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A Comprehensive Review of Microencapsulation Techniques and Application

Navyashree P S*, Sumanji Bala, Navaneetha B N

Department of Pharmaceutics, Vivekananda college of pharmacy, Bangalore-55, Karnataka, India.

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ABSTRACT:

Microencapsulation is a process where tiny particles or droplets are surrounded by a coating, forming microcapsules with various useful properties. This technique is used in multiple industries, including pharmaceuticals, food, cosmetics, agriculture, construction, and more. Microencapsulation provides benefits such as controlled release, protection of sensitive ingredients, and enhanced stability. The process involves different methods, including spray drying, solvent evaporation, in situ polymerization, interfacial polymerization, suspension polymerization, emulsion polymerization, coacervation, and sol-gel method. Each method has its advantages and disadvantages, and the choice of method depends on the specific application and desired properties of the microcapsules. Microencapsulation has various applications, including drug delivery, pharmaceuticals, food industry, taste masking, flavors, fragrance protection, and self-healing materials. The evaluation of microencapsulation includes parameters such as percentage yield, incorporation efficiency, particle size, angle of repose, scanning electron microscopy, and drug release. Overall, microencapsulation is a versatile technology with a wide range of applications and benefits.

Keywords: Microencapsulation, solvent evaporation, controlled release, drug delivery, encapsulation method.

INTRODUCTION

A continuous layer of polymeric materials envelops or coats tiny droplets or particles of liquid or solid material in a process known as microencapsulation. Microencapsulation is the process of encasing small capsules in a coating or embedding them in a homogeneous or heterogeneous matrix to produce tiny capsules with a variety of advantageous properties. Microcapsules are a contemporary dosage form that is frequently used for various medical applications, including enhancing the stability of active ingredients. A physical barrier separating the product's core compound from its other constituents can be created by microencapsulation. Using this method, gas compounds, solid particles, or liquid droplets are trapped in thin layers of a food-grade microencapsulating agent.[1]

Liquids can be turned into solids, surface and colloidal characteristics can be changed, the environment can be protected, and the release characteristics or availability of coated materials can be controlled through microencapsulation. The small size of the coated particles and their subsequent application and adaptation to a wide range of dosage forms have made microencapsulation unique, even though macro packaging techniques can achieve several of these qualities.[2]

With an average diameter ranging from 1μm to several hundred micrometers, microcapsule-based molecules are the most appealing of the many functional structures created to meet these stringent requirements. They can overcome the drawbacks of single constituents to satisfy the utilization process and offer a profitable solution in oil recovery and oil well cementation. In this review, we provide an overview of microcapsules, their uses in oilfield chemicals, recent developments in evaluation, and our thoughts on potential future research avenues.[3]

Different structure of microspheres and microcapsules

Microcapsules contain at least one distinct domain of active agent, and occasionally more, while formulations depicted as microspheres are generally thought to consist of a fairly homogeneous mixture of polymer and active agent. Several microcapsules are depicted in the figure. Microcapsules transform into microparticles as their domain and subdomain get smaller and smaller. Microcapsules typically have a core substance and are spherical particles between 50 and 2 nm in size.[4,5] Strict definitions state that microspheres are spherically empty particles. However, the terms microcapsule and microsphere are frequently used interchangeably.[6] Given the captivating qualities and broader range of uses of microcapsules and microspheres, it is appropriate to

examine their applications in controlled drug release formulations. Since spherical microparticles are specifically referred to as "microspheres," microparticles with an enclosed core are addressed by the subcategory of "micro-capsules.[7,8]

Techniques for solvent evaporation have grown in utility when compared to alternative approaches. This technique allows for particle size control in the nano to micrometer range, but it should not be overlooked how crucial it is to carefully choose the encapsulation materials and other conditions in order to achieve high encapsulation efficiency and a low residual solvent content. Pyrrolidone-based microcapsules are being prepared using the solvent evaporation method, which has a high drug entrapment efficiency.[9] the main objectives of microencapsulation, as well as the procedures and methods used. This encompasses the physiochemical, chemical, physical, and other techniques. In particular, the application of microencapsulated microorganisms in inflammatory bowel disease, colorectal cancer, cardiovascular disease, renal disease, and other conditions is covered in detail in this paper. Mammalian cell microencapsulation has been reported for parathyroid insufficiency, diabetes, and hepatic disorders.

