Encapsulation Techniques of Food Bioproduct

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Encapsulation is a technique in which the liquid droplets individual shells can be packed with solid particles by the continuous system. Shells may be called wall, the shells are designed to protect the material from factors that may cause deterioration. The wall design controls the release of encapsulation material core under the required conditions. The applications of encapsulation in the food industry have their importance. Sensitive materials such as essential oils and oleoresins protect from chemical reactions, oxidation and evaporation, by encapsulation in the dry form. Encapsulation of aromatic compounds preserve the organoleptic profile of the product until use and this can ensure the high quality and commercial value of the product. In this review article, several encapsulation techniques that are used commercially or are being applied in the food industry are discussed. This includes spray drying, spray cooling, spray chilling, fluid bed, extrusion, co-crystallization, co-acervation, molecular inclusions and liposome.

Keywords: Encapsulation, Shells, Food industry, Bio product, Encapsulation techniques.

Introduction

Consumers are more and more concerned with nutritional aspects of foods and the recent developments of functional food correspond to this reality. A better balance between the nutrients and the addition of indigestible fractions and complementation of trace element, vitamins or special components appear as a necessity in the development of novel products.

The introduction of lipophilic vitamins or other lipidic nutritive compounds like fish oil into a product create some problems that could be solved by the encapsulation of these compounds. By encapsulation protection of the active compound from oxidation is achieved. Also, transformation of the fat into a free flowing powder can mask off the undesired flavor or bad taste. Many types of foods are encapsulated like that containing flavoring agents, antioxidants agents, colorants, non-natural sweeteners, acids bases, leavening, and preservatives, with disagreeable nutrients, odors and flavors and among others.

Flavors are added to some food products as beverages, snacks, soup mixes, cake mixes...etc. to give it perfect taste profile, wherein the technical properties of the flavors have to be specially design for processing methods, application and consumer needs. The wide majority of flavor compound is used in industry as liquid at ambient temperature, for this, it is very important to utilize some process to dry these flavors chemicals.

The industry of flavor is depending on various manufacturing techniques to produce flavor solids and protect them till consumption. There are two major encapsulation industrial techniques, this comprise spray drying, and extrusion [1,2].
the other hand, minor techniques are employed including freeze drying [3], coacervation [4], wax or fat encapsulation [5], molecular inclusion in cyclodextrin [6] and cocrystallization [7].

Generally, encapsulation is defined as the surrounding of active ingredient (solid, liquid or gas) by a definite continuous coating or film (Figure 1).

Theoretically, typical diameters of microcapsules are in the range of 0.01 and 1,000 μm and the thickness of wall material is in the range of 0.5-150 μm [8].

The major reasons for using the encapsulation are as follows[9]:

- Securing the product from the surrounding conditions (temperature, moisture …etc.).
- Protecting the active ingredient against deterioration and limiting the evaporation (losses) of volatile material.
- Saving the environment from the hazards and toxic product to be more safely during its handling.
- Dry handling by conversion of liquids and sticky solids to free flowing powders.
- Masking of undesired properties of the active components, like taste and odor.

The objective of this paper is to review the encapsulation techniques of food bioproduct in a three perspectives. First, it focuses on the theoretical aspects of the process and required criteria for encapsulation agents. Next, it discusses microencapsulation of various bioactive food ingredients such as omega-3 fatty acids, polyphenols, enzymes, protein, microorganisms, vitamins and minerals and its applications. The third section summarizes controlled release mechanisms of microcapsules.

Encapsulation techniques:

Encapsulation techniques of food ingredients and the potential utilization of the technology in the food industry had been discussed in the literature by various authors [2,10-32].

The application of encapsulation processes in the food industry includes some special considerations, primarily those concerned to the solvent and to the wall material which could be generally recognized as safe (GRAS).

Various encapsulation methods are now available. A fundamental distinction can be made between two categories; True encapsulation, in which a liquid flavor core is surrounded by a shell such a gelatin capsule, however because it is fairly costly, it is only an option in a relatively limited number of applications. On the other hand, there are also methods in which numerous ultra-fine flavor droplets are enclosed in a matrix consisting of a wide variety of carriers. Encapsulated flavors possess greater stability and are better protected against such external influences as oxidation. In addition, they also provide a dry version of what are usually liquid flavors, this means that they can easily be worked into dry products. Also a specific properties for example a water solubility can be designed through the selection of encapsulation technology, a flavors release properties continue to be the key issue in selecting a given encapsulation technology.

Fig. 1. Schematic presentation of the capsule structure [8].

According to capsule size, it can be classified into:

- Microcapsules size range between 0.2 and 5,000 μm for encapsulated particles.
- Microcapsules size range larger than 5,000 μm.
- Nano capsules particle size is smaller than 0.2 μm (200 nm) [33].

Table (1) demonstrates the particle size of capsules produced by different encapsulation technologies, while Figure (2) illustrates the classification of microparticle.

