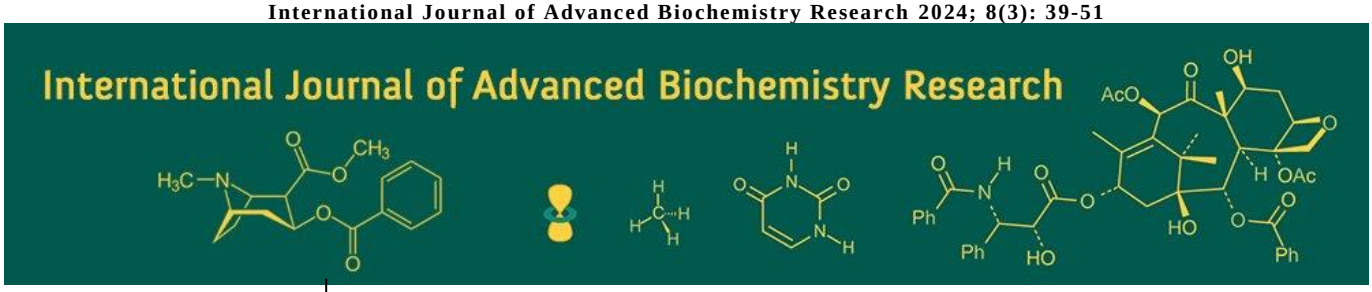

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Encapsulation technologies for the delivery of spice extractives: An overview

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Abstract
Nowadays consumers are very much conscious about their health and are looking for natural products with nutritional benefits. Most natural products like spice extractives contain functional ingredients which may degrade rapidly due to the presence of external atmospheric factors like oxygen, moisture, heat, light etc. It is complicated to apply them directly to different applications. To overcome such issues, several recent technologies like encapsulation have been developed. Encapsulation is a promising method extensively employed for safeguarding core materials from factors like light, heat, and oxygen. This advanced technology enhances stability, boosts bioavailability, masks bitter taste, facilitates controlled release, preserves functional characteristics, and finally improves handling practices. Selecting the appropriate microencapsulation technology and the suitable carriers is determined by the product's specific application and the associated processing conditions. This review paper highlights the most important encapsulation techniques and a few of their implementations in the encapsulation of spice extractives.

Keywords: Active ingredients, encapsulation, protection, spice extractives, wall materials

Introduction
In the modern era, the consumer demand for novel functional food products containing bioactive compounds has grown significantly which in turn leads to the advancement in the research field to determine the qualities and potential need of these substances, as well as the development of advanced technologies.

Encapsulation is one of the recent technologies, that safeguards the food constituents or active components from different conditions of processing and distribution. This is achieved by enclosing them within the carrier molecules enabling their targeted release and increased absorption rate under controlled conditions (Hasanvand *et al*., 2015) [40].

The internal core ingredients are protected from external factors by using numerous carrier molecules. The major carrier molecules include sucrose, gum Arabic, xanthan gum, gum acacia, shellac, pectin, starch, maltodextrin, pullulan, galactomannan, whey protein isolate, whey protein concentrate, sodium caseinate, chitosan, sodium alginate, carboxymethylcellulose and zein (Ali *et al*., 2021) [2]. These shell materials perform an essential function in retaining functional ingredients within the protective matrix formed during processes, facilitating the prolonged release of bioactive compounds for a prolonged time (Mirmazloum *et al*., 2021) [58].

Additionally, turning the hard-to-handle bioactive compounds into user-friendly form can streamline the different processing conditions and lower the cost of production by enabling batch processing in place of continuous processing using less expensive types of equipment. Moreover, encapsulation technologies prolong the storage stability of the active component and help delicate and fragile materials withstand the stresses of processing and packaging (Arenas-Jal *et al*., 2020) [7].

2. Encapsulation
Veiga *et al*., (2019) [92] reported that encapsulation is an advanced approach in which the functional ingredient is entrapped into a matrix of carrier material. The matrix material used to entrap the core material is known as coating material. It is otherwise known as the external phase, encapsulant, carrier, shell, or wall material.

The material over which the outer protective coating is applied is referred to as the core material internal phase, or filling material.

2.1 Justification for Encapsulation

The major reasons for the choice of encapsulation/ entrapment of functional ingredients are listed below

- Protecting the internal active ingredients from the external environment factors.
- Securing the active ingredient from spoilage and reducing the loss of highly sensitive bioactive compounds.
- Enhancing the marketing strategy of the ultimate product.
- Securing the environment from toxic and hazardous products and thus maintaining safety during handling and distribution.
- The conversion of liquids and semi-liquid products into user-friendly low-moisture powders reduces the bulk of the product and easily enables transportation and storage.
- Encapsulated powder forms will ease the transportation and handling of liquid products.
- The undesired characteristics of the active components like bitter taste and off odours are concealed.
- Enhance the processing and physical properties of the finished products. (Desai & Park, 2005) [28].

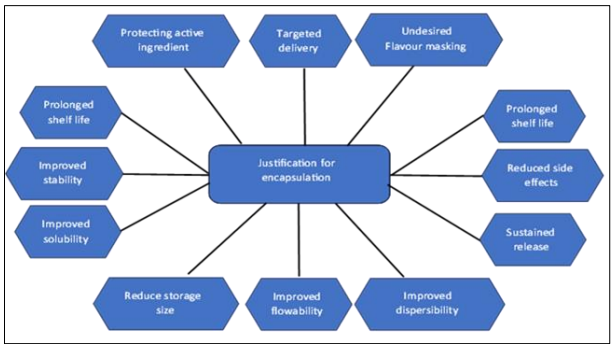

Fig 1: Justification for encapsulation [Adapted: (Zarrabi *et al*., 2020) [100]

2.2 Major steps in encapsulation

1. Creating an outer covering over the internal active ingredient.
2. Keeping the integrity of the core material inside the wall material to prevent its release at undesired time and proportion.
3. Targeted release of the core material at a suitable time with a suitable proportion (Mishra, 2015) [59].

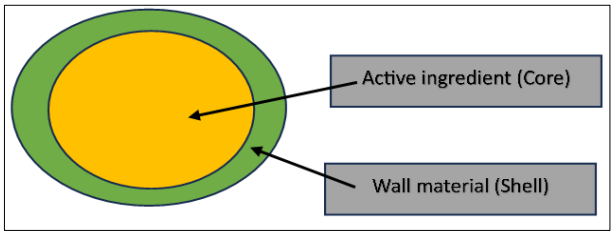

Fig 2: Simple schematic representation of encapsulation [Adapted:(Potdar *et al*., 2020) [75]]

2.3 Core material

Fang, (2010) [33] emphasized that the core material is a highly sensitive compound that needs to be protected. The core material may be assorted as liquid core or solid core in a single or mixed form. The core substances mainly include flavouring agents, colour stabilizers, vitamins, minerals, antioxidants, spice oils and oleoresins, perfumes and oils, thickeners, low-calorie sweeteners, and nutrients.