Capsule

Capsules can be divided into two groups: microcapsules (>5,000m), microcapsules (0.2 to 5,000m) and nano capsules (0.2m) Microcapsules and microspheres are two categories of capsules based on their shape and construction. Particles known as microcapsules have a relatively central inner core that contains the active ingredient and is coated in a polymer layer that serves as the capsule membrane. The difference between mononuclear and polynuclear microcapsules is whether the core is divided.[10]

Wall Material

The stability and effectiveness of the microcapsule are determined by the wall material selection, which makes it crucial. The wall material should be nonreactive to the core; it should be able to seal and maintain the core inside the capsule; it should provide maximum protection to the core against adverse conditions; it should not taste unpleasant in the case of food applicability and economic viability; it should be non-toxic in the case of food applicability and economic viability.[11]

Advantages[12,13,14]

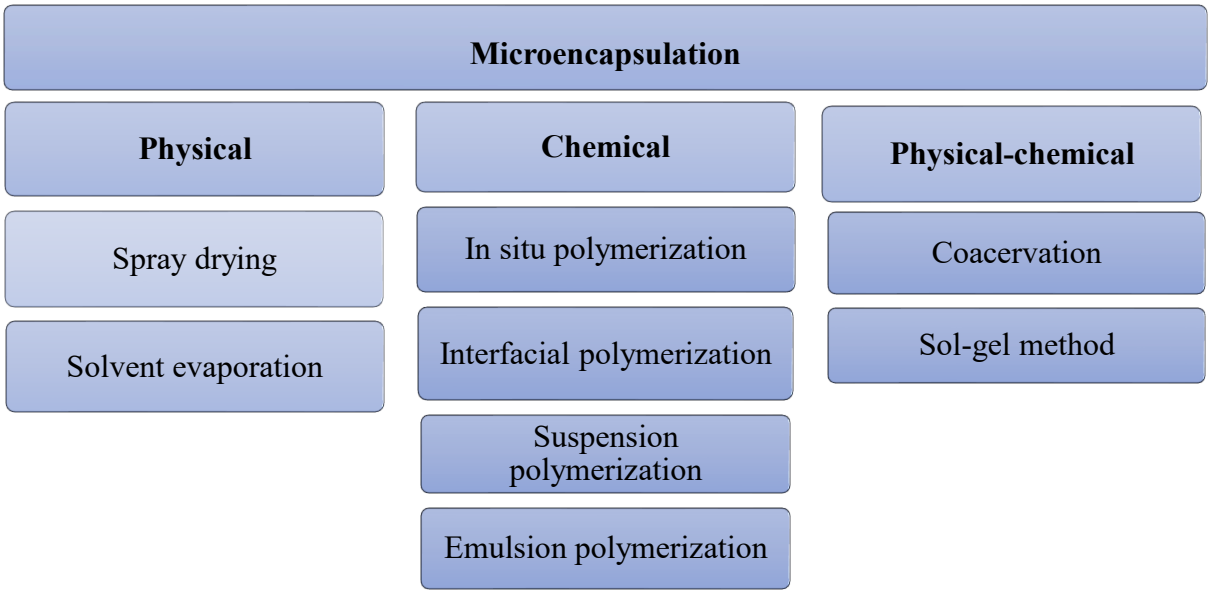
- Easy handling of product powders
- High production rates and efficiency
- Reproducibility
- Low operating costs
- Its use in a wide range of compounds with different compositions and polarities.
- Short processing time
- Heat resistance, stability of products
- Controlled release of actives
- Solubility and stability of hydrophobic actives
- Reduction of compound loss and volatility
- Suitability for heat-sensitive actives and good choice for temperature-sensitive compounds.
- The product has outstanding stability and encapsulation efficiency
- Making it suitable for heat-sensitive food ingredients.

