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OVERVIEW OF MATERIALS AND TECHNIQUES FOR ENCAPSULATION OF NATURAL PRODUCTS: A MINI-REVIEW

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ABSTRACT: In the recent advancement of the food and pharmaceutical industry, the encapsulation process has gained interest in the production of functional food and the sustained release of drugs to the target site. The designing and the fabrication of micro or nanocapsule has become the active field of research over the few decades. Encapsulation increases the shelf life of the bioactive materials, increases their stability and protects the bio-actives from the harsh environment. Therefore, the encapsulation process has a great potentiality in the development of functional food and drugs. Many bioactive materials, oils, probiotics, and drugs are encapsulated in a microcapsule made up of varieties of polymeric and non-polymeric substances. Successful encapsulation of materials depends on the choice of the wall materials and the correct techniques adopted for the formation of the microsphere. In this review, an attempt has been made to discuss the different materials and the techniques used in the encapsulation process with reference to the food and pharmaceutical industry.

INTRODUCTION: Encapsulation is a process by which a substance or a mixture of substances are entrapped in another material system. The nature of the substance that is encapsulated are liquids, solids, and gas also. Nowadays, nanomaterials are targeted for encapsulating the drugs molecules since it enhances the loading capacity and reduces the systemic toxicity of the drugs. The major limitation of the bioactive materials from plant sources is their hydrophobicity, less chemical stability, and short-term effectiveness.

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The encapsulation process overcomes these challenges and provides a means of delivering hydrophobic drugs to the target sites, increasing the shelf life of the bioactive substances. Thus, it becomes an important tool to preserve the stability, bioavailability of the active materials and improve the delivery of these compounds into foods and organisms. It also protects the compounds from various physicochemical agents [1]. The materials that are used for encapsulation are of varied types.

Starch and its derivatives, proteins, gums, lipids, and combinations are commonly used to encapsulate in food industries. Apart from that, biodegradable synthetic polymers are also routinely used for the development of microcapsules. Polymer materials have a wide range of applications for the development of microcapsules. Recently, a method of formation of microcapsules from the nanosponge derived from polymer β-cyclodextrin was described [2] that showed the effective result of encapsulating piperline, an alkaloid that is a poor soluble substance. Various methods are employed for encapsulation purposes, including spray drying, liposomal entrapment, coacervation, inclusion complexation, cocrys-tallization, nanoencapsulation, freeze-drying, emulsion, *etc*. Depending on the nature of the bioactive materials to be encapsulated and their usage, selection of wall materials and different techniques for the formation of microcapsules are adopted.

Over the past few decades, designing the encapsulating materials and delivering bioactive agents to the target sites has been a major active research area. In this review, an attempt has been made to review the materials utilized and techniques used for encapsulating the materials with reference to food and bioactive materials used in the food and pharma industry.

Materials Used for Encapsulation: Depending on the application, the coating material for encapsulation is designed to serve a specific purpose. The selection of coating material is an essential component for the development of any micro or nanocapsule. This material decides the chemical and physical properties of the resultant microcapsules. Moreover, the material used for encapsulation should be inert to the core active ingredient and should not change the core materials' chemical properties and stabilize the core materials. The polymers that are used for encapsulation are broadly classified into two types- Natural polymers and synthetic polymers.

Natural Polymers: The natural polymers used for encapsulation include natural gums like arabic, alginates, carrageenans, *etc*. The protein-based natural polymers include soy protein, gelatin, albumin. The carbohydrates-based natural polymers are maltodextrin and cellulose derivatives. These natural polymers are biodegradable and are biocompatible in nature.

Alginate: Alginate is the natural polymers that is obtained from brown algae (Phaeophyceae) [3,] including *Laminaria hyperborea*, *Laminaria digitata*, *Laminaria japonica*, *Ascophyllum nodosum* and *Macrocystis pyrifera* [4]. It is a linear polymer containing blocks of (1, 4)-linked β- D-Mannuronate (M) and α-L-guluronate (G) residues. The blocks in turn, are composed of consecutive residues of G (GGGGGG), consecutive M residues (MMMMMM), and alternate M and G residues (GMGMGM) [3]. Based on the length of M and G residues, more than 200 different alginates are currently being synthesized [5]. Alginate has wide application in forming hydrogel bead ranging from preservation of human stem cells in hydrogel bead [6], encapsulation of bacteria [7], encapsulation of egg protein [8]. Recently encapsulation of plant protein in alginate has been reported [9].

In combination with other materials, Alginate provides the better stability of the core compounds as reported in case of wheat germ oil [10]. It was found that the addition of corn starch to the alginates improved the physicochemical properties of the bead and provided stability during storage. Alginate in combination with other wall materials, could be a promising material in the development of functional food and nutraceuticals to enrich the nutrients of the food products.