### Methods of encapsulation:

The encapsulation can be produced by different methods (Figure 3) [17,35-37]:

1. Physical process: spray drying; fluid bed coating, spray chilling, freeze drying extrusion and co-crystallization.
2. Physicochemical: solvent evaporating (organic phase separation), liposome entrapment, simple or complex coacervation (liquid phase separation).

<table>
<thead>
<tr>
<th>Encapsulation Technology</th>
<th>Particle size of capsules (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spray drying</td>
<td>10 – 400</td>
</tr>
<tr>
<td>Fluid bed coating</td>
<td>5 – 5000</td>
</tr>
<tr>
<td>Spray chilling / cooling</td>
<td>20 – 200</td>
</tr>
<tr>
<td>Melt injection</td>
<td>200 – 2000</td>
</tr>
<tr>
<td>Melt extrusion</td>
<td>300 – 5000</td>
</tr>
<tr>
<td>Emulsification</td>
<td>0.2 – 5000</td>
</tr>
<tr>
<td>Emulsions with multilayers</td>
<td>0.2 – 5000</td>
</tr>
<tr>
<td>Coacervation</td>
<td>10 – 800</td>
</tr>
<tr>
<td>Microspheres produced by extrusion or dropping</td>
<td>200 – 5000</td>
</tr>
<tr>
<td>Microspheres produced by emulsification</td>
<td>10 – 1000</td>
</tr>
<tr>
<td>Co-extrusion</td>
<td>150 – 8000</td>
</tr>
<tr>
<td>Inclusion complexation</td>
<td>0.001 – 0.01</td>
</tr>
</tbody>
</table>

**Fig. 2. The classification of microparticle** [34]
Fig. 3. Schematic description of the microencapsulation of food ingredients along with some examples of core and wall materials, wall material properties, aims, and different techniques of the microencapsulation [38].

The selection of encapsulation for a detailed application is based on parameters as; mean physical and/or chemical characteristics of active agent and carrier, particle size required, application of encapsulation material, required release mechanisms, acceptable process cost and industrial manufacturing scale.

Typical core substances for food:
The core material is defined as the specific material to be coated (a biologically active substance). The composition of the core material can be varied as liquid core which can include dispersed and/or dissolved material, or solid core that can be single solid substance or mixture of active constituent. The typical core substances for food are as follow; colorants and dyes, flavors, minerals, vitamins, animal feed ingredients, deodorants, oils, perfumes, stabilizers, sweeteners, nutrients, and antioxidants.[18].

Type of capsule wall materials [9,39]:
Several materials are commercially suitable for use as flavor encapsulating agents. The most commonly utilized are selected from the following types [40].

- Carbohydrates (cyclodextrins, corn syrup solids, starch and maltodextrins).
- Proteins (gelatin, sodium caseinate, whey protein, soy protein).
- Cellulose esters and ethers (ethyl cellulose, carboxy methylcellulose, methylcellulose).
- Gums (agar, gum acacia, sodium alginate).
- Lipids (fats, waxes, paraffin and oils).

Applications:
Encapsulation has found an extensive application in the food production generally involves volatiles, flavoring materials, vitamins, mineral, essential oil oleoresins bacteria, enzyme and pigment (Figure 4.) (Tables 2 & 3).

Profit of encapsulation:
1. Enzymes and microorganisms immobilization:
   Encapsulation of microorganism was used to enhance the stability of starter cultures. Enzymes of cheeses had been encapsulated to develop ripening and facilitate labor. The enzymes encapsulated enzymes which were sheltered from high ionic strength and low pH in cheese (Figure 5) [44,45].
2. Prevents oxidation and provide protection against acids, heat, bases and UV [38,48-50].
3. Improves shelf life because of preventing dehydration, oxidation derivative reactions.
4. Masking of odors or taste.
5. Fine texture and processing with less degradation of ingredients.
6. Manages of hygroscopic.
7. Promotes dispensability and flow ability.
8. Cleans fine particles.
9. Increases solubility.
10. Handles solids as liquids.
11. Encapsulation can deliver important needed nutritious foods in a pleasant and tasty way for children which gives children the required minerals and vitamins during growth age [51].
12. Enhances characteristics and marketing concepts.
13. Microcapsules were employed firstly in the market for carbonless copy paper where the coating of encapsulated colorless ink was functional to the top paper sheet and the developer applied it on subsequent sheet. By applying pressure during writing on the sheet the capsules breaking where the broken reaction producing ink and a copy dark color.
14. The materials which encapsulated are ready to be used in the industry of textile today’s to evaluate the properties of well finishing. The important application used in incorporation of microencapsulated phase change materials which absorb and release heat in related to changes in room temperatures. The rise in temperatures resulting in phase change and material melt due to the absorbing of excess heat, and finely feels cool. Conversely, as temperatures fall, the materials releases heat as it solidifies, and feels warm.
15- Pesticides which encapsulated is facilitated to release overtime, thus allowing farmers to use the pesticides by a fewer amounts to avoid the toxicity of higher concentration (Pemsel et al., 2010 and Ahmed, et al., 2019).
16- The Control and target release of active ingredients:
   - Oral and injected formulations used in

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Fig. 4. Markets of micro encapsulation [41].