Disadvantages

- Degradation of highly temperature-sensitive compounds

- Scarcity of good wall materials
- Moderate yields in cases of small batches
- Agglomeration proper ties of the microcapsule powder
- Rapid release of actives
- Difficulties with viscous solutions
- Long processing times
- High energy, high production cost and highly sensitive to pH and ionic strength
- Use of a limited number of emulsifiers.

Techniques of microcapsulation



Spray drying

This process is widely used in the food industry for manufacturing of milk powder, in addition to the pharmaceutical and cosmetic sectors. The process has found use in encapsulation because of its widespread use and the accessibility of equipment. The basic idea behind the spray drying process is to dissolve and emulsify the core material in an aqueous solution of the carrier material. followed by atomizing the mixture in a hot chamber where the active particle is coated and smaller water molecules evaporate. Nevertheless, highly volatile fragrance compounds evaporate more quickly than water, so it's critical to select the appropriate carrier to preserve volatile compounds while letting water evaporate. When choosing an appropriate carrier, the following factors need to be taken into account.[15,16,17]

- Good water solubility
- Good emulsifying qualities
- Low viscosity at high concentrations (45 percent)
- Low hygroscopicity, taste or odor.

1. creation of a slurry or emulsion using the carrier and primary ingredient. With the high pressure and high mixing speeds emulsions are typically made in colloid mills. The product is subsequently subjected to additional mechanical processing techniques, including ultrasonic emulsification, micro fluidization, and high-pressure homogenization. These techniques are employed to stabilize the emulsion for a predetermined amount of time. The best encapsulation results are obtained with moderate emulsion viscosity values, which also influence the next drying step. Excessively viscous emulsions may clog the feeder nozzle or collect on the chamber walls.[18]

2. The emulsion's dispersion and atomization. An atomizer is used to pump the emulsion into the drying chamber. High-pressure nozzles and centrifugal wheels are two of the most popular methods for atomizing the emulsion. The atomizer divides the emulsions into tiny droplets, the size of which is determined by the atom's pore size.[19,20]

3. Particle gathering. In total, the encapsulation 10 of 20 process takes a few milliseconds to a few seconds. In the spray-dried product the amount of the active ingredient can range from 2 to 50%. The availability of the equipment, its affordability, and its excellent encapsulation outcomes are the benefits of spray drying. The techniques of product concentration and agglomeration are complementary to spray drying. The result is the production of larger encapsulates in both situations. Compaction occurs when products that have been spray-dried are compressed under high pressure into lumps and then crushed into tiny pieces that range in size from 0 to 7 mm to 3 mm (good for tea applications). Low porosity is the result of compaction. Agglomeration is the fluidization of the spray-dried product.[21,22]

Solvent evaporation[23]

Microencapsulation by solvent evaporation is a widely used technique to encapsulate active ingredients, such as drugs, flavors, or nutrients, within a polymeric matrix. This method helps in protecting sensitive compounds, controlling release rates, and improving stability. Here's a detailed breakdown of the process.

Oil/Water emulsion followed by solvent evaporation

Dissolution or dispersion of the drug substance is achieved by the polymer/solvent system. After that, it is continuously stirred into the aqueous phase. The system is continuously agitated until the solvent separates into the aqueous phase and evaporates. The hardened microsphere produced by this process contains the drug's active moiety. The oil phase has been dispersed in the continuous phase using a variety of techniques. Using a propeller-style blade connected to a variable speed motor is the most popular technique. The final product has a significantly smaller particle size than the emulsion made by traditional agitation because high shear is utilized to create the emulsion. Other techniques include potentiometric dispersion, sonication and also used for micro fluidizer to create micro-emulsions.

