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### **Recent advances in Microencapsulation Technology and their Applications**

M. Arsalan Pasha<sup>a</sup>, Shubham Utekar<sup>a</sup>, Ritoban Ghosh<sup>a\*</sup>

aDepartment of Polymer and Surface Engineering, Institute of Chemical Technology, Mumbai -400019; India.

**The microencapsulation process is essentially creating a barrier within the core and its wall materials. Microencapsulation provides us the solution to encapsulate materials like food ingredients, enzymes, cells, marine and vegetable oil like micrometric scale materials. Microencapsulation has a special type of capacity to improve thermostability, oxidative stability, shelf-life, and sensitivity towards the core material's temp-pressure. The encapsulation process includes various methods like emulsion polymerization, ionic gelation, spray drying, centrifugal extrusion, extrusion, and thermal gelation. This article describes an introduction about microencapsulation, methods/techniques used for encapsulation of core material, types of polymer and carrier used with their special imparting properties and application; as this technology is majorly applied in polymer, pharmaceuticals, oleochemicals, agricultural, foods, and textile industries. The most recent development in this technology is related to improvement in the controlled release of encapsulation with various physical as well as chemical processing conditions. This article gives information about microencapsulation to scientists and industries who are interested in current encapsulation technology and trying to improve some methods.**

Keywords: Microencapsulation; encapsulate; drug delivery; controlled release;

#### **1. Introduction**

The mononuclear, as well as multinuclear materials that are enclosed by a protective coating or any membrane over core material, are referred to as microcapsules. These microcapsules were developed for the medicine, which contains solid or liquid core material, i.e., to enclose one or more drugs. The core can be referred to as the nucleus and protective coatings as a sheet or wall. Similarly, the method where tiny particles of liquid or solid mass are coated or surrounded by a protective film of polymer material is microencapsulation [1]. This technique produces several tenths of micrometers to approximately 5000 micrometers film. In the process liquid is converted to solid, changing the properties protecting surrounding in bioavailability, modifying the release mechanism for every material used [2].

Two fundamentals of microencapsulation are the core material and the coating material. (Figure 1) The core material is either solid or liquid; the shape of the core material can be uniform or regular.



**Figure 1:** Constituents of microencapsulation

To maintain uniform coating shape and size of core material, the polymer used, techniques used are considered. The coating material selection requires multifaceted considerations aiming to impart flexibility, reduce



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brittleness, increase segmental mobility, and increase resistance failure by mechanical stress. Coating material properties are stability of the core, non-reactivity towards active ingredients, controlled release (depending on the external condition), and stable film development.

The examples of coating materials are natural polymers as well as synthetic polymers including polymethyl methacrylate, epoxy polymers; in natural polymers, there are many natural polymers such as chitin, chitosan [3], and sodium alginate and derivatives [4]. The most important characteristic of microencapsulation is their tiny size which corresponds to a very high surface area. This advantage is responsible for the adsorption, absorption of chemical reactions, and light scattering. There are lots of techniques used for microencapsulation, such as spray cooling, spray drying, extrusion, coacervation, lyophilization, interfacial or suspension polymerization, emulsification, in situ polymerization, top spray, bottom spray, a spinning disc, complex coacervation, centrifugal extrusion processing, fluidized bed coating (Figure 2). The appropriate technique choice is governed by the core material, application environment, approximate diameter of the particle, the compatibility of core and wall material, release mechanism characteristics, and manufacturing scale.

The main release mechanism involves slow disintegration, heat and acid/base stimulated diffusion, solvent attack, enzyme attack, and pressure. A synergy of two or more mechanisms is used today for applications. Microencapsulation safeguards the core material from the environment till the core is meant to be released. This release actuation process is a bulk of the study in this discipline, understanding the influence different stimuli have on the release of the core. The benefits are reduction in the required amount of core material which could be a flavorant for example. Therefore, it is important to understand the whole dynamic of the core, wall, and environment to find the perfect fit for every application.

Moreover, other factors influencing the release process are the core's reactivity, wall material degradation products, wall thickness, and viscosity of wall and core material. Microencapsulation has traditionally been used in pharmaceutics, agriculture, and the food industry. It is widely used to encapsulate active pharmaceutical ingredients, oils, colorants, flavorants, and microbes. Now, it is finding use outside of these industries in textiles, construction, and cosmetic industries [5].