TABLE 2. Applications of different encapsulation method in food industry [42].

<table>
<thead>
<tr>
<th>Encapsulation technique</th>
<th>Encapsulated form</th>
<th>Application area</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coacervation</td>
<td>Pasta/powder/capsule</td>
<td>Chewing gum, toothpaste, baked foods</td>
</tr>
<tr>
<td>Spray drying</td>
<td>Powder</td>
<td>Confectionery, milk powder, instant desserts, food flavours, instant beverages.</td>
</tr>
<tr>
<td>Fluid bed drying</td>
<td>Powder/granule</td>
<td>Prepared dishes, confectionery</td>
</tr>
<tr>
<td>Spray cooling/chilling</td>
<td>Powder</td>
<td>Prepared dishes, ices</td>
</tr>
<tr>
<td>Extrusion</td>
<td>Powder/granule</td>
<td>Instant beverages, confectionery, teas</td>
</tr>
<tr>
<td>Molecular inclusion</td>
<td>Powder</td>
<td>Confectionery, instant drinks, extruded snack</td>
</tr>
</tbody>
</table>

Fig. 5. Lactic bacteria culture [46,47].
TABLE 3. Food ingredients needed to be encapsulated before incorporation in food systems [43].

<table>
<thead>
<tr>
<th>Food Ingredient</th>
<th>Example</th>
<th>Advantages of Encapsulation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Colorens</td>
<td>Carotenoids</td>
<td>Easy incorporation into aqueous medium</td>
</tr>
<tr>
<td></td>
<td>Flavonoids</td>
<td>Help in storage and utilization</td>
</tr>
<tr>
<td></td>
<td>Betalains</td>
<td>Impede chemical degradation</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Increase efficacy</td>
</tr>
<tr>
<td>Flavors</td>
<td>Citrus oils</td>
<td>Easy incorporation into aqueous medium</td>
</tr>
<tr>
<td></td>
<td>Natural extracts</td>
<td>Help in storage and utilization</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Impede chemical degradation</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Control release profile</td>
</tr>
<tr>
<td>Antioxidants</td>
<td>Tocopherols</td>
<td>Easy incorporation into aqueous medium</td>
</tr>
<tr>
<td></td>
<td>Carotenoids</td>
<td>Help in storage and utilization</td>
</tr>
<tr>
<td></td>
<td>Flavonoids</td>
<td>Impede chemical degradation</td>
</tr>
<tr>
<td></td>
<td>Phenolics</td>
<td>Control release profile</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Increase efficacy</td>
</tr>
<tr>
<td>Antimicrobials</td>
<td>Essential oils</td>
<td>Improve matrix compatibility</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Help in storage and utilization</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Impede chemical degradation</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Mask undesirable off-flavors</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Increase potency</td>
</tr>
<tr>
<td>Bioactive peptides</td>
<td>Milk peptides</td>
<td>Retard degradation in stomach</td>
</tr>
<tr>
<td></td>
<td>Meat peptides</td>
<td>Reduce bitterness and astringency</td>
</tr>
<tr>
<td></td>
<td>Plant peptides</td>
<td>Control release profile and bioactivity</td>
</tr>
<tr>
<td>Bioactive carbohydrates</td>
<td>Prebiotics</td>
<td>Avoid adverse ingredient interactions</td>
</tr>
<tr>
<td></td>
<td>Chitocain</td>
<td>Enhance product texture</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Control delivery in the gastrointestinal tract</td>
</tr>
<tr>
<td>Bioactive lipids</td>
<td>ω-3 fatty acids</td>
<td>Allow incorporation in aqueous medium</td>
</tr>
<tr>
<td></td>
<td>Conjgated linoleic acid</td>
<td>Improve ease of utilization</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Avoid chemical degradation (oxidation)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Controlled delivery in GIT</td>
</tr>
<tr>
<td>Vitamins</td>
<td>Vitamins A, D, E</td>
<td>Allow incorporation in aqueous medium</td>
</tr>
<tr>
<td></td>
<td>Vitamin C</td>
<td>Improve ease of utilization</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Avoid chemical degradation</td>
</tr>
<tr>
<td>Minerals</td>
<td>Iron</td>
<td>Prevent undesirable oxidative reactions</td>
</tr>
<tr>
<td></td>
<td>Calcium</td>
<td>Check precipitation</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Increase bioavailability</td>
</tr>
<tr>
<td>Probiotics</td>
<td>Lactic acid bacteria</td>
<td>Enhance viability</td>
</tr>
</tbody>
</table>

Fig. 6. Microencapsulation techniques [34].
pharmaceutical that encapsulated for slow release in time or at certain organ in the body.