Carrageenans: Carrageenan is a linear sulphated polysaccharide that is extracted from the red marine algae. Chemically it is made up of β – D-galactose and 3,6- anhydro-α- D- galactose units containing sulfate groups [11]. It has a wide application in the food and drug delivery system. It was also reported to have been used as a gelling agent and viscosity-enhancing agent for controlled drug release [12]. It was used for making nanomaterials for coating food bioactive ingredients [13] and for making hydrogel beads for encapsulating enzymes and probiotic cells as a whole [14, 15]. With other wall materials, Carrageenan is routinely used in the development of microbeads and offers better bead properties.

A study that was conducted with encapsulated *Brucea javanica* oil with a mixture of alginate carrageenan and found to have an enhanced oil encapsulation efficiency, better gastric retentive properties, and release properties [16]. K-carrageenan, together with tween 80 was reported to develop zein nanoparticles [17], which offered great stability to the system that could be used as a delivery system for fat-soluble drugs. Moreover, it also offers high biological efficacy of the encapsulated product as reported in a study where microcapsules stabilized by carrageenan and β-lactoglobulin for epigallocatechin-3-gallate (EGCG) was also found to have high biological efficacy [18].

Albumin: It is a type of water-soluble protein and is found in varieties of sources, including human as human serum albumin (HSA), α- lactalbumin in milk, Ovalbumin in egg white, and bovine serum as bovine serum albumin (BSA). This protein is found to be stable at the pH range of 4-9 and can stand the temperature of 60 °C up to 10 h [19]. Albumin is reported to bind a diverse range of compounds and can transports, deliver, and clears drugs [20]. Because of these properties, it is used in the encapsulation process for forming micro to nanocapsules. Nanoparticles formed from bovine serum albumin (BSA) offered a better source in the delivery of drugs to the target site. Resveratrol encapsulated nanoparticles in folate-conjugated human serum albumin were reported to increase the efficacy of resveratrol than the un capsulated one and also increased loading capacity and bioavailability [21]. In a study, it was demonstrated that the carvacrol, which is a potent terpenoid from a plant source, when encapsulated in bovine serum albumin (BSA) to form nanoparticles, resulting in a considerable increase in the efficacy of carvacrol *in-vivo* system of rats [22].

Maltodextrin: It is a polymer of carbohydrate linked by α- 1, 4-glucosidic bonds. It can be produced by enzymatic hydrolysis with or without acids of the starch. It is primarily used for the encapsulation of oil. It was reported to provide oxidative stability for oil encapsulation but showed poor emulsifying capacity and low oil retention [23]. Together with another encapsulant like soy extract, maltodextrin was used for encapsulating probiotics [24]. With a different combination of gum Arabic and maltodextrin, it was used for encapsulation of egg peel extract which is a source of natural color and antioxidant [25]. Zorzenon *et al*. [26], successfully encapsulated the ethanolic extract of stevia in maltodextrin, and the resulting microcapsules showed higher antioxidant activity under different *in-vitro* digestion. In combination with other wall materials, maltodextrin has been found to offer a shelf life stability to the bioactive compounds [27]. Thus encapsulated bioactive could serve as a functional food in the future trend.

Synthetic Polymers: Over the past few decades, biodegradable synthetic polymers have gained interest in the field of encapsulation for the development of gel beads for drug delivery systems, food engineering, etc. The commonly used synthetic polymers are polyethylene glycol, Poly-L-lysine, polyvinylpyrrolidone, poly (vinyl acetate), poly (acrylic acid), *etc*.

Polyethylene Glycol: Polyethelene glycol (PEG) is a polyether having versatile applications. It is soluble in water, ethanol, benzene, dichloroethane and insoluble in diethyl ether and hexane. PEG forms a steric barrier by forming a hydration layer. In combination with other natural polymers, it is used in forming microcapsules. It was reported that microsphere or nanosphere coated with PEG with lipid [28] alginate, chitosan, and poly-L-Lysine provide the highest cell viability [29] and the controlled release of the drug to the target site.

Polyvinylpyrrolidone: Polyvinylpyrrolidone (PVP) is also known as polyvidone is a polymer made up of monomer N-vinylpyrrolidone. It is hygroscopic in nature. Being biodegradable in nature, it is used to encapsulate both hydrophilic and hydrophobic drugs [30]. A different formulation like hydrogels, films, fibers, micro to nanoparticles with PVP has been reported [31]. It has an importance of the development of encapsulated nanoparticles for mammalian cells [32] and drugs [33].

Methods of Encapsulation: There are a variety of techniques used in the formation of microsphere. Broadly these techniques are classified into two types depending on the starting materials used in microencapsulation. In the first type, the starting materials are monomers/polymers, where chemical reactions occur along with sphere formation. In the second type, the starting materials are polymers, where no chemical reactions are involved in microsphere formation; only fabrication occurs. Generally, the selection of microencapsulation technique depends on the choice of materials and its nature. Some of the encapsulation techniques are discussed below in this article.

