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An overview of microencapsulation technology in the application of aroma and antibacterial finishes

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Abstract

There is a need to develop fabrics with special finishes. With the help of technological advancement researchers can bring about innovation in fabrics. The purpose of the study is to investigate the technology that can be used for application of functional finishes on fabrics. This review paper investigates the techniques applied in the formation of the microcapsules, its purpose, and method of application for fixing it on textiles, the binders and polymers used and release of aroma. There are various release mechanisms which can be applied as per the requirement or end use. The essential oils used are highly volatile in nature hence very difficult to maintain the durability of the finish in terms of storage and washability. In this review, microencapsulation techniques of application to obtain innovative fabrics for the textile sector has been highlighted.

Keywords: aroma textiles, binders, bio-polymers cosmetic textiles, control release

1. Introduction

The wide range of benefits for aromatherapy and controlled release of essential oils and value addition in fabrics is expected to be appreciated by consumers [26]. Fragrance finishes have been applied on different fabrics such as cotton, Jute, Jute-cotton and Bamboo fabrics. Microencapsulation is a technique to prepare small capsules, which contain an outer layer coating, shell or external phase, membrane or matrix and an Inner layer or core, also known as encapsulations, payloads or fillers [1]. The term "Microcapsule" can be defined, as a spherical particle with the size varying between 50 nm to 2 mm containing a core substance [5]. The size of the nanocapsule was investigated in relation to the loss of the fragrance from the cotton fabric. It was found that, the smaller the nanocapsule size, the better the sustained release property and improved wash fastness [14]. Silva, M. synthesized polymers of different molecular weights for the microencapsulation of limonene, using a step-growth polymerization process. The core-shell microcapsules were spherical in shape with a mean diameter in between 10 and 20 μm [23].

The core is made up of the active ingredient that could be solid, liquid or gas [3, 4]. Some of the commonly used core materials by various researchers are Rose, lemon, geranium oil [28], Vanillin [12, 16], Jasmine, Benzoic Acid [12], lavender limonene [21, 23], lemon grass oil [25] and aloe vera [20].

The coating protects the active ingredient from the external environment such as sunlight, oxygen and moisture. The shell could be made up of polymers, waxes, resins, proteins or polysaccharides. The coating material should be inert towards active ingredient, non-hygroscopic, pliable, and stable also be able to form a film. It should have low viscosity and should be soluble in an aqueous medium or solvent. It should be able to stabilize the active ingredient and release it in a controlled manner under specific conditions. The outer layer can be flexible, brittle, hard or thin etc. Polymers used for coating microcapsules should be compatible with the body [11]. Commercially, microencapsulation is done using melamine-formaldehyde, urea-formaldehyde or phenol-formaldehyde resins. They have a high thermal stability, at the same time, easy to modify the release mechanism. They can be highly toxic to the environment and human beings. They are non recyclable and carcinogenic, hence they need to be replaced with something that is more eco-friendly and less toxic [21].

A number of synthetic biodegradable polymers have been developed for various applications due to the ability to be readily modified by simple chemistry [6].

2. Natural polymers or Biopolymers

They can be used instead of chemical-based finishing agents. They are derived from various sources such as agricultural

and marine food resources. Chitosan, Gelatin, Sodium alginate and Albumin are the most commonly used natural polymers in the preparation of polymeric nanoparticles. Biopolymers are natural, available in abundance, biodegradable and biocompatible [7]. Biopolymers based on its origin have been classified as in Fig. 1 below.