- Salicylic acid may cause ulcers and bleeding of the stomach if the doses are introduced all at once. So aspirin tablets were compressed with quantities of microcapsules that will gradually release which decreasing the stomach damage risk.

17. Encapsulation allows combination of mismatched compounds.

Microencapsulation techniques:

Figure (6) represents the microencapsulation technologies, the most important techniques will be discussed in the following sections.

Physico-chemical processes:

Coacervation:

Coacervation can be divided into “simple” and “complex” coacervation [54]. This method involves the phase separation of one or many hydrocolloids from a polymeric solution layer around the core material that is suspended in the same reaction media [13,14]. In simple coacervation, only one coating material (typically pectin) is used [10]. The simple coacervation method is dependent on conditions such as pH, ionic strength, temperature, and structure of the macromolecules. For example, when the pH is adjusted to a value near the isoelectric point (pI) of gelatin at low ionic strength, the net charge of gelatin becomes balanced. The molecules unfold and settle to form microcapsules [55]. Complex coacervation is mostly dependent on pH and the concentration of polymer. This process involves the reaction between two oppositely charged polymers (protein and polysaccharide) and is called the “polymer-polymer interaction method”. Negatively charged polysaccharides (such as acacia, pectin, alginate, and carboxy methyl cellulose) interact with positively charged proteins (such as gelatin, soy protein isolate).

- Coacervation steps [56,57]:

In coacervation, the materials that used to produce the capsules (usually gelatin and gum arabic) are firstly dissolved in water–insoluble flavor then added. By altering temperature or pH, the interfacial surface between the water and flavor droplet forms a thin skin that envelops the flavor droplet. In a further step, the gelatin has to be chemically treated to cross-link and curve it further after it has been separated from the surrounding water. For various applications the resulting pasty capsules have to be gently dried in a final step. In general, the coacervation process can applied in encapsulation of flavors, fragrances, vitamins, bacteria and cells. The flow diagram of coacervation plant is shown in Figures (7) & (8).

Microencapsulation by solvent evaporation [9,59]:

Microencapsulation by solvent evaporation is considered as the best widely used method of encapsulation, the steps of the process are as follow (Figure 9):

1- The polymer is dissolved in water immiscible volatile organic solvent like chloroform or dichloromethane, into which the active principle is dissolved.

2- The resulting solution is added drop wise to
a stirring aqueous solution having a suitable stabilizer like poly vinyl alcohol to form small polymer droplets containing encapsulated material.

3- Droplets hardened to produce the corresponding polymer microcapsules. This hardening process is accomplished by the removal of the solvent from the polymer droplets either by solvent evaporation (by heat or reduced pressure), or by solvent extraction.

4- The microcapsules can then be washed and dried.

**Microencapsulation by supercritical fluids:**

Supercritical fluids are highly compressed gases that possess numerous properties of both liquids and gases (Figures 10 & 11) (Table 4). The best commonly used being nitrous oxide (N₂O) and supercritical carbon dioxide (CO₂) [61]. The steps of the microencapsulation by supercritical fluids are as follow:

1- Supercritical fluid containing core and wall material are kept at high pressure then released at atmospheric pressure through small nozzle.

2- Sudden drop in pressure causes desolations of the wall material, which is then deposited around the core (active ingredient) and forms a coating layer.

Generally, different active ingredient such as flavors, pigments pesticides, vitamins, and dyes are encapsulated using this method. Various wall materials like wax paraffin and polyethylene glycol are used for encapsulating active agent [61]. The disadvantages of process are that both core and coat material must be very soluble in supercritical fluids.

**Physical processes:**

**Spray drying:**

Several encapsulation techniques have been proposed. Amongst them spray drying is the most common technique to get flavor powders from flavor emulsion (Figure 12). Also, spray-drying
is widely used in the food and pharmaceutical industries [9,22,32,51,63-67].

Because of its several advantages including lower cost and high efficiency, spray drying is widely usable process [68].

**Spray drying steps:**

The production follows different steps includes mixing of the ingredients (preparation), homogenization of the mix to obtain a fine emulsion, atomization of the feed materials through the nozzle, drying of the sprayed droplets in hot air and collection of the dried solid particle.

- **Preparation:**
  During preparation the emulsifier and the carrier are dissolved in water. The core compound is then added slowly while stirring to ensure the formation of a coarse emulsion.

- **Homogenization:**
  The treatment reduces the size of the core droplets to produce a fine emulsion which is obtained with high pressure homogenizer by applying a high pressure or by recycling the slurry several times at lower pressure.

- **Spray drying:**
  The spray dried product is feed by a pump. The emulsion is atomized through the nozzle in fine particles which are rapidly dried in hot spray chamber (Figure 13).