Oleoresins are extractives of spices which contain aromatic and pungent principles in concentrated form (Clemente *et al*., 2016; Vasconcelos *et al*., 2018) [25, 91]. The properties of the coating substance and the oleoresins significantly affect the efficiency of the process, storage stability, yield, and the controlled release of oleoresin. These interactions improved the retention and targeted delivery of these compounds (Arshad *et al*., 2020) [8].

2.4 Wall material

The wall material is otherwise known as a shell which acts as a protective matrix over the core material from undesirable environmental factors and enhances the physico-chemical properties, helps in undesirable flavour masking and controlled release which improves the absorption efficiency of functionally active ingredients (Choudhury *et al*., 2021) [23].

According to Nedovic *et al*., (2011) [64] the carrier material can be utilized singly or collectively to attain the desired characteristics. The composition, selection of processing technology and cost of operation are the important influencing factors for the developed encapsulated product.

The desired properties of the wall material include neutral odour and colour, good film forming, low viscosity, solubility, stability, food grade, biodegradability, and barrier properties (Cárdenas-Bailón *et al*., 2013) [17].

The materials utilized for encapsulation in the food industry applications must provide maximum protection of the functional ingredient from external factors like oxygen, light, moisture, heat etc. to keep the active ingredient intact inside the carrier molecules during various manufacturing circumstances and not interact with the encapsulated material to produce toxic compounds(Eratte *et al*., 2018) [32]. The eventual purpose was to increase the acceptability and keep the quality of the final product(Weinbreck *et al*., 2010) [95].

The widely used carrier materials for encapsulation in food-based applications are polysaccharides which include starch, cellulose (Sodeinde *et al*., 2021) [80], amylose, amylopectin, maltodextrins, gum, carrageenan, alginates pectin, chitosan etc (Koksal *et al*., 2021) [50]. In addition to carbohydrate sources, protein and lipid-based wall materials were also used for this purpose. Examples of protein sources include whey proteins like casein, whey protein isolates and whey protein concentrates, gelatine (partially hydrolysed collagen), gluten (wheat protein), zein (maize protein) etc. Lipid-based wall materials include esters of fatty acids and glycerol, waxes like beeswax, carnauba wax and candelilla wax, mono and diglycerides of fatty acids and phospholipids etc (Wandrey *et al*., 2009, Zabot *et al*., 2022, Zuidam & Nedovic., 2010 [94, 99, 102].

Table 1: Wall materials utilized for encapsulation of bioactives

Material Group	Types of material	Techniques used
Proteins	Milk proteins - Sodium caseinate, whey protein, casein Gelatine, Soy proteins, Wheat proteins, Egg proteins, zein, hydrolysed proteins	Spray drying, extrusion, coacervation, freeze drying emulsification
Carbohydrates	Sugars - fructose, galactose, glucose, maltose, sucrose, oligosaccharide, corn syrup solids, dried glucose syrup Gums - agar, alginates, carrageenan, gum Arabic, pectin, carboxymethyl cellulose, ethyl cellulose, methylcellulose, chitosan Starch and starch products - maltodextrins, cyclodextrins, starches, resistant starch, modified starches	Spray drying, fluidized bed coating, lyophilization, coacervation, extrusion, emulsification
Lipids	Natural fats and oils, mono and diglycerides of fatty acids, beeswax, carnauba wax, phospholipids, glycolipids.	Fluidized bed coating, extrusion, spray chilling or cooling

Source: Desai & Park, 2005, Augustin *et al*., (2008) and Singh *et al*., (2010) [28, 9, 79]

2.5 Encapsulation technologies

Several encapsulation methods have been developed over the years. However, the possibility of appropriate technology for the protection of targeted active ingredients depends upon the properties of the core material, the characteristics of the shell material and the necessity of the finished product for expected needs(Dubey *et al*., 2009) [30]. The subsequent section provides information regarding frequently employed encapsulation methods.

2.5.1 Spray drying

Spray drying is an important technique for the encapsulation of active compounds as it uses a continuous mode of production (Wu *et al*., 2014) [97]. It consists of atomization of liquid raw material into fine droplets, removal of moisture from the fine mist of droplets using heated air and collection of the dehydrated product(Abd El Kader & Abu Hashish, 2020) [1].

Microencapsulation by spray-drying is predominantly utilized for the protection of oils, flavours, and aromatic compounds. The process is an adaptable cost-effective technology, which uses different microencapsulation matrices and produces particles with better attributes. Most of the encapsulates are made by the process of spray drying (Yang *et al*., 2020) [98].

The active ingredient to be encapsulated is mixed with the carrier molecules and homogenized to create a stable emulsion. The stable mixture is then fed into the drying chamber of the spray dryer where it is atomized into fine droplets. The heating medium comes in contact with the atomized droplets and makes the wall material entrap the core material by removing the moisture which leaves the fine free flowing particles(Mishra, 2015) [59].

The desirable characteristics of the carrier material used for spray drying are as follows; it should produce a stable emulsion, be an effective emulsifier, show appropriate dissolving characteristics, be able to build networks, and form low viscosity solutions at higher concentrations. gum Arabic, maltodextrin, and sodium caseinate are the most used carrier matrices for spray drying (Hogan *et al*., 2001) [41]. Spray drying is a potential method used mainly for the encapsulation of milk and milk products. The core material is blended with the carrier material usually at a ratio of 1:4 (Gibbs *et al*., 1999) [38].