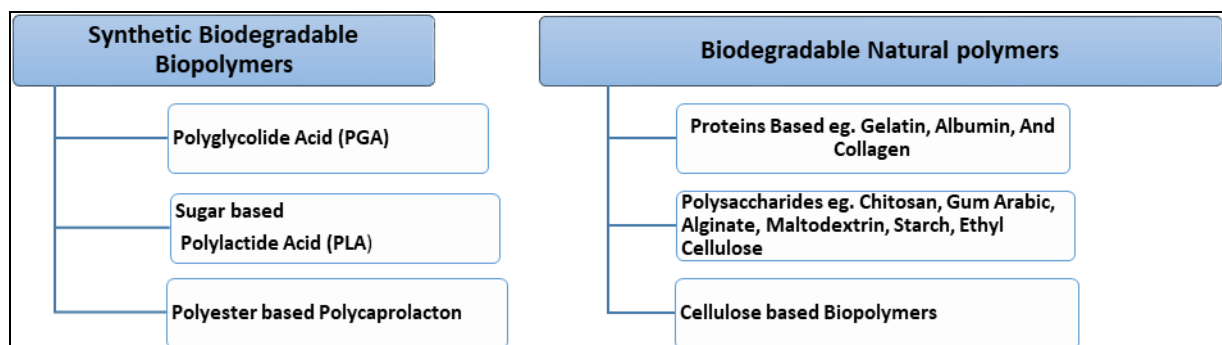


Fig 1: Types of Biopolymers [6, 7, 11]

Hak *et al.* used B-cyclodextrin as a protective wall. B-cyclodextrin was embedded onto cellulose fibers by using N-methylolacrylamide. Benzoic acid acted as an anti-bacterial agent and vanillin aroma were encapsulated and applied to fabrics. It was claimed that the anti-bacterial activity was retained after 10 laundering cycles [12]. Fei (2015) Applied fragrance finish with a methanol-modified melamine-formaldehyde resin shell with an in situ polymerization method in the presence of styrene-maleic anhydride copolymer (SMA). The thermal stability improved by this process due to the polycondensation reaction and the pH effect.

The micro capsules are formed using Binders, Cross linking Agents, Organic or Inorganic Pigments, Fillers, Antifoaming Agents and/or other Surfactants and Viscosity- controlling Agents/Thickeners. They should be selected carefully so that, they do not bring about a noticeable change in the color or feel of the fabric.

3. Binder

It is one of the most important raw materials used for the

fixation of the microcapsules onto the fabrics. The durability and washability of a finish is highly dependent on the use of binders. They could be either natural or synthetic. Binders may be acrylic, polyurethane, silicone, starch, etc. Butadiene Acrylonitrile acrylic latex was found to have a firm hand can be used in print application for special finishes as mentioned in the Technical bulletin, ISP1008

Khanna, S. (2015), explored the performance of chitosan as well as citric acid as a potential binder between beta-cyclodextrin and the cotton fabric for imparting aroma to the textiles. The chitosan based aroma finishing was found to be more effective than citric acid for the aroma retention in terms of retention of vanillin aroma [16]. Sharkway *et al.* (2017) studied the effect of two emulsifiers with respect to the encapsulation efficiency, the size morphology and their release profiles. The microcapsules were grafted onto cotton fabrics through an esterification reaction using citric acid as a nontoxic cross-linker followed by thermo fixation and curing. The antibacterial assays were conducted which indicated a sustained antibacterial activity [22]. The Fig 2. gives a comprehensive list of binders to choose from.

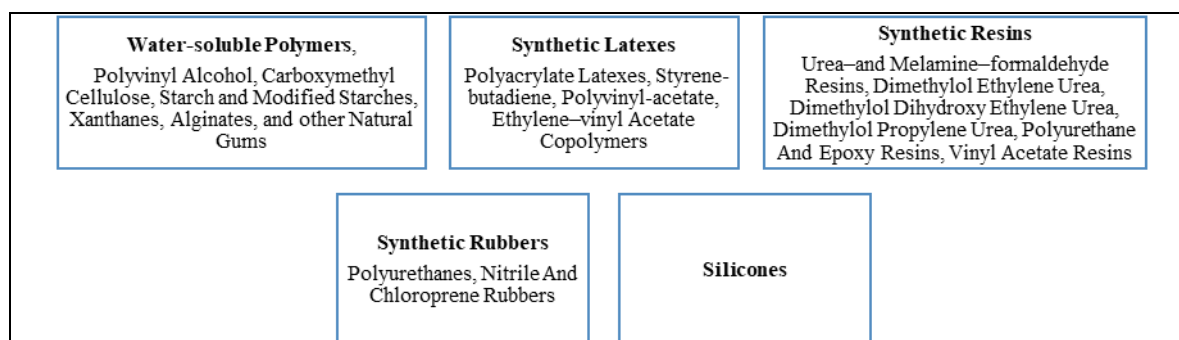


Fig 2: Binder selection List [8]

4. Application of microcapsules to the substrate

Microcapsules can be added to the fabric at different stages mechanically right from polymer stage to fabric stage.