![Fig. 10. Encapsulation by supercritical solutions rapid expansion [34].](image1)

![Fig. 11. Schematic representation of supercritical fluid extraction of emulsions (SFEE) [61].](image2)
TABLE 4. Examples of bio products encapsulated using the Supercritical Fluid Extraction of Emulsions (SFEE) process [61].

<table>
<thead>
<tr>
<th>Active Ingredient</th>
<th>Coating Material</th>
<th>Particle Size and Morphology</th>
</tr>
</thead>
<tbody>
<tr>
<td>β-carotene</td>
<td>Soy lecithin</td>
<td>10–500 μm Agglomerates of partially fused spheres</td>
</tr>
<tr>
<td></td>
<td>Polycaprolactone (PCL)</td>
<td>CAPA 2403D: 110–130 μm</td>
</tr>
<tr>
<td></td>
<td>(CAPA 2403D and CAPA 6100)</td>
<td>CAPA 6100: 270–650 μm Flat or sphere-like particles attached and agglomerated by long filaments of polymer</td>
</tr>
<tr>
<td>Coffee oil</td>
<td>Polyethylene Glycol (PEG)</td>
<td>78 μm Spherical shapes of various sizes to amorphous shapes</td>
</tr>
<tr>
<td>Cylindrocyclic granulovirus</td>
<td>Palm oil-based fat: 77%</td>
<td>Lecithin-based surfactant: 9% Modified titanium oxide and benzophenone derivative UV protectants: 2%</td>
</tr>
<tr>
<td></td>
<td>&lt;85 μm Almost spherical particles were obtained</td>
<td></td>
</tr>
<tr>
<td>Lavandin essential oil</td>
<td>PEG 9000</td>
<td>30–100 μm Spheres and needles</td>
</tr>
<tr>
<td></td>
<td>Soy lecithin</td>
<td>1.4–24.8 μm Dry and fine but aggregated particles</td>
</tr>
<tr>
<td>Limonene</td>
<td>Modified starch</td>
<td>60–90 μm Spherical shapes with few broken shapes smaller than the others</td>
</tr>
<tr>
<td>Omega-3 polyunsaturated fatty acids and</td>
<td>PEG 6000</td>
<td>67.26–165.81 μm Irregular spherical shapes to amorphous shapes of various sizes</td>
</tr>
<tr>
<td>astaxanthin-rich salmon oil</td>
<td>Soy lecithin and Pluronic L64®</td>
<td>0.138–0.158 μm Complete encapsulation of amorphous quercetin</td>
</tr>
</tbody>
</table>

Emulsions:
Emulsion is widely used to encapsulate of bioactive compounds in water solutions. Emulsions divided into two immiscible liquids, one of them being dispersed in the other, with droplet sizes ranging from 50 to 1,000 nm [69]. It is well known to encapsulate a high concentration of oil-soluble biologically active food supplements. The lipophilic compounds such as β-carotene, plant sterols, carotenoids, and dietary fats which carried the process of encapsulate can be delivered by oil in water emulsion (o/w), while water in oil emulsion (w/o) was used to encapsulate watersoluble food active agents such as polyphenols [18]. Nano encapsulation can be accomplished through the drying of nanoemulsions by different drying techniques example spray drying or freeze drying besides using nanoemulsions directly in liquid state. High Kinetic stability of nanoemulsions is due to their extremely droplet sizes. This type of stability, in nanoemulsions, is of maximum value for the retention of surface oil content of the product [58]. Nanoemulsions preparation needs a lot of energy input from mechanical devices as being non-equilibrium systems. Emulsification methods needs high
energy due to use of high shear stirring, high speed or high-pressure homogenizers, ultrasonicator, or microfluidise for the formation of nanoemulsions. These methods supply the available energy in the shortest time and have the most homogeneous flow to produce the smallest droplet sizes [69,70].

Flavor retaining mechanism:
Spray drying is suitable for encapsulation of flavors. The flavor compounds are evaporating faster than the water so this it is important to find suitable carriers that prevent the volatile flavor compounds from being lost during the drying process.

Essentially there are two steps in the spray drying process of flavors. After a suitable carrier has been selected, it is first dissolved in water. The liquid flavor is then added, finely distributed and placed in the spray drier in the form of a homogenous emulsion. Various techniques are used to atomize, loose emulsion in the spray drier, where it is dried through shock contact with hot air at temperatures of 180-200°C.

As a result of the shock water evaporation, the carrier substances form a fine membrane around the flavor droplet which contained in the enclosed droplet and continue to be able to permeate and evaporate. The large flavor compound molecules on the other hand, are retained and enriched, after remaining in the dryer for a period up to 30 seconds (Figure 18).

Compacting and agglomeration

The processes of compacting and agglomeration are normal ways of complementing spray drying in both processes. The object is to coarsen the product with lower porosity (strength). While agglomeration produces a loose product with high porosity (instant properties) (Figure 19).