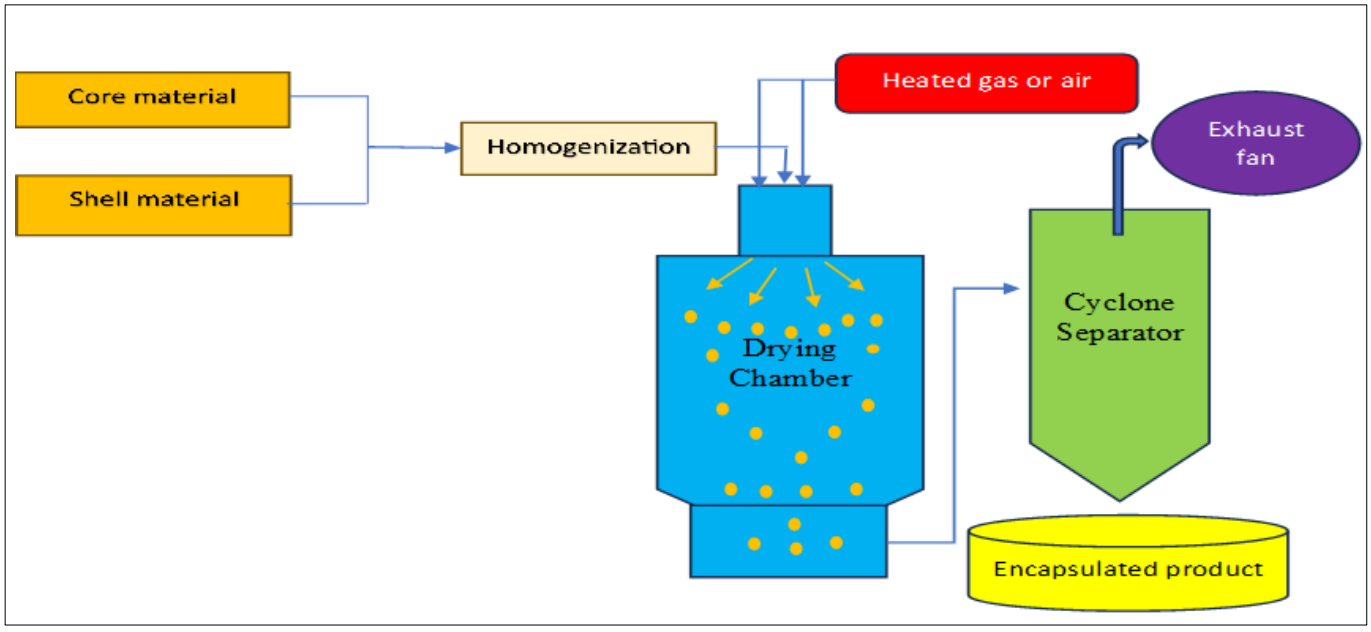

Fig 3: Schematic representation of Spray drying [Adapted: (Mohammed *et al*., 2020) [60].

2.5.1.1 Basic procedures involved in spray drying

1. Liquid feed atomized into fine droplets.
2. Atomized fine droplets mixed with the heating medium (heated gas or air) which ensures the evaporation of the liquids and leaving the dried particles.
3. Separation of dried particles from the heating medium and were collected in a suitable container (Mudit *et al*., 2010) [61].

Delfiya *et al*., (2014) [27] carried out a study on the encapsulation of turmeric oleoresin by spray drying using gum Arabic and maltodextrin as carrier materials. Maximum oleoresin retention and maximum curcumin content recovery were obtained for solvent-extracted industrial oleoresin by using gum Arabic as wall material and with an inlet air temperature of 175 ºC. Encapsulated oleoresin is more stable against surrounding factors than non-encapsulated oleoresin.

Taylor *et al*., (2007) [88] observed that cinnamon bark is one of the spices which is used exclusively for seasoning purposes. The deep reddish-brown oleoresin of cinnamon bark contains cinnamaldehyde as its main active ingredient. But it is highly sensitive to oxygen and light. Microencapsulation ensures the safe entrapment of this active ingredient. A study was conducted by utilizing encapsulation of cinnamon oleoresin using binary and tertiary blends of wall materials like gum Arabic, maltodextrin, and modified starch by spray drying. The results revealed that a 4:1:1 blend of gum Arabic: maltodextrin: modified starch provided improved safeguarding than gum Arabic alone.

Shaikh *et al*., (2006) [84] emphasised the microencapsulation by spray drying black pepper oleoresin using gum Arabic and modified starch as shell materials. The stability of aromatic and pungent principles, and the total and encapsulated piperine content were analysed for one and a half months. The study concluded that gum Arabic provided enhanced protection for pepper oleoresin than modified starch after the storage period was completed.

Chatterjee and Bhattacharjee, (2013) [19] examined the microencapsulation of eugenol-rich clove extract in maltodextrin and gum Arabic through a spray drying technique. The study aimed to investigate the antioxidant properties of these microcapsules in soybean oil. It was found that maximum encapsulation efficiency was obtained when a composite of clove extract, maltodextrin and gum Arabic was utilized. These results emphasized the potential use of microencapsulated essential oils in food processing sectors.

Ahad *et al*., (2021) [3] optimized the concentration of wall material and processing conditions of spray-dried ginger oleoresin powder. The investigation identified the optimal conditions for ginger oleoresin powder production as follows: a mixture of gum acacia and whey protein isolate at a concentration of 30%, gingerol concentration ranging between 23% and 25%, an inlet temperature of 170°C, and a feed flow rate of 350 mL/h.

Najafi and Kadkhodaee, (2011) [63] compared the sustainability of encapsulated cardamom oil, by using spray drying and freeze drying. The powders were retained under varying conditions of relative humidity and temperature. The results showed that spray drying was a better method than freeze drying to encapsulate active ingredients of cardamom oil. Release of 1, 8-cineole at different temperatures was also observed and found that the release is higher at 50 °C.

Kausadikar *et al*., (2015) [48] investigated the microencapsulation of lemon oil using binary and tertiary blends of gum Arabic, modified starch, and maltodextrin by utilizing the process of spray drying. The results showed that the combination of gum Arabic: maltodextrin at equal concentration provided the best quality encapsulated product.

According to the studies carried out by Ahad *et al*., (2021) [3] whey protein isolate emerged as the superior choice among carrier agents for producing ginger oleoresin powder, exhibiting advantages like improved rehydration ratio, an increase in yield and a decrease in hygroscopic nature. The optimized conditions for spray drying the ginger oleoresin powder involved maintaining a 30% concentration of carrier material (both GA and WPI), an inlet air temperature of 170 °C, and a feed flow rate of 350 ml/hr, all aimed at achieving the significant characteristics of the finished product.

Ostroschi *et al*., (2018) [67] demonstrated the successful application of the spray-drying process for creating an active ingredient containing proanthocyanidin-rich cinnamon extract encapsulated using maltodextrin as wall material. This formulation exhibits characteristics such as low hygroscopicity, excellent solubility, and high stability, resulting in an extended shelf-life. Additionally, the use of maltodextrin in formulating the spray-dried microparticles helps reduce the undesirable flavour characteristics of cinnamon extract. This improvement could enhance consumer acceptance, potentially increasing the utilization of antioxidants obtained from cinnamon.

2.5.2 Spray chilling and spray cooling

According to Oxley, (2012) [68] in the process of spray chilling a uniform mixture of feed and the carrier molecules are fed into the cooling chamber in an atomized form. The tiny mist of the slurry is carried into the cooling chamber and cooled into fine particles. The particles settled down by gravity in the cyclone separator and were collected from the bottom. Some of the particles are separated using filter bags and fine powders and collected from the bottom of the chamber.