Water-oil-water multiple emulsion system

The low encapsulation efficiency of water-soluble drugs made using the traditional water/oil emulsion solvent evaporation method was reportedly resolved by this microsphere preparation technique. The most common organic phase in which polymer [usually polylactic-co-glycolic acid, or PLGA] dissolves is DCM (dichloro methane). To create the water-to-oil (w1/o) primary emulsion in this organic phase, a high-speed homogenizer running at 15000–20,000 rpm for approximately 30 seconds is used to emulsify an aqueous drug solution. For 30 seconds, this primary emulsion is added to an external aqueous phase that contains a surfactant (polyvinyl alcohol is most commonly used to prepare $w^1/o/w^2$ emulsion). It is then stirred at 300 rpm for three hours at room temperature to allow DCM to evaporate, or it can be done under vacuum. After ultracentrifugation and filtration, the resulting microsphere is collected and lyophilized, which lessens the burst effect.

Multiple emulsion of the Water/Oil/Oil or Water/Oil/Oil/Oil type

A multiple emulsion solvent evaporation technique was used to create a multiphase microsphere of PLGA containing water-in-oil (W/O) emulsions. The polymer was dissolved in acetonitrile, and the continuous phase of the encapsulation process was made up of light mineral oil. The theoretical drug loading efficiencies of model water-soluble compounds varied from 80 to 100%, depending on the particular preparation conditions. transverse cross sections of the W/O/O/O type multiphase microsphere, which belonged to the class of reservoir-type drug delivery devices, were examined using scanning electron microscopy. By using this kind of multiple emulsion system, the primary water-in-oil emulsion can be enclosed in a polymeric microsphere. The primary emulsion's oil keeps the internalized protein from coming into contact with the polymer/solvent system, preventing the protein from potentially becoming denaturated by either the solvent or the polymer. Similarly, reactive pro has the potential to cause polymeric degradation.

A modified water-in-oil-in-water ($W_1/O/W_2$) double emulsion solvent evaporation

They have developed gas foamed open porous biodegradable polymeric microspheres.In order to deliver cells, highly opened porous biodegradable polymeric microspheres were created and used as injectable scaffold micro carriers. This was achieved by modifying the water-in-oil-in-water double emulsion solvent evaporation process. Carbon dioxide and ammonia gas bubbles spontaneously form when ammonium bicarbonate, an effervescent salt, is added to the primary W1 droplets and spontaneously formed when the solvent evaporates. This makes the resulting microspheres have well-connected pores and stabilizes the main emulsion. We provided an explanation of the porous microspheres produced under various gas foaming circumstances. Different gas foaming conditions led to the development of surface pores with a diameter of up to 20 µm. The concentration of ammonium bicarbonate increased with the diameter, indicating sufficient cell infiltration.

In situ polymerization

The in situ polymerization involves prepolymerization of the monomers followed by their polymerization on the surface of the droplet to form a shell around it; general steps are as follows: preparing the O/W emulsion by adding PCMs to an aqueous solution of surfactant; forming a prepolymer solution, which is prepared from monomer and hardener mixtures via prepolymerization; addition of the prepolymer solution into the O/W emulsions with subsequent adjustment of reaction conditions to suitable ones; and microcapsule synthesis. Using polyurea formaldehyde (PUF), polymelamine formaldehyde (PMF), and polymelamine urea formaldehyde (PMUF) the decanoic acid was effectively microencapsulated. While the microcapsules with PMF shells demonstrated greater thermal stability, the microcapsules coated with polyurea formaldehyde demonstrated superior heat storage capacity, reduced heat resistance, and diminished mechanical strength but smaller thermal energy storage capacity. The PMUF-encapsulated microcapsules showed the highest heat resistance among all the systems.[24]