Spray drying is customarily complemented by the process of compacting and agglomeration to coarsen the product. The spray dried flavors are compressed under high pressure into lumps and then crushed into small pieces ranging in size from 0.7 to 3.0 millimeters. This process would be advisable, for example, the intention is to utilize a grainy structure to assure that flavors will not separate in tea bags for example, and sift out of the thick- pored bags. It is useful in a combination of flavor and functional ingredients, e.g. vitamins, and to produce real fruit granules with adjustable fruit content.

In the case of agglomeration, a spray dried powdered flavor is swirled in hot air. The swirling singles out the stable particles of powder from all sides. By spraying on a binder such as water, the powder particles gradually stick to one another to form larger particles.

Compacting and agglomeration process have the following advantages:
1. Increasing the shelf life.
2. Stability of enhanced process.
3. Reducing a negative interaction with other food constituents.
4. Improved handling properties: dust free flowing.
5. Adjustable flavor properties: solubility, texture and color particle size.
6. Controlled the flavor release during food processing.
7. Excellent flavor profile retention.

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*Fig. 13. Principle of a spray drying plant [58].*

*Fig. 18. Compacting and agglomeration process.*

*Fig. 19. Spray drying is customarily complemented by the process of compacting and agglomeration to coarsen the product.*
Fig. 14. Flavor emulsion (Liquid state) & Encapsulation flavor powder [69].

Fig. 15. Shear mixer before spray drying [70].

Fig. 16. Homogenizer before spray drying emulsion [70].
Fluid-bed methods (granulation and coating):

In these method flavors coated by selecting a specific coating material. In the case of agglomeration, spray granulation occurs in a fluid bed. The technology allows specific particle size (0.2mm-1.2mm) and porosities to be designed into the fact that extremely precise particle sizes and distribution can be achieved.

By repeating spraying, applying and drying drops in a fluid bed, it is possible to form

Fig. 17. Schematic illustrating the process of encapsulation by spray-drying [58].

Fig. 18. Flavor retaining mechanism [10].

*Explain by the “Selectiva Diffusion Theory” (Thijssen, 1968). As the water concentration at the droplet surface decreases, the diffusion coefficients of the volatile components decrease by several orders of magnitude, more sharply than that of water. The dry surface acts as a semi-permeable membrane permitting the continued loss (or diffusion) of water, but efficiently retaining (or stopping) volatile flavor.*
granulate like the peels of an onion. Moreover this technology also enables the properties of the encapsulated flavors.

A particular advantage offered by this technology, it is possible to produce kibbled flavor without the need for any intermediate steps, while achieving significantly better encapsulation than with spray drying.

**Comparison of fluid bed granulation and spray drying:**

Both processes start off identically, with an aqueous emulsion in the spray drying process, but in fluid bed granulation, repeated spraying and drying produces a particle with onion–like structure and an approximately 100- micrometer flavor droplet forms. In addition to coating with sugar– based (i.e. water-soluble encapsulation material), is also possible to employ the same fluid –bed method to provide fatty coating.

**Fluidized-bed coating:**

Fluid bed coating is commonly used for coating solid particles [71]. The formation mechanism of microparticles using this technique can be divided into three stages: nucleation, transition, and ball growth. In the beginning of the encapsulation process, particles are suspended in the coating chamber. Droplets of the polymer solution are then sprayed, increasing the probability of particle–droplet impact and spread on the particle surface. At the end of this process, droplets are evaporated and formed into microparticles [72] (Figure 20). This manufacturing technique is ideal for the food industry, because of its high versatility, relatively high batch size, and simplicity [73].

The different styles of fluid-bed are bottom spray, top spray and tangential spray used for encapsulating liquids or solid absorbed into porous particles (Figure 21).

**Wurster coating:**

Particles of the active ingredient, spheres or granules, are suspended in an upward-moving stream of air and then covered with a spray of liquid coating material.

The capsules are then shifted to an area where their shells are solidified by cooling or solvent vaporization (Figure 22). The whole cycle of suspending, spraying and cooling is repeated until the capsule’s walls are of the desired thickness. The size of the capsules for this technique is commonly large (~100 microns). This method gives improved control and flexibility as compared to pan coating.

Unfortunately, the fluid- bed methods have various disadvantages including; water soluble and polar cores lost, processing problems, aggregation, core wet ability and difficult to operate and control, high costs, and using of batch process.

**Spray chilling [53,74]:**

Spray chilling method can be accomplished by equipment of spray drying when protective coating is applied like melt (Figure 23).

It is conducted according to the following steps:

Dispersed an active ingredient in Fat (coating material melt). Spray into a cool chamber to allow to solidify. Collect as a powder.

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Fig. 19. Principle of plant compacted products (A) and agglomerated products (B) [58].

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Microencapsulation of vitamins with digestible waxes for taste masking.

Spray chilling is applied in encapsulation of minerals, yeasts, leavenings, flavorings, and high potency sweeteners (aspartame).