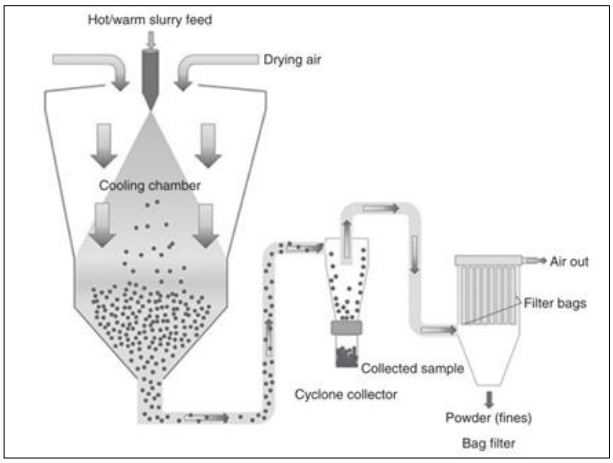

Fig 4: Schematic representation of Spray chilling process [Source: (Oxley, 2012) [68]

The major difference between spray chilling and spray cooling is the temperature of the reactors. A semisolid mass as the wall material, covering the core materials are spray cooled and the suitable wall materials for the purpose are vegetable oils and mono- and diacylglycerols (Desai & Park, 2012) [28].

Spray-chilling and spray-cooling are methods utilized for the creation of lipid-coated active substances. The primary difference between these two approaches lies in the melting point of the lipids used for the purpose. In spray chilling, this point typically falls within the range of 34-42 °C, whereas in spray cooling, the temperature is higher. The

active agent can be incorporated into lipids, and exist as dry particles or as an aqueous emulsion. Spray cooling can be conducted either in batch or continuous mode (Mishra, 2015) [59].

The encapsulated products made by these two techniques are insoluble in water due to the presence of a lipid-based outer coating. Recent research indicates that spray-chilling encapsulation can help preserve the stability of vitamin C, cholecalciferol, and proanthocyanidins under different keeping conditions (Carvalho *et al*., 2019; Paucar *et al*., 2016; Tulini *et al*., 2017) [45, 69, 89].

Spray cooling or chilling is a physical process with safety, high speed, and reproducibility. Along with that, it is an environment-friendly process when compared with the spray drying technique. This process can be operated in a continuous mode which helps to ease the operation conditions (Balanc *et al*., 2014) [11].

A recent study conducted by Procopio *et al*., (2022) [76] viewed the combined antifungal impact of cinnamon and paprika oleoresins, examining their co-encapsulation through the process of spray chilling. The microparticles obtained by this technology have improved the entrapment efficiency of volatile compounds because of their increased solubility in lipid materials.

Tulini *et al*., (2017) [89] assessed the release pattern and durability of a cinnamon extract rich in proanthocyanidins and α-tocopherol are co-encapsulated together using the spray chilling encapsulation technique. Additionally, the research investigated the combined antioxidant effectiveness of proanthocyanidins and α-tocopherol.

Boesso *et al*., (2016) [19] observed the creation and analysis of solid lipid microparticles containing ginger oleoresin by utilizing the spray chilling technique. Carriers comprised of palm fat or palmitic acid with oleic acid. The aromatic and pungent principles are retained efficiently and the results indicated that spray chilling holds great promise as a technique for producing solid lipid microparticles loaded with ginger oleoresin.

2.5.3 Coacervation

Augustin & Sanguansri, (2008) [9] pointed out that a coacervate is a tiny round droplet formed from organic molecules, held together by hydrophobic forces, with sizes typically falling between 1 to 100 µm. The name is derived from Latin which means “to assemble or cluster.” Coacervation techniques are classified into simple and complex coacervation. In simple coacervation, a desolvation agent is used for phase separation. But in complex coacervation, oppositely charged polymers combined to form a complex.

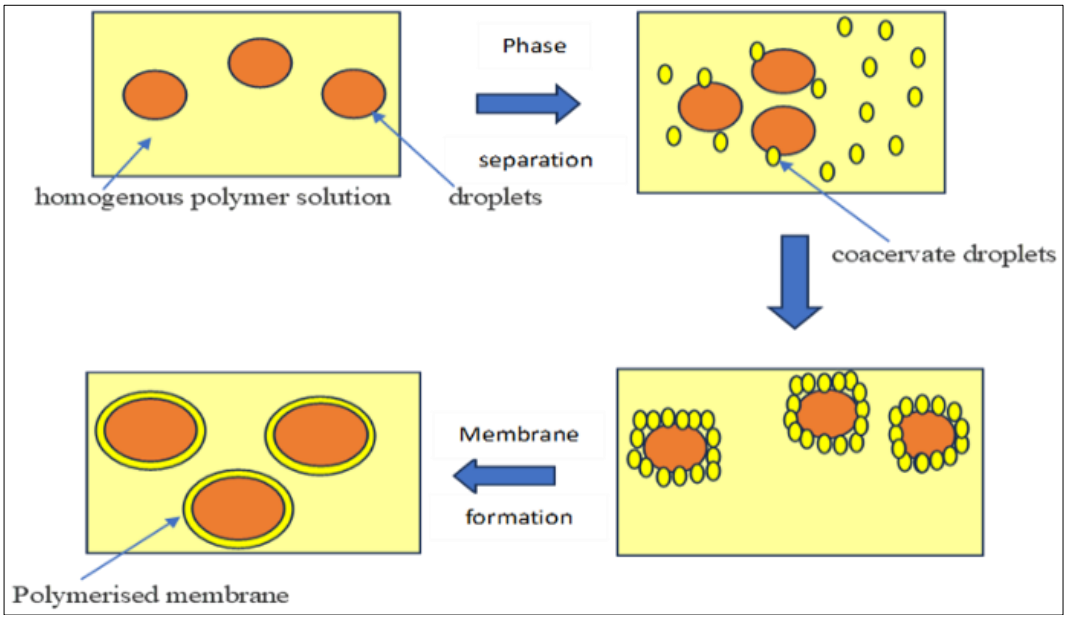

Fig 5: Diagrammatic representation of Coacervation [Adapted: (Choudhury *et al*., 2021) [23]

According to Konuklu *et al*., (2014) [51] a single type of polymer is needed for the process of simple coacervation while two or more polymers are required for the process of complex coacervation. The encapsulation of paraffin using formaldehyde and urea indicates a basic coacervation while employing gelatine and Arabic gum for paraffin encapsulation exemplifies complex coacervation.

In this technology, a chemical complex made of carbohydrates or protein is formed around the bioactive compound to be protected. This formation occurs under controlled conditions of pH, temperature, ionic strength, homogenization, the molecular weight and concentration of the polymers (Zabot *et al*., 2022) [99].

Tavares and Noreña (2020) [87] assessed the ginger essential oil encapsulation by the complex coacervation method. The wall material combinations which were selected are the binary combination of whey protein isolate with gum Arabic and gum Arabic with chitosan. The results showed that the binary combination of gum Arabic chitosan complex as wall materials provided the best result for the successful application in ginger essential oil encapsulation.

Brito *et al*., (2020) [16] conducted a study by encapsulating Ceylon cinnamon extract through complex coacervation encapsulation technology using gelatine along with five polysaccharides as wall materials. The study involved assessing particle morphology and examining the phenolic compound stability and proanthocyanidins over time. Encapsulation utilizing gelatine/κ-carrageenan and gelatine/cashew tree gum as wall materials led to improved retention of total phenolic contents and proanthocyanidins throughout the storage study. Organoleptic evaluation results indicated that encapsulation with gelatine/gum

Arabic and gelatine/κ-carrageenan masked the bitter taste of the extract. These findings confirmed that complex coacervation microencapsulation serves as the best alternative to enhance the stability of cinnamon extract for its application in foods.