**Figure 2:** Techniques for Microencapsulation [2]

### **2. The need for microencapsulation**

Recently microencapsulation technology is being used widely in several areas. The enhancement in microencapsulation technology demand is due to its advantages [6].

Reasons for microencapsulation (Table 1)

- I. To prevent core materials from reacting with the environment.
- II. To adapt a liquid to a dry powder.
- III. To protect the environment from potentially harmful core material.
- IV. Ease of handling of viscous material.
- V. Covering the taste, odour, flavour, and colour of the core material
- VI. To improve process ability and bioavailability

### **3. Core material**

It is the material to be coated. It may be dissolved/ dispersed or liquid/gas.

- Classification by size:
	- I. Microcapsule- more than  $5,000\times10^{-6}$  m
	- II. Microcapsule-  $0.2$ -5,000×10<sup>-6</sup> m
- III. Nano capsule-less than  $0.2\times10^{-6}$  m

By shape and construction, capsules are categorized into microcapsules and microspheres.



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**Table 1:** Core Material Distinctive Property and Reason

#### **3.1. Microcapsules**

These are particles with a core usually an active agent layered by a polymeric film acting as the wall. This can be further classified on the basis of the number of core divisions into mononuclear and polynuclear into mononuclear and polynuclear microcapsules.

#### **3.2. Microspheres**

They are systems where the core is dispersed homogeneously in a polymer. The core material is dissolved and in some cases suspended, each forming a distinct structure.

### **4. Wall material**

The wall material affects the efficiency of encapsulation and the stability of the microcapsule. Hence, the correct selection is important (Table 2). While selecting wall material some properties should be inclusive such as be able to form a cohesive film with the core material, should be chemically inert towards core material, and should contain surface properties which provides strength, required elastic behavior, required impenetrability, stability, required optical properties, etc.





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**Table 2**: Polymer and Key Characteristics

#### **4.1. The polymer in wall material**

The material encapsulating the core is referred to as the coating, membrane, shell, or wall material. Not all the coating materials used are polymers, some wax and lipids are used such as Paraffin, Bee wax, or stearic derivatives. Among various properties of the polymer, such as holding ability of core material, inertness for a bio-active agent or any chemical in the core and acting as sensor and actuator for release, the main basis of choice is between either a water-soluble (e.g. gelatin, methylcellulose, etc.) or waterinsoluble (e.g. Polymethylacrylate, silicone, etc.). Thus, the major criteria of selection of the wall material are dependent on the properties of the core material. Some of the wall materials which are compatible with the core material are shown below. (Table 3).



**Table 3:** Wall materials and core materials

#### **5. Release mechanisms**

Release mechanisms are of different kinds providing target release of the core material in a controlled and sustained manner. The release mechanisms of microencapsulated active substances depend on several factors, including the final motive of the system, the expected properties of the active substance, and the main physical, chemical, and structural characteristics of the material forming the microcapsules. The liberation of the core material at the appropriate time and targeted location are key aspects. The principal characteristics influencing the rates of the releasing mechanism of the specific system are related to the interactivity involved between the material of the wall used and the material of the core (Figure 3). Other factors influencing the release of core material are the viscosity of wall material, the proportion of wall material and core material, and volatility of core material.

Three ways in which core material is released from the capsule are mechanical rupture in the wall of the capsule, wall material getting dissolved in the changing environment, or the melting of the wall due to a temperature change. Less popular mechanisms include slow erosion of the wall (ablation) and biodegradation.

Some applications of these long-term textile materials are insect repellents, cosmetics, perfumes, deodorants, and medical textiles. In addition, the release mechanism can be classified by the migrant molecular weight (low or high).

Microcapsules with non-permeable walls are used to isolate different active compounds for specific periods, and further quick release under special conditions intentionally changed to permit release. In this sense, some examples could be cited, such as the protection against oxidation of vitamins, essential oils, or lipids in cosmetic textiles or the application of non-permeable microcapsules to textiles with enzymes and pro-oxidant chemicals in laundry detergents.