- In the polymer (inclusion of microcapsules in the fibers during the spinning process)
- On the fabric (impregnation or immersion /Padding)
- In a coating (with an air knife or rod coater)
- Spraying on the surface of textiles

- Printing on the surface of textiles (by Screen, Photographic, Electrostatic, Pressure-Transfer, Thermal Transfer and Inkjet Printing Techniques)
- Incorporation into polymer foams, coatings and multilayer composites that are placed or inserted into selected parts of textile clothing or footwear [8, 11].

Some of these application methods have been studied by researchers. Hipparagi (2016) microencapsulated aroma chemical which was used for the finishing treatment. Impregnation method, Exhaust method, Dip–Pad–Dry method and Spray methods were used to see the influence of the application method on the uptake and performance. Subjective evaluation of the aroma treated material was done as per Odor Intensity Reference Scaling (OIRS). Effect of the aroma finishing on the physical properties of the fabric was studied [19]. Elseini (2016) studied the microcapsules further (in two trials) applied with a screen-printing technique. It was found that, during wear, the fragrance faded, but by rubbing the surface of the bow-tie and consequently rupturing the microcapsules, the release of the fragrance was initiated again, before or after wear [18]. Chang integrated the processing procedures of fabric treatment techniques. With low temperature plasma and microencapsulation of natural oil essence and fabric coating techniques to improve the adhesion property of microcapsules with fabrics. This invention increased not only the adhesion area of microcapsules on the fabric, but also enhanced the use for oil essences and promoted the additional value of the fabric (US Patent, 2005).

5. Role of encapsulation technique

Microencapsulation protects the substances against environmental effects. It also helps in separation of reactive components, masking, improves stability, reduces or prevents volatility, sustained release or controlled release. Microencapsulation shows several advantages for textile industries since active compounds, such as vitamins, essential oils, antimicrobials and/or antibiotics can be isolated to protect them from environmental factors (oxygen, light,

moisture and temperature). It can be used for the controlled release of fragrances or even heat, to mask undesired properties of the active components and to convert liquid substances into solids with increased compatibility. The only drawback being the cost and time involved. Ghayempore and Montazer (2016) reported that encapsulation technique plays an important role in the finishing of plant extracts on the textile substrates [17].

Alessandro (2017) applied fragrance finish to cotton fabric using lavender and aloe vera; but the results of washing fastness were found to be poor. Here we understand the importance of microencapsulation technique [20]. Sharkaway *et al.* (2017) developed a functional fabric with durable antibacterial and fragrant properties using ecofriendly materials. Microencapsulation of aroma compounds was done by the complex coacervation method using biodegradable polymers. They are attached to cotton fabrics by means of thermo fixation grafting process using a polycarboxylic acid. The grafting of the microcapsules to cotton fabrics was done by the pad-dry-cure method using citric acid as a cross-linker. They also prepared poly (L-lactide) microcapsules for fragrant fibres by an interfacial precipitation method through solvent evaporation from water-in-oil in-water emulsion. The microcapsules were then uniformly printed on cotton fabrics and the resulting fabric could withstand 15 washing cycles (Hong.*et al.*, 2000).

The techniques used for the microencapsulation process for value added textile applications are explained in Figure 3. Generally the microencapsulation is done using one of the following techniques. Selection of the technique depends on the characteristics desired and end uses of the products.