Pinto *et al*., (2020) [73] described the structural properties and gastrointestinal nature of black pepper essential oils using complex coacervation encapsulation using sodium alginate and lactoferrin as carrier materials. The effect of cross-linking agent transglutaminase was also studied. The observations revealed that black pepper essential oil is retained intact within the shell matrix using polymers and cross-linking agents. So, this technique assured the sustained release and protection of heat-sensitive active ingredients.

Complex coacervation encapsulation of some of the spice essential oils like Sichuan pepper essential oil, star anise essential oil and ginger essential oil using plant-based carrier material like quinoa protein isolate-gum Arabic was developed to evaluate the improvement in encapsulation efficiency and the results indicated that this encapsulation system having excellent absorption of essential oils (Chen *et al*., 2023) [21].

2.5.4 Freeze drying/lyophilisation

Freeze drying is ultimately a drying method in which the freezing of solvent occurs initially followed by the sublimation from the solid state directly into the gaseous stage without passing through the liquid stage (Oetjen & Haseley, 2004) [66]. There are three major steps involved in the process of freeze-drying which includes freezing, primary drying and secondary drying. Initially, the diluted core materials are cooled to −20 °C and −40 °C before drying.

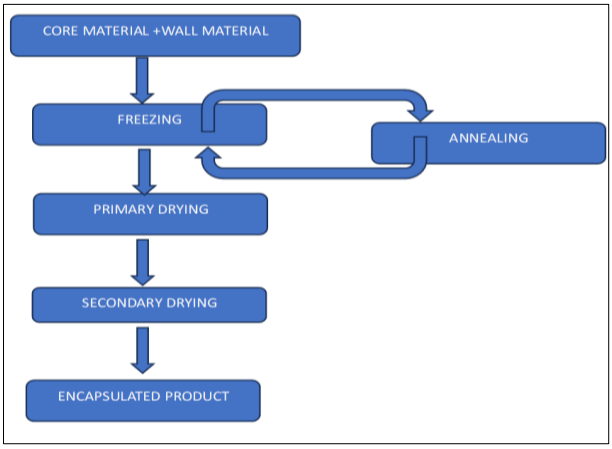

Fig 6: Schematic representation of steps in freeze drying [Adapted: (Gaidhani *et al*., 2015) [36]]

According to Gaidhani *et al*., (2015) [36] lyophilization preserves the heat-sensitive compounds for a long time because it is conducted at vacuum conditions with low temperatures. The formation and size of ice crystals are determined by the freezing rate. The slow rate of freezing produces large ice crystals which sublime simply and raise the rate of primary drying. In primary drying, a vacuum is used to raise the temperatures for the initiation of sublimation. Product temperature maintained 2–3 °C below at which the product may lose its characteristic nature.

Chumroenphat *et al*., (2021) [24] carried out studies on the alterations in chemical nature and curcuminoid content of turmeric using various drying techniques including freeze drying. The results revealed the potential effect of freeze drying on safeguarding the bioactive components and antioxidant characteristics while comparing with alternate low-temperature drying techniques.

Jan *et al*., (2022) [44] conducted an experiment on the efficiency of freeze drying on the active constituents of ginger and licorice extracts and the results concluded that freeze drying results in a potential increase in antioxidant activity. There is an effective rise in total phenolic and flavonoid content. The nutraceutical properties of these extracts along with their unique flavour provide their potential usage in value-added products.

Ion *et al*., (2020) [43] reported that freeze-drying is a sustainable encapsulation method for the preservation of phenolic compounds and antioxidant activity according to the observations obtained from the studies on the volatile oil of common basil.

2.5.5 Fluidised bed coating

Guignon *et al*., (2002) [39] pointed out that fluidised bed coating is a batch or continuous type coating technique for the protection of core materials. The air stream fluidises the powder particles at a specific temperature and then applies a fine mist of coating material over them. Wall materials such as starch derivatives or cellulose, gums or proteins dissolved in water are mostly used for this process Dewettinck & Huyghebaert, (1999) [29].

Shahidi *et al*., (2020) [83] reported that fluidised bed coating/agglomeration is considered a potential method for energy and time saving because elevated temperature is not needed for this process. The primarily encapsulated product is coated secondarily by this approach to improve stability. For example, a secondary coating of corn starch may be applied to fish oil encapsulated with caseinate to improve its stability.

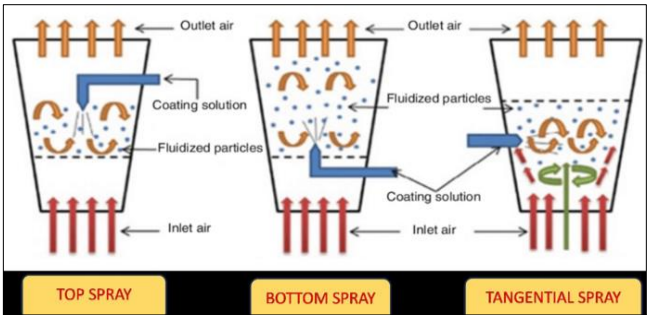

Fig 7: Schematic diagram to show top, bottom, and tangential-spray fluidized-bed coating [Source: (Bakry *et al*., 2016) [10]]

The moisture evaporation is controlled by the parameters like the rate of flow of air, the spraying level, the inlet air the temperature, humidity and moisture content of the coating solution and the characteristics of the material in the chamber(Guignon *et al*., 2002 [39]).

According to Szafran, (2013) [86] fluidized bed coating is used in the encapsulation of nutritional substances like Ascorbic acid, B complex vitamins, sodium ascorbate, ferrous fumarate, potassium chloride, and a variety of minerals, leavening agents, acetic and lactic and sorbic acid, sorbates of potassium salts, propionates of calcium salts etc.

Benelli & Oliveira, (2019) [14] examined the efficiency of the fluidization encapsulation method for coating cellulose using a lipid-based system loaded with the extracts of

rosemary. The potential effect of the intensity of wall material and characteristics of the processing parameters on the working of fluidization coating and product properties were identified and found to be satisfactory.

The drying kinetics, quality and process standardization of essential oil of nutmeg mace enriched extracts encapsulated by microwave-assisted fluidized bed drying was demonstrated by Srinivas *et al*., 2020 [82] and the results revealed that there is no reduction in the active compound of essential oil when compared with conventional drying techniques.