Eight different release mechanisms to obtain such effects in permeable and non-permeable microcapsules have been reported.

- 1.**External pressure:** mechanism resulting in a mechanical break of the microcapsules.
- 2.**Internal pressure:** could also allow the microcapsule wall to break, for instance, if the core-shell contains substances which, under special conditions if the



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interaction between the core and the wall material is such that under specific circumstances, (e.g., radiation activation) gets converted into gaseous products, such in the case of the production of light synthetic leather [1].

- 3. **Microcapsule walls abrasion:** This mechanism is generally used in case of fragrance release.
- 4. **Burning**: release of Fire Retardants when heat increases to a specific value
- 5. **Radiation:** Photographic and light-sensitive processes can be activated through this mechanism resulting in changes in the color of these textiles by the release of microencapsulated dyes.
- 6. **Temperature Changes:** Temperature changes can enhance release of core material. There are two distinct release mechanisms:
	- I. Temperature-sensitive: Wall swells and collapses when the critical temperature has been attained.
	- II. .Fusion-activated: Wall melts due to increase in temperature.
- 7. **Chemical reactions:** This is the case of microcapsules with ingredients added to textile washing or cleaning compositions released during the washing cycle by changes in chemical composition or pH [9].
- 8. **Enzymatic degradation:** This is the case of microcapsules, which are degraded under controlled conditions by enzymes.

#### **6. Techniques of Microencapsulation**

#### **6.1 Spray cooling**

For solidification of the capsule, cold air is sprayed. Core and wall material are made in the form of a droplet that makes up the micro particles [2]. The mixture is then passed through a chamber containing air flowing at low temperatures to allow settlement of the wall material on the core material forming an encapsulation over it.

It is highly affordable and economical primarily because of the low temperature operation. This method is economically the most viable method with a scale-up prospective; however, it has less encapsulation capacity and causes wastage of core material because of adherence to the walls of the equipment. The spray cooling technique mainly is used for the encapsulation of temperature-sensitive compounds like vitamins and minerals [11].



**Figure 3:** Release mechanism Variables

The objective of spray chilling is to produce a lipid-coated active agent such that the microparticles are rendered insoluble in water. The core and wall materials are selected appropriately and then the mixture is atomized into the cool air coming from the cooler. Whenever the mixture comes in contact with the chilled air immediately the wall material will solidify, and a protective coating is formed around the core material. There is no evaporation of water as no heat is involved during the process. The particle size ranges from 20 to 200µm.

#### **6.2. Extrusion**

Extrusion microencapsulation (Figure 4) is extensively targeted to encapsulate volatile and unstable materials inside carbohydrate matrices. It finds its main application



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in the food industry for flavor encapsulation. It is beneficial as it prevents exposure of materials/flavor compounds that are prone to oxidative reactions hence providing a longer shelf life. This is possible because of the presence of a hydrophilic glassy matrix, which doesn't allow air/oxygen to pass through. This type of microencapsulation also serves to protect thermally sensitive materials such as volatile substances, certain enzymes, and microbes. An increase of 5-10 times in the shelf-life value has been reported in the case of encapsulated flavor oils. Carbohydrate matrices are generally used in the food processing sector to encapsulate flavors. These glassy materials have good barrier properties. With the advantage of providing a better shelf-life and chemical resistance, this process possesses disadvantages such as an increase in the cost of the product; an increase in the carbohydrate content of the food product; also particle diameter increases which affect the flavor of material encapsulated.



 **Figure 4: Extrusion Technique** 

#### **6.3. Ionic gelation**

Also known as ionotropic gelation (Figure 5), it is a physicochemical process of droplet hardening by chelating polyelectrolytes with ions. This causes the polyelectrolyte molecules to cure and forms an outer film polymer gel layer.

The first reported ionic gelation procedure was experimented with nano-encapsulating proteins. [47]

The binding force of the capsule is the strong ionic forces of the species in it.

A bioactive compound is added and is clamped between the polymers forming a microcapsule or even a nanocapsule.

Advantages-

- Consistently shows higher encapsulation efficiency than popular techniques like spray drying which produces significant waste.
- Also, as opposed to solvent evaporation like techniques, ionic gelation's water based systems provide relief from any trouble for using organic solvents.