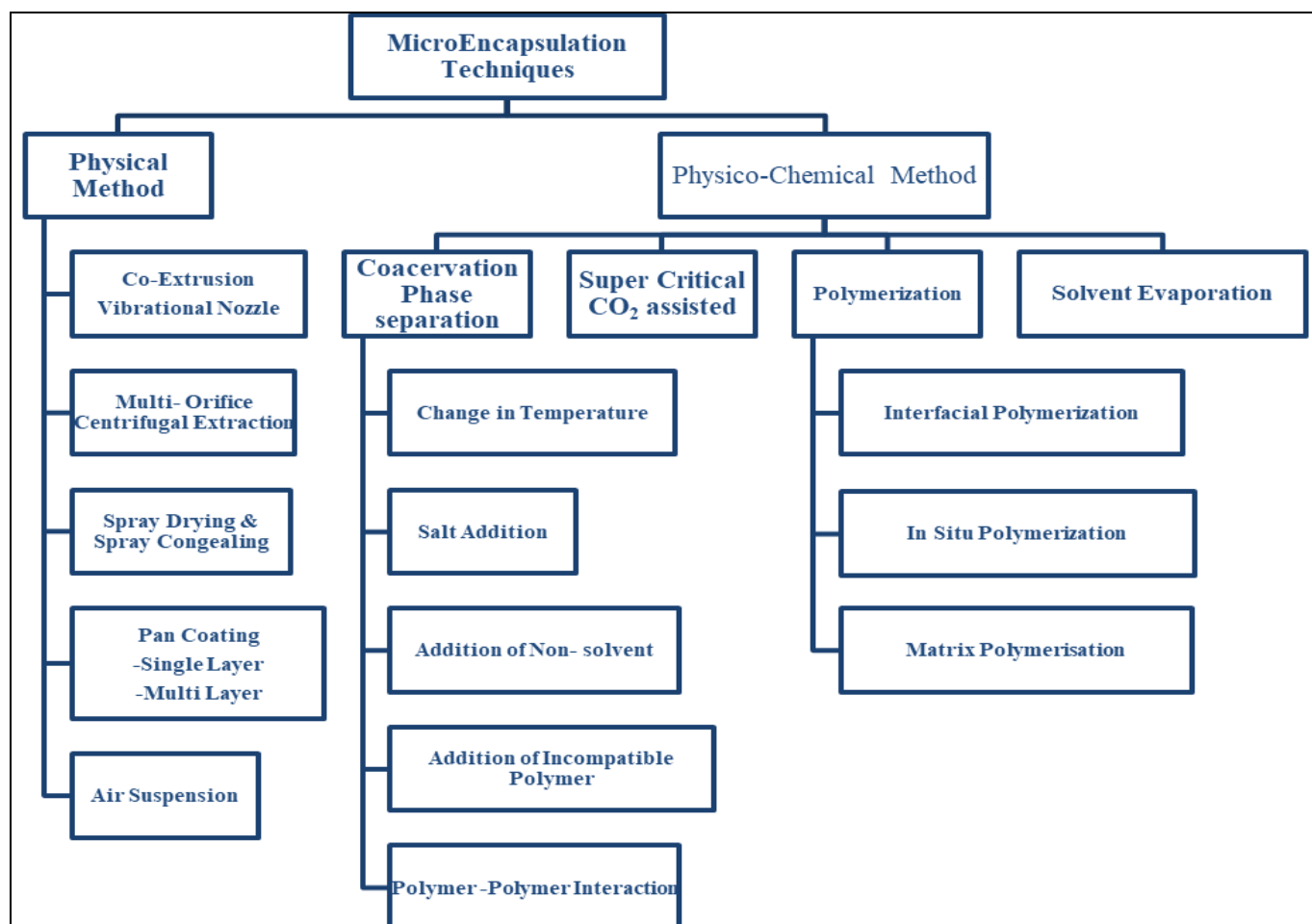


Fig 3: Techniques of Microencapsulation [8, 9, 26].

