and functional fabric finishing agent	2012	119
Double-layer aromatic phase change microcapsule textile finishing liquor as well as preparation method and application thereof	2015	120
Tea tree oil microcapsule antibacterial healthcare fiber and preparation method thereof	2015	121
Preparation and application of modified melamine resin essence microcapsule finishing agent	2015	122
Intelligent temperature adjustment textile and making method thereof	2016	123
Non-woven fabric, finishing method and application of non-woven fabric	2017	124
Durable flame-retardant finish method for cotton fabric based on ultraviolet light curing reaction	2017	125
Preparation for pinus koraiensis phytoncide finishing agent, fabric finished by same and finishing method thereof	2018	126
Preparation method and application of finishing agent containing rose essential oil microcapsules	2018	127
In situ microencapsulation treatment using a coacervated polymer system of asbestos fibres and other hazardous materials	2018	128
Aroma-loaded microcapsules with antibacterial activity for eco-friendly applications	2018	129
The preparation method of microcapsule coated insecticide-controlled release textile	2019	130
Ultra-thin glass, the method for ultra-thin ceramic and its manufactured goods and application are manufactured with fibre assist formation	2019	131
Encapsulation system for prolonged release of active agents	2019	132

The current trends in the development of functional textiles using microencapsulation techniques are mainly focused on the development of mosquito repellent, multifunctional, thermally sensitive, temperature adjusting and flame-retardant textile materials. The summarized patents cover the introduction of innovative microencapsulation techniques, functional polymers, novel EOs and additives. Moreover, the durability of finish and development of sustainable process are the prime concerns among the researchers.

4 Conclusion

The textile industry is currently experiencing many challenges due to the varied demands of modern customers. Functionalization of textile products is among the top requirements of the present market. Since the last two decades, microencapsulation technology is the most appreciated technology to functionalize the textile products. The beauty of this technique lies in the combination of two materials, both of which can be functional, which can synergistically enhance the functional properties. Microencapsulation could be one of the most promising techniques for functionalization of textile materials, especially with the use of volatile essential oils. The technical limitations like the need of fixing agents, limited durability, especially towards repeated washing and the changes in physical properties of textiles after application of microcapsules will remain the quest of research for the future. There is a broad scope in functionalization of textiles using microencapsulated materials, and it is expected that the development of multifunctional textiles will continue to grow and open up the new opportunity shortly. The limitation, however, must be countered through continuous research in this important area of chemical science. In future, innovative technologies and modern manufacturing techniques for micro-encapsulation and application on textiles will be required. The development of sustainable textile products using microencapsulation will be an interesting challenge for research in the near future.

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