#### Disadvantages-

- Higher quantity non uniform particle size is obtained in this technique and as a result the core material loading varies proportionally.
- The ionic pair combinations and polymers tested in literature are relatively limited. On the flipside, this shows there is more work to be done.



**Figure 5: Technique of Ionic Gelation**

#### **6.4. Fluidized bed coating**

Fluidized bed coating, also known as suspension coating, is a technique that works on the principle where a liquid material is sprayed onto the core material particles and then rapid evaporation forms a layer of wall material over the core particles. This technique finds its major application in the pharmaceutical and food industries. Some commonly used wall materials are protein derivatives and starch derivatives [6]. Particle size in the range of 20-1500 micrometers can be obtained by this method. There are



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three types of fluidized bed coating techniques, top, bottom, and tangential spray. This technique efficiently applies a uniform wall. This technique is capable of coating any wall material like polysaccharides, proteins, emulsifiers, fats, enteric coating, powder coatings, gums, ethanolic solutions of synthetic polymers, melted waxes yeast cell extract, and other complex formulations. Due to many variations and choices in terms of core material, versatility can be observed in this technique when compared to other techniques. An additional layer of fat can also be imparted on a spray-dried capsule with a fluidized bed technique for better results in shelf life and protection. Out of these wall materials, the use of fats, waxes, or emulsifiers is at its initial stages and shows promising results. Here, particles are suspended in a controlled environment (temperature and humidity kept according to requirements) and then the wall material is atomized. Particle size in the range of 50-500 microns is obtained. The final concentration of wall material in the capsule formulation is dependent on the time the particles are suspended in the chamber.

#### **6.5. Spinning disk**

The spinning disk encapsulation method (Figure 6) is very easy and simple in operation, and it has high production. The advantage of this process is that it serves for a long duration. In this process, core and wall material are gushed into a rotating disk. The rotating action of the disk ensures the coating of the core particles with the wall material [13].

#### **6.6. Interfacial polymerization**

Interfacial polymerization also known as polycondensation is a method where a wall over the droplets of the dispersed core material is made by rapid polymerization of wall material. Initially, droplets are formed containing core material and oil-soluble reactive monomers which are present in an aqueous phase. Later, a monomer that is soluble in water is introduced which results in rapid polymerization of the two monomers at the interface of the droplet to form a polymer shell [14].



**Figure 6: Spinning Disk Technique.**

#### **6.7. Suspension polymerization**

In this process, the dispersion of the liquid form of monomer is poured into an agitated stabilizing medium having water containing small amounts of suspension and dispersion agents. An example can be microencapsulated hexadecane in a styrene and methacrylate copolymer.

Ease of heat, product handling, and temperature control, no viscosity effects, impurities are limited to surfactant and remnant water and process economics are the main advantages of this process.

#### **6.8. In situ polymerization**

This polymerization reaction progresses in a continuous phase instead of taking place at the interface of the droplet. There is no requirement for another monomer (in the organic phase).

Here, synthetic thermosetting microcapsules with low softening temperature phase change materials are mentioned for energy transport [15]. Improvements by using copolymers in this application have been reported in the literature [13].

#### **6.9. Emulsion phase separation**

The core material is put in either the polar or nonpolar layer of an oil-in-water emulsion (O/W) or W/O emulsion. An emulsion is organized using a surfactant. Polar, non-polar, and amphoteric substances can be integrated. The first step is dispersing the active agent in the polymer solution. The concentrated colloidal phase is then separated from the dilute phase. This is followed by gradual adsorption and settling of polymer solution on the active agent and then finally, curing of the coated polymer.

#### **6.10. Spray drying**



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This method (Figure 7) provides a wide choice of coating material. This method has good efficiency, the stability of finished products [16]. This method has huge production in continuation mode [17]. It works on the principle of dispersing core material in trapping materials. Later, hot air desiccants into the chamber in which spraying of the mixture with atomization is done. Spray drying is a continuous process in which the liquid fed is converted to dried particle form by spraying the liquid into a hightemperature medium. With this technique, we get good quality microcapsules with particle sizes ranging from 10- 50 micrometers. This is a low-cost process [18]. In this way, silver nanoparticles-polyvinyl pyrrolidone (PVP) blends with 100 ppm of silver were applied by spray drying on wool and cotton matrices to establish a new antimicrobial fabric with clear resistance over *Staphylococcus aureus*, *Escherichia coli*, and *Pseudomonas aeruginosa* [19].