Silva, *et al* (2017), recognized the human health problems concerning formaldehyde emissions that led to the use of poly(urethane–urea) (PUU) systems. Here the fabrics were impregnated with PUU microcapsules containing limonene produced by interfacial polymerization. To aid the microcapsule impregnation, a specific binder was used followed by drying and thermo fixation process ^[21]. Bhatt,*et.al* (2018), studied the application of lemongrass oil on fabric by two methods: oil microcapsules applied through padding and exhaust method. Lemongrass oil microcapsules were prepared using complex coacervation technique and applied on fabric using pad dry cure technique ^[24]. Valdes (2018), has discussed the main techniques and the different mechanisms based on the different properties and the development and application of microencapsulation. The use of different additives with medical properties, insect-repellent systems and fragrant solid carrier materials ^[26]. Kumari (2020) selected Geranium oil as the core material and gum acacia as wall material for encapsulation using complex coacervation technique and ratio of 1:4:4 of oil, gum and gelatin ^[28].

6. Release Mechanism

Mechanisms of releasing active ingredients from the microcapsule core depend on the purpose of microencapsulation, on the functions and desired effects of encapsulated components, and on the microcapsule wall characteristics, particularly on permeability ^[26]. release mechanism feature is important specially for aroma encapsulated textiles. Fragranced textiles, containing microencapsulated essential oils, aromas and perfumes, have been developed to either

- Slowly release their contents through permeable walls, or
- To have completely impermeable walls, and open only by application of mechanical pressure and rubbing whenever the wearer moves.
- A combination of both release mechanisms is also possible.

To release microencapsulated active components from microcapsule core, numerous ways of release mechanisms have been invented and applied in added-value textile products. The release mechanisms of the core contents vary depending on the end use ^[31, 32].

The Fig. 4 demonstrates the relationship between the textile end uses and the release mechanism. The content of core may be released by either by friction, pressure, change of temperature, diffusion through the polymer wall, dissolution of the polymer wall coating, biodegradation etc.

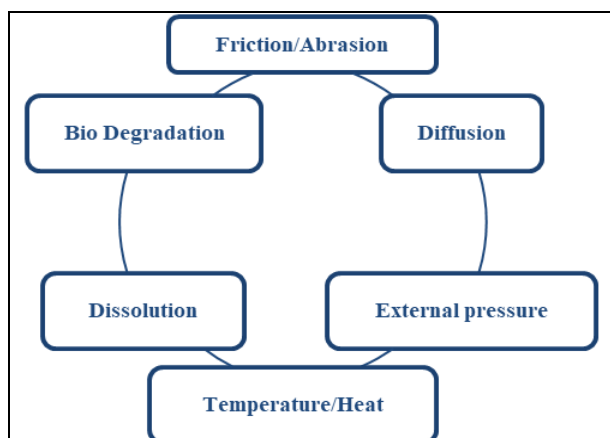


Fig 4: Release Mechanism ^[26, 31, 32]

Microencapsulation methods and processes of applying microcapsules to textiles have been studied by various researchers. It has been observed that temperature plays an important role in the fixation, release, loss of aroma or efficiency of the finish. Wang and Chen (2008) developed aroma therapeutic textiles by using fragrance with B-cyclodextrin inclusion compounds and fixing them onto cotton fabrics with low temperature by using a conventional pad-thermo fixed method. The fragrance release rates were greatly decreased and the results of sensorial evaluations showed that the performance of the fabric lasted for over 30 days (Wang *et al.*, 2005). Li, *et al.* (2008) investigated the effects of UV curing for encapsulated aroma finishing on cotton. Cotton fabric was finished with the selected aroma capsule and UV resin, and cured under optimal conditions. It was found that in order to fix the microcapsule, a high temperature thermal process can lead to loss in aroma. Three fixing agents, three kinds of thermal curing equipment, and various curing conditions were investigated. By analyzing the properties of the fixing agents, heat transfer characteristics of the curing apparatus, and delivered energies in the curing conditions, a washing durable aroma capsule finishing process for cotton fabric was developed. The aroma function was prolonged to 50 wash cycles; whereas the traditional curing method could only withstand 25 wash cycles ^[13].