2.5.6 Liposome entrapment

Lipid-oriented encapsulation techniques are one of the most accepted methods to entrap active ingredients containing hydrophobic and hydrophilic molecules. It enhances the organoleptic properties and storage stability of the food product and acts as an efficient method to entrap the active ingredients. This technique increases the solubility and sustained release of functional ingredients which in turn permits targeted release of the encapsulated material(Subramani & Ganapathyswamy, 2020) [85].

Liposomes are artificial microscopic vesicles which are derived from phospholipids and attached to water molecules making them amphiphilic containing hydrophilic and hydrophobic substances with different solubility, providing several applications in food and nutritional sectors. (Khorasani *et al*., 2018) [49].

The highly sensitive water-soluble and oil-soluble bio actives are entrapped intact inside the liposomes before their release and mainly depend on the physico-chemical characteristics and efficiency of the liposomes. (Zeisig and Cammerer 2001) [101].

By considering the lamellarity liposomes can be classified as unilamellar vesicles (ULV), multilamellar vesicles (MLV) and oligolamellar vesicles (OLV). Vesicles can be further classified according to their size as small unilamellar vesicles (SUV) giant unilamellar vesicles (GUV), and large unilamellar vesicles (LUV). The size of the particles ranges from 30 nm to a few microns (Fathi *et al*., 2012) [34].

The application of functional ingredients from plant and food materials can be increased by liposomal encapsulation (Karimi *et al*., 2019) [47]. The advantages of this encapsulation technique are enhanced protection, controlled release, intensified bioavailability, and the increased bioavailability of the active ingredients thereby expanding its application in the food industry (Singh *et al*., 2012) [79].

Lin *et al*., (2016) [53] investigated the antibacterial effect of nutmeg oil encapsulated in liposome and the results indicated that the encapsulation using liposome entrapment method preserved the bioactive principles of nutmeg oil, and can be used as a potential encapsulation technology to increase the retention of these sensitive compounds when they are applied in the food processing sectors.

The studies carried out by Pentak (2016) [71] indicated that the piperine loaded in liposomes showed higher stability when compared to free piperine when subjected to higher temperatures. During incubation 37 °C also showed greater stability of liposomal piperine than free piperine.

The liposomal curcumin was subjected to incubation at a temperature of 80 °C for one hour, and the results revealed that the retention of curcumin increased significantly with the inclusion of lecithin Peng *et al*., (2018) [70].

The storage life of bioactive cinnamaldehyde was carried out and the results showed improved keeping quality due to liposomal entrapment (Chen *et al*., 2019) [20]. The liposomal piperine extract has exhibited improved storage stability when the product is stored at 4 °C in dark conditions (Dutta and Bhattacharjee, 2017) [31].

The liposomal ethanolic ginger extract showed elevated antioxidant property than non-encapsulated extract using different antioxidant capacity assays (Ganji and Alangi, 2017) [37]. The liposomal entrapment of extracts of turmeric showed an improved level of antioxidant properties upon storage (Karimi *et al*., 2019) [47].

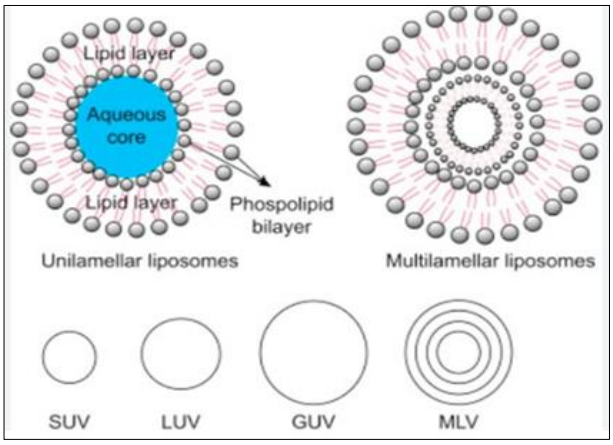

Fig 8: Schematic diagram of liposomes [Source: (Nag alingam, 2017) [62].

2.5.7 Extrusion coating

Extrusion-based encapsulation is a simple coating method which was used to produce concentrated products. It is a co-extrusion technique in which the feed and carrier materials are pumped through separate openings which is located on the outer edge of a revolving cylinder. The extrusion coating occurs commonly in a double-walled tube in which the core material flows through the inner tube and the carrier material flows through the outer tube. The equipment is connected to a revolving shaft in which the core and shell matrix are co-extruded through the concentric aperture. During this process, the rod is driven outward due to centrifugal force, which breaks into fine particles. The special characteristics of the coating material move it in a faster way over the outer surface of the core material to obtain a completely entrapped core material. (Timilsena *et al*., 2020) [90].

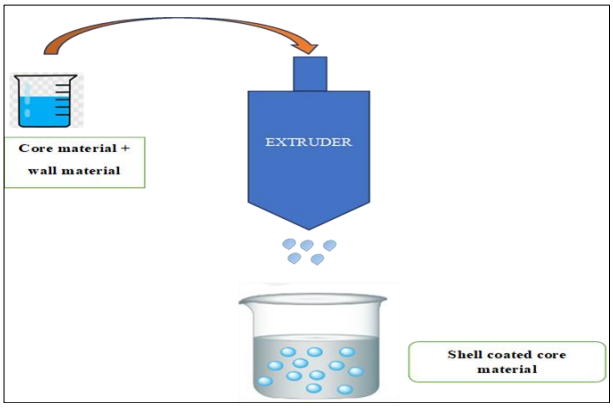

Fig 9: Schematic representation of extrusion encapsulation [Adapted: (How *et al*., 2022) [42].

A coating system of encapsulation is accepted to encapsulate products like flavourings, seasonings, vitamins, minerals etc. In this method, carbohydrates like glucose, fructose and sugar alcohols are used as wall material. The encapsulated product obtained from extrusion coating is of larger particle sizes when compared with the products prepared by other entrapment techniques (Nedović *et al*., 2011) [64].

Carrier materials like sodium alginate, starches, gelatine, cellulose, gum Acacia, carrageenan, fatty acids, and waxes are commonly used for the entrapment in extrusion encapsulation (Wandrey *et al*., 2009) [94]. Extrusion technology was used as a mode for encapsulation of sensitive compounds and ensures increased storage stability of the bioactive compounds (Bamidele & Emmambux, 2021) [12].

The utilization of extrusion encapsulation technology has proven its effectiveness in encapsulating bioactive compounds aiming to reduce various issues and enhance their application in the food processing sectors. The extrusion technology has contributed to an increased capacity for encapsulating functional ingredients facilitating particle size reduction of core materials and thereby enabling broader utilization in the food and allied fields. (Manzoor *et al*., 2022) [54].

2.5.8 Emulsification

Emulsification encapsulation is known as emulsion polymerisation where it can be used as an encapsulation process for both water soluble and lipid soluble active ingredients. In emulsification, the surface-active agent is dissolved in water until the needed concentration is acquired. Polymerization occurs within the interior part of the micelle. As the polymerization proceeds, the enlargement of nuclei occurs gradually which in turn entraps the core material to form the final encapsulated product (Appelqvist *et al*., 2016) [6].