## **6.11 Centrifugal extrusion processes**

This process involves two immiscible liquids that flow into a rotating two-fluid concentric nozzle. Core material present in liquid form is injected through the middle tube and the wall material is injected through the encompassing circular space. At the nozzle which is at the end of the column, a thin layer of wall material is formed at the nozzle and the core material flows into it and dislodges the layer and falls while forming droplets. Polymer wall hardens by heat exchangers or by passing through a gel. Then in the downstream stages, mechanical operations are performed to separate capsules and remaining fluid recycles [20]. This

happens continuously. Different polymers use different solidification mechanisms.

#### **6.12. Sol-gel**

This emerging technology has several advantages for microencapsulation, like its low cost, low use of chemicals, and effectiveness to obtain very high-purity materials (powders, fibers, film coatings, monoliths, or bulk structures), having a low environmental impact. This process involves the colloidal form which then gels to form a polymeric gel network. The main compound for the preparation of a sol is the precursor, normally an elemental metal surrounded by various ligands, as examples. Natural castor oil and bisphenol-A bis (diphenyl phosphate) were used as effective fire retardants after encapsulation in silica shells to previously manufactured non-woven textiles. Phosphorus-doped SiO has been reported to be effective as a flame retardant in cotton.

A novel composite based on microencapsulated paraffin in SiO was also prepared by in situ emulsion interfacial hydrolysis and polycondensation of tetraethyl orthosilicate to obtain materials with high heat storage capability and good thermal stability in textile applications [21].

#### **6.13. Pan coating**

Pan coating (Figure 8) finds major application in the pharmaceutical industry. It is a traditional method for the formation of small coated particles. The particles fall over the pan where the wall material is added steadily. Particles that are greater than 600 microns show efficient coating. An atomizer is used to spray the coating material in an aqueous dispersion or organic solution form on the core material in the tumbler. The solvent is evaporated using warm air and exhausted through a vent.

#### **6.14. Solvent evaporation**

The wall material is dissolved in a solvent. In the polymer solution now, depending on the core material solubility, the core material also dissolves or suspends. This mixture is thoroughly homogenized [22].



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 **Figure 8: Pan-Coating technique**

The solvent is generally volatile and is now removed by evaporation. This has an evaporative cooling effect. Then the microcapsules are collected for further processing (Figure 9).

Process variables studied in literature-

- 1. Types of solvent and its volume
- 2. Polymer to core material ratio
- 3. Solvent transfer rate
- 4. Water amount

The main focus is to understand and experiment with all process variables to obtain high core material loading and design release mechanisms.

But an issue is a below average encapsulation efficiency of water soluble components, since they are extracted from the organic phase to the aqueous phase.

Every technique has its own characteristics, advantages and disadvantages. These have been tabulated in Tables 4 and 5.

### **7. Applications**

#### **7.1. Sustainable Drug Delivery**

The drug is encapsulated in a polymer, and allowed to release at a pre-decided rate in response to external stimulus -to prevent its quick metabolism and exiting the body soon after administration [24].



**Figure 9: Solvent evaporation Technique.**



**Table 4** : Microencapsulation Processes & their approximate particle size [7] [8]



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**Table 5:** Summary of microencapsulation techniques [10]



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#### **7.2. Targeted Drug Delivery**

Such a delivery system serves local therapeutic effects and requires less quantity thereby, reducing side effects. It is known to be used in anesthetics. Drugs can be targeted to specific locations [26] such as antitumor micro particles targeting organs.