7. Evaluation of finishes

The structure and properties of the Nano capsule can be studied by using the Transmission Electron Microscope (TEM), Dynamic Light Scattering (DLS), Gas Chromatography etc. Ultraviolet–visible analysis may be done to study the encapsulation efficiency and loading capacity Liu (2017). The properties and the effect of the aroma finishing on the physical properties of the fabric such as the surface morphology can be studied by Scanning electron microscopy (SEM), thermal properties measured by Simultaneous Thermal Analysis (STA), thermo gravimetric analysis, size and size distribution by SEM and ImageJ software, and release behavior of the microcapsules can also be analyzed. SEM may be used to examine the treated fabrics before and after washing and Fourier transform infrared spectroscopy (FTIR) to confirm the covalent attachment of the produced microcapsules to the cotton fabrics. The textiles may also be subjected to abrasion and dry cleaning tests (Silva M. 2017).

The fragrance release rates can be tested by sensorial evaluations (Wang *et al.*, 2005). Evaluation of the aroma treated material may be done through subjective evaluation as per Odor Intensity Reference Scaling (OIRS) Hipparagi (2016). Electronic nose may also be used for evaluation.

The Antimicrobial assays may be evaluated using the Agar Diffusion Method, whereas the antibacterial activity of the treated fabrics can be assessed by the Standard Test Method (American Society for Testing & Materials, ASTM Standard E 2149-01). The antimicrobial assessment of the control and treated fabric can be done through AATCC-100 (Bhatt 2018). The release data can be analyzed by applying the Korsmeyer–Peppas model (Silva M. 2017).

Testing the treated sample using the appropriate method is of utmost importance to enable analyzing and come to conclusions about the efficiency of the finish applied.

8. Discussion

Microencapsulation may be the most suitable technique for aroma finish on cotton fabric Ghayempore, S (2016). Microcapsules were prepared using various technique

complex coacervation technique was used most commonly. This technique can be used to apply essential oils on the fabric in order to impart fragrance finish. Imparting fragrance to fabrics has been done earlier in the form of fabric conditioners or washing detergents etc. but the effect is short lived. Variations in the size of the microcapsule can also affect durability. Jing H. (2011) found that smaller the size better the sustained release property and also the durability. As per the studies, (Bhatt and Singh, 2018) it can be concluded that high temperatures can lead to loss of aroma or efficiency of a finish. Since application using an exhaust method seems to be less durable and application at printing stage seemed to be a good option. This technique can work wonders provided the right kind of temperatures is used. Curing at high temperatures should be avoided and other alternatives such as low temperature fixation and exposing to UV rays could be experimented (Li *et al.*, 2008). After microencapsulation of the core material, its application on to the substrate by pad-dry-cure method was more advantageous than exhaust method. Formaldehyde and its compounds were commercially used (Boh *et al.*, 2006), since they are found to be toxic in nature (Silva M.2017), replaced them by biopolymers to attain environmentally sustainable finish. Beta-cyclodextrin is used by many researchers as a coating for the capsule and chitosan and citric acid as a binder or cross linking agent are a good choice for attachment to the substrate (Khanna S.2015),

9. Conclusion

In this world of advanced technologies, the technique of microencapsulation has a vast application in the field of textiles alone. It is found to be an effective technique which improves the functionality of the fabric. Microencapsulation technology seems to be a promising alternative for application of such functional finishes with good durability and performance. The techniques of producing microcapsules containing essential oils have been studied extensively. Different essential oils can be taken to give an overall wellness finish to the fabric. The fabrics could be soothing, calming, fragrant and antimicrobial all at the same time if the fragrance and the antimicrobial effect stay for a prolonged period of time. These finishes can be applied in large scale production, that is an important area for research. There seems to be a vast scope for further research by trying different combinations of core material, shell material, binder application techniques, release techniques and variations in the size of the microcapsule. Researchers have tried improving the durability of microencapsulated functions. Further studies need to be done to improve the washing fastness and durability of these finishes. Such aroma fabrics will be appreciated by the consumers provided they get variety of aromas to choose from and are pocket friendly and long lasting.

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