Freeze-drying [75]:
Freeze-drying (lyophilization) is used for the dehydration of volatiles materials that are sensitive for heat, it is suitable to encapsulate of natural aroma and water-soluble essences as well as drugs (Figure 24). Generally, this process required long dehydration period (commonly 20 h), the retention of volatile compounds during this process is dependent upon the chemical nature of the system.

Pan coating [41,76]:
Pan coating are widely used in the pharmaceutical industry. It is old industrial procedure to form small, coated particles or tablets. The particle is tumbled in pan while carried materials are applied slowly (Figure 25). The pipe of the blower stretches into pot for an evenly heating distribution while the coating pan is rotating.

Extrusion:
Extrusion processes taken a great significance in recent years. Under this method even highly viscous wall material can be processed into glassy methods that are characterized by long shelf life and high stability, water or other plasticizers and softeners is added to carbohydrates or sugar, which are then melted. The flavor is added next, the flavored melt is forced out of the extruder die under high pressure, when quickly dried, the extruder forms an amorphous, glassy yet firm mass that completely encloses the flavor droplets, forming small needle shape pellets (Figure 26).

This process is especially well suited for encapsulating of highly sensitive citrus flavors [40,77-80].

The advantages of encapsulation of volatiles in glassy carbohydrate matrices by extrusion are as follow:

- Long shelf life (up to 5 years) compared to spray drying (~1 year).
- Rather large particles (500 to 1000 μm).
- Low load with simple carbohydrates (~10 %) e.g. "Locked-in flavors", citric oils in glassy
Fig. 22. Wurster Coating [42].

Fig. 23. Spray chilling (Prilling) [53].

Fig. 24. Freeze-drying [75].
sucrose-glucose-glycerin matrix.

- High load with hydrophobically modified starch (50 %) e.g. hydrophobically modified starch (octenyl substituted starch) for encapsulation of up to 50 % flavor oils

**Co-extrusion [81]:**

In co-extrusion, the dual fluid stream for liquid active ingredient and the wall materials are pumped into the concentric tubes and droplets formed under vibration influence (Figure 27). The shell hardened by chemical cross linking, solvent evaporation or cooling. Also, different nozzle extrusion types were developed in order to optimize the process.

**Spinning disk:**

These methods are relied upon spinning of disk and simultaneous core material motion and wall material exiting from this disk in droplet form. Wall material particles and capsules will collect below disk. The capsules will separated from wall particles by the operation size (Figure 28) [12,25].

The Spinning Disk process operates according to the following steps:

1. Active particles suspended in liquid wall are poured into rotating disc.
2. Due to the disc spinning action, active particles coated with wall material.
3. Active particles are cast from the disc edge by centrifugal force.
4. After that wall material will solidify by

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**Fig. 25.** Representation of a typical pan coating [34].

**Fig. 26.** Flow diagram of encapsulation of food flavors by extrusion [32].
This technology is characterized by its low cost, relatively simple, rapid and high production efficiencies.

*Cocrystallization [7,11,82]:*

Cocrystallisation means the inclusion of flavorings in carbohydrate surface between lipophilic flavorings and the water phase (Figure 29). It is applied in encapsulation of colorants, flavors and honey.

The product obtained by cocrystallization is offer little protection to active continuous wall, not emulsion, has larger particle and free flowing.

*Chemical process:*

*Molecular inclusion (β-cyclodextrin)*

The complexes of molecular inclusion are the other technique to the flavoring encapsulation substances. β -Cyclodextrin is very good suited to this method [6,84].

The cyclodextrins are cyclic and non-reducing oligosaccharides that is form crystalline complexes to chemical variety. The cyclodextrins are formed by starch fermentation and it consists of 6, 7, 8 -gluopyranose subunits-alpha, β, gamma forms, respectively (Figure 30). Inclusion complex is formed when the flavoring material mixed with cyclodextrins in the solution. The guest molecule are resides inside cyclodextrins ring structure. Ring size can limit complexion of guest molecule. The polarity, hydrocarbon saturation and the flavor components functional groups can affect whether or not molecule will include or to what degree inclusion will take place (Figure 31).

Cyclodextrins and flavor components were filtered, air dried and coprecipitated. The solubility of the beta, alpha & gamma cyclodextrins tend to be best candidate for flavor inclusion chemicals like they can accommodate. The guest molecules wide variety was readily available reasonably priced and provide best stability once in dry form. [85].

The seven precipitation methods are used at lab and are not particularly practical for
large scale manufacturing but are acceptable to demonstrate process properties. Cyclodextrins (CDs) had found a large applications number in many areas. Since they are became available in high quantities like food chemistry, cosmetics and pharmaceutical industries. They are used in organic or polymer chemistry.

β – CDs are most natural form of common CD (Figure 32). It has 21 groups of hydroxyl, which are 7 primaries and 14 secondary. All these groups of hydroxyl are available like starting points to structural modification and various functionally groups had introduced into macro cycling.