#### **7.3. Agriculture**

The applications in agriculture have led to lower wastage and greater safety to users and the environment [27]. Encapsulated Insect pheromones are used to disrupt the mating season of insects. This is used in the stead of Insecticides. Polymer microcapsules and Arabic gum are often used as a delivery vehicle [28]. Encapsulation protects such bioactive ingredients from the environment as well. Similar other crop production applications include micro particle-sized chemical agents used instead of pesticides and encapsulated microbes for plant nutrition. [29]

#### **7.4. Pharmaceutics** [30]

Promising avenues have been identified for therapeutic medicines, immunization, and in use of gene therapies. Certain proteins were pointed out to be benefitted from oral delivery [31]. But, so far no one-stop encapsulation solution has been developed that can manufacture all the possible scales of parameters. Irrespective of the technique, they all aim to lessen the cost of production, to escalate the stability of compounds, to cover undesirable tastes, reduce the volatility, and reduce gastric irritation, among other reasons. Commercially, gel particles and spray coating are the most implemented processes.

#### **7.5. Foods**

The food industry has been looking at using these techniques mostly for applications that include handling bio-active compounds that are unstable and to mask undesired organoleptic properties[32][33], bulk of these come with claiming health benefits as well[34]. Food additives can gradually degrade and suffer from reduced

efficacy or turn potentially unsuitable for consumption due to oxidizing [35]. Microencapsulation can provide delectable aroma release, and taste, olfactory effects, texture blending, and color modifications. Along with these benefits, it has become convenient to add minerals and vitamins. In foods that require handling large quantities of fluids, microencapsulation can convert them to powders, which are easier to handle [36] [5].

Most published Literature on food application with microencapsulation is on probiotics, lipids, flavours, antioxidants, vitamins, and enzymes [37] [38].

#### **7.8. Energy [39]**

Void microspheres filled with deuterium in the gas phase are used to harness nuclear fusion. High powered lasers are bombarded on the shells which increase density and temperature in the shell leading to the fusion of deuterium to give helium, tritium and other particles.

#### **7.9. Catalysis [40]**

Microencapsulated catalytic processes value has been recognized by pharmaceutical and specialty chemical industries. Studies have shown a trend of enhancing properties like easier recovery, reuse, and safety. Polymers are selected that resist degradation and dissolution.

More research is geared towards employing chelating and ligating groups within functional polymers to magnify the rate of reaction.

A ruthenium-based complex microencapsulated within poly (4-vinyl pyridine) with divinylbenzene was synthesized as a novel catalyst to [hydroformylation](https://www.sciencedirect.com/topics/chemical-engineering/hydroformylation) 1-hexene. The capsules were stable at elevated temperatures and showed higher recyclability than previous processes.

Different metal catalysts have been encapsulated in polyurea and showed similar recoverability and recycling benefits.

In textile processing, features like crease retentivity or visual effects on fabric surfaces- to get different surface finishes and wrinkles recovery work has been done on microencapsulated catalysts and enzymes.



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#### **7.10. Defense [41]**

In defense and important construction engineering applications- a sparked interest in self-healing polymers and composites has seen improvement in this technology. Within the matrix of structures, monomers are microencapsulated; polymerization catalyst and initiators are dispersed in the structure. Therefore, in the case of mechanical damage, the capsules break and monomers leak out and polymerize in the gaps formed.

#### **7.11. Textiles [42] [43] [44]**

In fabrics, microencapsulated colorants that are sensitive to heat or light can be made to change colors. These are known as thermo/photo-chromic effects. It has not become ubiquitous yet but has seen considerable commercialization in the short duration of its acceptance.

Microbial encapsulation is a delicate process with stringent requirements [45]. Factors such as

- **Toxicity**
- Reduced activity
- Protection
- Strength of capsule
- Diffusion of core material
- Mechanical properties
- Reaction conditions are studied

Polymers found to be used in scientific literature are Starch, Chitosan, whey protein, Alginate, Gelatin [46], Xanthan gum, casein, Polyacrylamides and Polyvinyl alcohol and co-polymers.

### **8. Conclusion**

The research in the field of micro encapsulations is gaining more popularity due to its potential to convert simple raw materials to better products. The continuous research in this field gives rise to the development of new products in all areas like initially carbonless copy paper then controlled drug release. Microencapsulation technologies are widely applicable in various areas, and research shows the scope in this field with several wall materials, each having its benefits; this leads to better quality products for us.

However, significant work is required to discover and formulate new wall materials. Moreover, improvisation and optimization of existing encapsulation methods are necessary to use microencapsulation and its inherent applications properly.

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