The most common type of emulsion is an oil-in-water emulsion, in which the oil droplets are emulsified in a continuous phase of water. Water-soluble active ingredients are encapsulated in water-in-oil emulsions or a water-in-oil-in-water type emulsion. Similarly, lipophilic active ingredients are encapsulated using oil in water emulsions. The prepared emulsion may be converted to user-friendly powders by the process of spray drying or freeze drying to provide a convenient encapsulated product (McClements *et al*., 2007) [57].

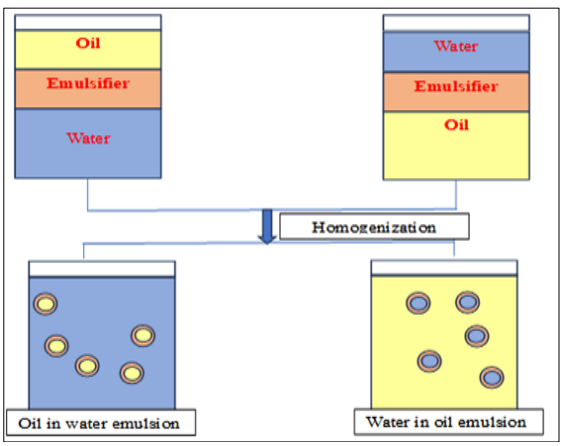

Fig 10: Schematic representation of types of emulsions [Adapted: (McClements *et al*., 2007) [57]

The emulsification process includes the formation of a suspension of oil-soluble or water-soluble active ingredients, before the addition of a carrier material and was homogenized. After homogenization, numerous tiny droplets of the core material were dispersed throughout the system. These fine droplets are covered by an emulsifier through different interactions(Choudhury *et al*., 2021) [23].

The use of mixtures of wall materials like gum Arabic, whey protein isolates or maltodextrin showed higher encapsulation efficiency due to synergistic effects in stabilizing emulsions than individual wall materials. (Anthero *et al*., 2020; Noghabi, & Molaveisi, 2019; Comunian *et al*., 2019) [5, 65, 26].

Mariana Costa *et al*., (2021) [55] carried out a study to investigate the combined impact of cinnamon and paprika oleoresins mixtures and encapsulating them into emulsions using gum Arabic, whey protein isolate, or maltodextrin as wall materials. The results revealed that the sample contained maltodextrin: whey protein isolate at a ratio of 1:3 as the wall material exhibited elevated antioxidant properties. These findings concluded that the emulsions provide a potential effect on the active compound recovery and ensured their targeted delivery in various food industry applications.

2.5.9 Co-crystallization

Co-crystallization is an encapsulation technology in which sucrose was utilized as a wall material. The sucrose syrup is heated up to the supersaturation temperature and the active ingredient in emulsified form is added rapidly before the beginning of crystallization. As the syrup reaches the temperature which is required for transition and crystallization, a significant rate of heat is generated. Continuous agitation is required to increase the crystallization process. The product which is obtained can be dried and moulded to the desired shape if needed (Chezanoglou and Goula, 2021) [22].

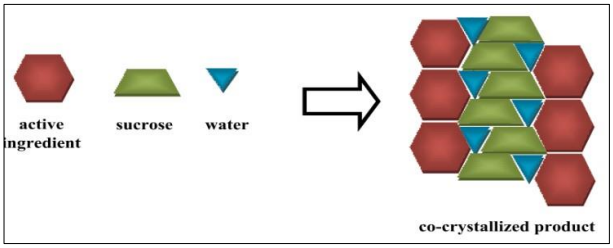

Fig 11: Schematic representation of Co-crystallization mechanism [(Source: Chezanoglou & Goula, 2021) [22]

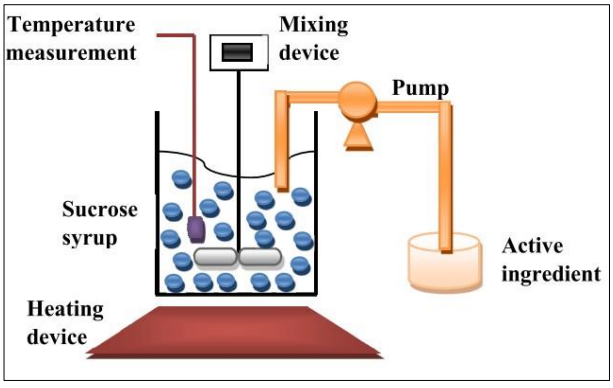

Fig 12: Schematic representation of Co-crystallization Equipment [(Source: Chezanoglou & Goula, 2021) [22]]

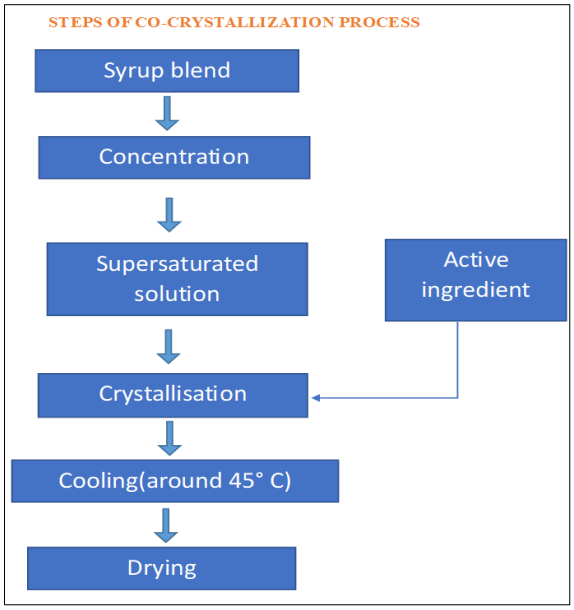

Fig 13: Steps in Co-crystallization process [Adapted: (Phanindra *et al*., 2018) [72]]

Phanindra *et al*., (2018) [72] reported that co-crystallization involves an encapsulation procedure where the typical crystalline arrangement of sucrose is transformed from a uniform structure to an uneven one, creating a porous matrix suitable for embedding core materials. The supersaturation temperature of sucrose syrup attained at a range of about 110 to 120 ºC.

Chezanoglou and Goula, (2021) [22] reviewed the co-crystallisation in sucrose, its main steps, the pros and cons of the technology and its application for encapsulation of various functional components. From that review, the authors concluded that encapsulation by co-crystallization was a cost-effective flexible method to protect the active ingredients from environmental factors.

Sardar and Singhal, (2013) [78] prepared spice-flavoured sugar cubes by using emulsified cardamom oleoresin by co-crystallization encapsulation. Products are prepared at different relative humidity with and without the addition of cardamom oleoresin. The gas chromatography separation technique was utilized for assessing the active components of cardamom oleoresin.