Interfacial polymerization

When two reactive monomers are dissolved independently in the aqueous and oil phases, interfacial polymerization takes place at the oil-water interface with the help of an initiator. Interfacial polymerization generally involves the following steps: creating an O/W emulsion with PCMs and hydrophobic monomer; adding the hydrophilic monomer to start the polymerization process under the right circumstances; and filtering, washing, and drying to create microcapsules. This technique is typically used to prepare organic shell materials like polyurea and polyurethane. The interfacial polymerization method was successfully used to microencapsulate butyl stearate (BS) and paraffin as binary core materials using polyurea/polyurethane as the shell material. The phase change temperature of the microcapsules was adjusted by varying the ratio of the two core materials.

Suspension polymerization

Using surfactants and mechanical stirring, dispersed droplets comprising PCMs, monomers, and initiators are suspended in a continuous aqueous phase during the suspension polymerization process. The oil-soluble initiator's free radicals are then released into the emulsion system to start the monomerization process at the right stirring rate and temperature. used thermochromic pigment/PMMA shells with five distinct pigment/MMA ratios ranging from 0 to 14 weight percent to successfully microencapsulate n-octadecane using suspension polymerization. By suspension polymerization the microencapsulation was achieved with polystyrene and a range of suspension stabilizers.[25]

Emulsion Polymerization

During emulsion polymerization, the dispersed phase of PCMs and monomers is initially suspended in a continuous phase as discrete droplets using harsh agitation and surfactants. Then polymerization is started by adding water-solution initiators. Organic materials like PMMA and polystyrene are frequently polymerized using this technique to create the shells of microcapsules. Using polymethyl methacrylate (PMMA) and four additional PMMA-hybrid shell materials, stearic acid (SA) cores were encapsulated via emulsion polymerization. The microcapsules were typically between 110 and 360 µm in diameter and 17 to 60 µm thick. Each microcapsule had a degradation temperature of over 290°C and a heat storage capacity of less than 80 J/g. Using PMMA shells and emulsion polymerization, paraffin eutectic mixtures (PEM) with four different contents were successfully microencapsulated.

Coacervation

Coacervation microencapsulation processes take place in colloidal systems, where dispersed microcapsule cores are encircled by macromolecular, colloid-rich coacervate droplets, which create viscous microcapsule shells that are cemented with crosslinking agents. In practice, an oil-in-water emulsion is created by emulsifying water-insoluble actives into a continuous aqueous phase that

contains a dissolved macromolecular colloid. Controlled changes to variables like pH, ionic strength, temperature, or solubility cause the coacervation process. The surface tension differential between the hydrophobic material, water, and the coacervate phase drives shell formation. Non-covalent hardening by hydrogen bonding between molecules or cross-linking and the creation of new covalent bonds are two methods for permanently hardening the microcapsule shells. frequently both.[26]

Sol gel method

The sol is defined as a stable mixture of colloidal particles in the solvent by the term sol–gel, which is an acronym for solutiongelation in the solvent and the gel as a three-dimensional network connected by intermolecular forces and covalent bonds (e.g. G. hydrogen bonds and van der Waals forces) made up of the sol particle aggregates. An extremely versatile technique for creating a wide variety of materials, the sol-gel process uses organic or inorganic metal solution-gelation ("sol-gel") polymerization at low temperatures to create a variety of nano-inorganic or composite materials. G. broad variety of nano- and micro-structures, and only mild conditions are needed. This approach uses a solution (e.g. G. sol) is first created by combining a solvent, catalyst, and complexing agent with a selected precursor, such as a specific metal alkoxide. As a result, a stable colloid or polymer dispersion is created.[27]

Evaluation of Microencapsulation

1. Percentage Yield[28] :

The microcapsule's measured weight was divided by the total of all the non-volatile components that went into making it.