Molecular inclusion is applied in encapsulation of colors for meat products and microorganisms. The product is characterized by free flowing dry load from 6 to 15% while the load is based on molecular weight of guest molecule. In addition,
the process has advantages of high binding, controlled release in application and low process costs. Also the disadvantages of the process are as follow:

1. Non-uniform component
2. Low flavor load
3. Low solubility (β, 0.01 - 0.035g/100 ml)
4. Perhaps a higher usage level
5. β and γ are self affirmed
6. Non-uniform component binding - flavor

imbalance or no active inclusion

**Liposomes:**

Liposomes are often used in the food and pharmaceutical industries for encapsulating of an aqueous solution within a membrane of phospholipids. Liposomes were microscopies vesicles phospholipid composed bilayers surrounding aqueous comport. As presented in Figures (33 & 34), the liposome encapsulates

![Fig. 30. Structure of β-cyclodextrin [32].](image1)

![Fig. 31. Model of cyclodextrin inclusion complex [86].](image2)
the lipophilic drug within the lipid bilayer of the hydrophobic region while also entrapping a hydrophilic drug within an aqueous medium of the hydrophilic region, [87,88]. Liposomal encapsulates can protect bioactive agent from chemical stresses and environmental, including the presence of reactive chemicals or enzymes, and exposure to extreme temperature, pH and high ion concentrations [89]. The form of liposomes capsules 1 or more phospholipids layers with particle size start by 25 nm to several micrometers (Figure 33). Liposomes were applied in cheese making. It’s used in food preparation of emulsions like mayonnaise, margarine and spreads are developing area (Figure 35).

**Control release [32,90,91]:**
Flavor release involves a wide variety of requirement: the solubility is driven in which a flavor capsule dissolved by water thus releasing flavor. The speed which capsule dissolves, and which flavor released, it can be governed through selection of carrier. On the other hand, it is possible to design water – insoluble encapsulation systems that keep the flavor encapsulated in aqueous products until the products are eaten [79].

The temperature that driven the flavor release can be achieved, for example by coating encapsulated flavor with a specific fat that melts at a given temperature. One field of application for this would be cake mixes. In other applications,
like candy, it is possible to use small gelatin capsules that are mechanically crushed when the consumer chews the product thus releasing the flavor immediately.

In these above examples, encapsulated flavors are released in entirely different ways either during the production process itself, when the food is prepared or not until the product is eaten. So flavors which are used in baked goods are developed in oven temperatures 70°C. While the kibbled flavors that are employed in teas, soup mixes or candies are not released until the product is consumed. Soup mixes, for example, develop some of their flavor after the hot water was poured over mix in order to produce soups characteristic aroma.

The situation is similar in connection with teas; the flavors develop while the tea steeping. In the case of chewing gum, the flavor should be released (instantly) as soon as the gum is chewed, yet it should still be clearly perceivable even after being chewed for 10 to 20 minutes (long-lasting effect).

Since no encapsulation process methods can satisfy all these requirements simultaneously, technologies for encapsulating flavor represent a core competence (Figure 36).
Flavor encapsulation is the key technology for flavor delivery in liquid and dry systems. Several different methods are available for producing the dried flavoring materials. These are freeze or spray drying, inclusion complexation, extrusion and coacervation. The selected type was depended on material of flavoring, equipment availability, budget, and applications.

The microencapsulation technology is yet to become a conventional tool for food industry to develop the healthy and novel food products which can be achieved by multidisciplinary based research approach and consideration of industrial requirements and constraint. In the applications of different encapsulation methods for food ingredients, only the approved products by U.S. food and drug administration (FDA) can be used and the generally recognized as safe (GRAS) list should be consulted.

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ENCAPSULATION TECHNIQUES OF FOOD BIOPRODUCTS...

نتيجة التغليف للمواد الغذائية الحيوية

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التغليف عبارة عن تقنية يمكن فيها تحويل القطرات السائلة إلى جزيئات صلبة بواسطة نظام مستمر. يمكن تسمية الغلاف بالجدار، فالغلاف مصمم لحماية المواد من العوامل التي قد تسبب تدهورًا. يتحكم تصميم الجدار في إطلاق مواد التغليف الأساسية وفقًا للشروط المطلوبة. تطبيقات التغليف في صناعة المواد الغذائية لها أهميتها. تحاول المواد الحساسة مثل الزيوت العطرية والزيوت الثابتة من التفاعلات الكيميائية والأكسدة والتبخر، وذلك بتغليفها في صورة جافة. يحافظ غلاف المركبات العطرية على المنتج حتى الاستخدام، وهذا يضمن الجودة العالية والقيمة التجارية للمنتج. يتم مناقشة العديد من تقنيات التغليف التي يتم استخدامها تجارياً أو التي يتم تطبيقها في صناعة المواد الغذائية. ويشمل ذلك التجفيف بالرزر، التجفيف بالبثق والثلوج وغيرهم من التقنيات.