According to Federzoni *et al*., (2019) [35] co-crystallized paprika oleoresin is used to prepare an organic water-soluble dye which is suitable for food applications. The colour retention ability of the co-crystallized and free oleoresins was studied and found that the product prepared from the co-crystallization process was found to be best for the preparation of co-crystallized product and also provides proper entrapment of paprika oleoresin intact inside the sucrose matrix.

Rai *et al*., (2021) [77] carried out a study on the encapsulation of ginger oleoresin on co-crystallised sucrose. HPLC method was used to quantify the functional ingredient. The study investigated the storage kinetics under various levels of relative humidity and temperatures and the results showed that [6]-gingerol exhibited the highest stability than other gingerols. Optimal storage conditions for ginger-flavoured sugar cubes were found to be a relative humidity of 33% and a temperature of 25°C. Additionally, the research concluded that co-crystallization for encapsulating ginger oleoresin offers protection for active ingredients and enables controlled release of flavours.

2.5.10 Ionic gelation method

Spherification is a recent technique used to create semi-solid spheres with delicate membranes formed from liquids, containing a non-gelled liquid filling. This structure facilitates a burst-in-the-mouth when the liquid is released. This process involves encapsulating a liquid within a gelatinous sphere. The flavour and texture of the products are enhanced with the spherification technique (Kulthe *et al*., 2019) [52].

There are two main types of spherification techniques which include the basic and reverse techniques. In basic spherification, sodium alginate is mixed directly with the material to be encapsulated and dropped into a bath of calcium chloride, calcium lactate or calcium gluconate to form a thin semi-solid sphere. The reverse or external technique is carried out by mixing the calcium source with the material to be encapsulated and dropped into a bath of sodium alginate solution. (Ahirrao *et al*., 2014) [4].

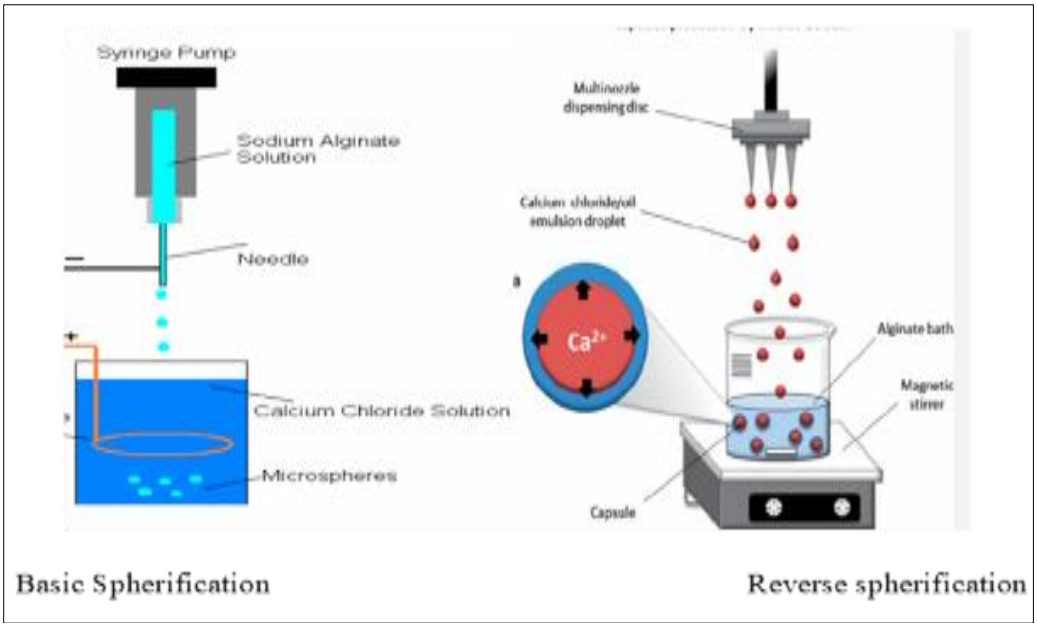

Fig 14: Schematic representation of the Ionic Gelation Process [(Source: Charron *et al*., 2005, Martins *et al*., 2015) [18, 56].

Patil *et al*., (2012) [74] pointed out that alginate is a harmless, biodegradable polysaccharide found naturally in marine brown algae and specific bacterial strains. Divalent cations like calcium ions are commonly used for cross-linking of alginates. Alginate is considered the polymer of choice in pharmaceutical, food and biotechnological applications.

Basu *et al*., (2010) [13] reported that sodium alginate has many beneficial properties as outlined below: it acts as a thickening agent, binding agent, gelling agent, and stabilizing agent in food processing sectors. It is employed in the encapsulation of volatile compounds for entrapment and sustained release because of its nature of forming ionotropic gelation with calcium ions under suitable conditions. Various calcium salts like chlorides, lactates, and gluconates are used for the spherification process. Most commonly calcium chloride is used because it forms better bonds with alginate producing gelatinous calcium alginate. One of the drawbacks is the bitter taste of calcium chloride.

Song *et al*., (2012) [81] carried out a study on the curcumin-loaded alginate beads, which contain different food emulsifiers. The scanning electron microscopy (SEM) analysis offers information related to the surface and cross-sectional characteristics of alginate beads, which exhibit important variations depending on the types of emulsifiers. The results indicated that the composition of emulsifiers affected the swelling, entrapment efficiency, controlled release, and release kinetics properties of the alginate beads.

A study was conducted on the ionotropic gelation method to entrap flaxseed oil, and garlic oil individually and a combination of both flaxseed oil and garlic oil by using sodium alginate as a wall material to produce gelled spheres and the results concluded that the combination achieved maximum encapsulation efficiency due to its synergistic effect (Kairam *et al*., 2020) [46].

Wibowo *et al*., (2021) [96] studied the clove oil encapsulation by making use of Calcium alginate-gelatine semi-solid spheres. Different process parameters like the mass ratio between alginate and gelatine, and the concentration of sodium alginate and calcium chloride were varied and results indicated that the increase in concentration of all these compounds decreased the encapsulation efficiency.

3. Conclusion

In recent years health consciousnesses among the consumers become greater and food products containing functional ingredients that are healthy, safe, and easy to use are in high demand. Spice extractives are one among them which are used as substitutes for synthetic chemical additives in today's health-sentient environment. Encapsulation is a significant technique employed to safeguard hard-to-handle spice extractives and the characteristics of functional components, and it finds broad applicability across food, agricultural and pharmaceutical sectors. The primary objectives of encapsulation technologies are to enhance stability, bioavailability, sensorial attributes, retention of sensitive properties of bio actives, controlled release, reduction of hygroscopicity, masking of bitter taste and ultimately to increase the storage stability of the products. This review provides information related to the most important encapsulation technologies and some of their applications in the encapsulation of spice extractives. Further research is necessary to explore the bioavailability of functional ingredients using new encapsulation technologies. Moreover, studies at both pilot and industrial levels are necessary for scale-up purposes.

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