% yield = (Actual weight of product / Total weight of excipient and drug) x 100

2. Incorporation Efficiency :

For 12 hours, 25 mg of crushed microcapsules were added to a 100 ml volumetric flask, mixed with a small amount of ethanol to bring the volume up to a pH of 6 to 8, and then stirred. A UV spectrophotometer 1800 was used to measure absorbance at 206 nm after the solution had been stirred and filtered through Whatman filter paper. The filtrate was then diluted appropriately.

Micromeritic Properties[29] :

3. Particle Size:

The optical microscopy method was utilized to determine the average particle size of the microcapsules. After a small number of microcapsules were dissolved in glycerin and spread out on a sterile glass slide, the average size of 100 microcapsules per batch was calculated.

4. Angle of Repose:

The angle of repose is determined. The microcapsules were made using the fixed funnel technique.

Angle of repose $\theta = \tan^{-1} (H/R)$

Where, H = Height of the pile; R = Radius of the pile

5. Scanning Electron Microscopy[30] :

The following procedure was used to prepare the samples for SEM analysis. Using a scanning electron microscope, the microcapsules' form and surface morphology were examined. Double-sided sticky tape was used to mount the microcapsules directly onto the SEM sample stub. Under low pressure (0.001 mm Hg), they were covered with 200 nm thick gold film. The accelerating voltage used to view the microcapsules was 10KV.

6. Drug Release[31] :

Using the g USP XXII type 2 basket method (900 ml of pH 6.8 phosphate buffer, 100 rpm, 37±0.5O C), the in vitro dissolution profile of each formulation was ascertained. 100 mg worth of microcapsules were put into the dissolution apparatus's basket. At appropriate intervals, a 5 mL aliquot was taken out of the dissolution media, and the same volume of dissolution medium was added

back in to maintain a constant total volume. Following a suitable dilution if necessary, phosphate buffer with a pH of 6point 8 was used as a blank to measure the samples absorbance at λmax 206 nm. findings from studies on drug release in vitro.

Application[32]

1. Pharmaceuticals

- Drug Delivery: Improves the bioavailability and stability of medications. Long-lasting therapeutic benefits and fewer adverse effects are possible with controlled release systems.

- Taste Masking: This technique, especially in pediatric formulations, improves patient compliance by masking the taste of unpleasant medications.

2. Food Industry

- Flavor and Fragrance: Preserves delicate flavors, colors, and scents from deterioration, guaranteeing stable flavor and extended shelf life.

- Nutraceuticals: Vitamins, minerals, and probiotics are kept potent and assured of targeted delivery in the digestive system by encapsulating them.

3. Cosmetics and Personal Care

- Active Ingredient Preservation: Improves stability and efficacy by preventing the deterioration of delicate ingredients like vitamins, antioxidants, and essential oils.

- Controlled Release: Enables the slow release of scents or skin-active ingredients for longer-lasting results.

4. Agriculture

- In agriculture, encapsulation can shield agrochemical from environmental deterioration and offer controlled release to boost efficacy and lessen environmental impact.

5. Construction Materials

- Self-healing Concrete: Concrete can be treated with microencapsulated healing agents to patch cracks and prolong the life of buildings.

6. Adhesives & Coatings

- Microencapsulation releases healing agents in response to damage, thereby generating self-healing properties. Additionally, it facilitates the creation of controlled-release adhesives for a range of uses.

7. Printing and Packaging

- Specialized inks, adhesives, and coatings with special qualities, such as self-healing or controlled release, are being developed for printing and packaging.

Conclusion:

Microencapsulation is a versatile technology with diverse applications across industries, enhancing stability, controlling release, and improving efficacy. Its techniques, such as spray drying and coacervation, offer various benefits. Microencapsulation's applications span pharmaceuticals, food, cosmetics, agriculture, and construction. It improves drug delivery, flavor preservation, and targeted nutrient release. This technology's value lies in its ability to control release and enhance stability. Microencapsulation's potential continues to grow across industries. Its benefits make it a valuable tool.

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