Emulsification: In emulsion techniques, the formation of microparticles or nanoparticles is dependent on the nature of the core phase used for emulsification.

If the core phase is hydrophobic, it is termed as oil-in-water (o/w). When the core phase is aqueous, it is termed as water-in-oil (w/o). The emulsion is formed by adding the core phase to an excess of the second phase containing the emulsifier with vigorous stirring. This technique has a wide application in pharmaceutical formulation [34]. This technique has several advantages over other encapsulation techniques, as this technique is relatively simple and does not require expensive instruments [35, 36]. Moreover, both hydrophilic and lipophilic substances can be encapsulated by this method [37, 38]. The success rate of the emulsion technique is limited in the case of encapsulation of protein drugs due to the high shear stress involved in manufacturing conditions [39, 40]. Recently, the encapsulation of probiotics by this technique has also been reported [41, 42].

Extrusion: This method involves the dispersion of core material in the molten carbohydrate mass, which is then subjected to hardening in a dehydrating fluid. The common dehydrating fluid is isopropyl alcohol. The hardened materials are broken into small pieces and dried. The extrusion method was first patented in 1957 [43]. Recently polymersome encapsulated hemoglobin and encapsulation of polyphenols by this technique have been reported [44, 45]. The advantage of this technique is that it provides true encapsulation where core materials have the maximum chances of getting surrounded by the wall materials.

Spray Drying: Spray drying for encapsulation is one of the most commonly used techniques. This technique is used to convert the liquid materials into dry powder form. It involves the formation of emulsion or suspension that contains the core and wall material which is followed by atomization in a drying chamber that is circulated with hot air. The water usually evaporates in hot air to form powder [46].

This technique has wide application in encapsulating bacteria [47], hormones [48], oils [49,] and extensively applied in the production of functional food, nutraceuticals, and pharmaceuticals. A high concentration of bioactive compounds encapsulation from the plant extracts by this technique and its successful efficacy was also reported [50]. The spray drying technique offers numerous advantages over other encapsulating methods. It is an efficient tool for large-scale production of powder with rapid drying and controlled particle size.

Freeze Drying: Freeze drying technique is used for sensitive compounds whose properties may be destroyed by the heat-sensitive method like spray drying. In freeze-drying, dehydration is carried out by sublimation of a frozen sample [51]. This has a wide application in encapsulating bioactive compounds from plant sources [52, 53, 54,] heat-sensitive probiotics [55]. The advantage of freeze-drying is that it provides better stability to physicochemical and biological properties of peptides [56]. This technique is simpler than the other techniques and involves lesser steps. Moreover, the product reconstitution is easy and fast [54].

Liposomal Entrapment: In this technology, a drug or bioactive compounds are entrapped in fat-like particles, preferably phospholipids. The properties of liposomes largely depend upon the phospholipid composition, size, and the methods of preparation [57]. This is the newest emerging encapsulation technology used for the entrapment of drugs. Liposomes possesses both hydrophobic and hydrophilic characters which provides a better drug delivery system. Moreover, drugs entrapped in liposomes have fewer side effects. Because of its amphiphatic nature, both hydrophobic and hydrophilic compounds can be encapsulated by this technology. Various types of unstable compounds from vitamins [58,] bioactive compounds [59], bioactive hydrophilic globular proteins [60] have been encapsulated by this method.

Coacervation: Coacervation is also called the phase seperation technique. This technique is employed for the encapsulation of hydrophilic molecules. The polymer types used and their number in the formation of coacervate may be termed as simple coacervation and complex coacervation. In complex coacervation, cationic and anionic polymers are used to form a coating solution rich in a polymer called coacervate; usually, polymers used are of proteinaceous in nature and polysaccharide molecules [61]. The complex coacervation technique has better-controlled release characteristics and has an advantage over other techniques having high loading of the core material with high encapsulation efficiency [62]. This method was successfully employed for the encapsulation of probiotics as well [63] and offered better cell protection at neutral pH. It has been reported that encapsulation by this technique increases the oxidative stability of oil [64].

CONCLUSION: Microencapsulation technique is used to protect the active ingredients and convert them into solid particles. The process is useful in the food and pharmaceutical industry. It provides a means of protecting the bioactive constituents, increasing the shelf life, and providing oxidative stability and controlled release of the drugs. The encapsulation process is largely dependent upon the types of biomaterials used for encapsulation and the techniques adopted for the formation of microcapsules or nanoparticles. In the pharmaceutical industry, suitable methods are required to improve the efficacy, shelf life of the bioacive compound, and delivery to the therapeutic agents' target site and bioavailability. Apart from that, encapsulation provides a promising tool for developing functional food. Research is still continuing in this field to develop better methods of producing microcapsules to meet the new challenges and new applications.

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