

Novel Technologies for Food Processing and Shelf Life Extension
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Lecture – 37
Microencapsulation (Part 1)

An important aspect of food processing i.e. microencapsulation technology will be studied. This has a lot of potential for being used in different products for various purposes. This topic will be taken up in two parts; in the part 1, the basics of microencapsulation technology including different methods of microencapsulation will be introduced and this will be followed by the application of this technology in food industry in the second part of the lecture.

Microencapsulation

- Microencapsulation is a process by which very tiny droplets or particles of liquid or solid materials are surrounded or coated with a continuous film of polymeric material.
- Particles having diameter between 3 – 800 μm are known as micro particles or microcapsules or microspheres.

Active ingredient > Process > Coated particle

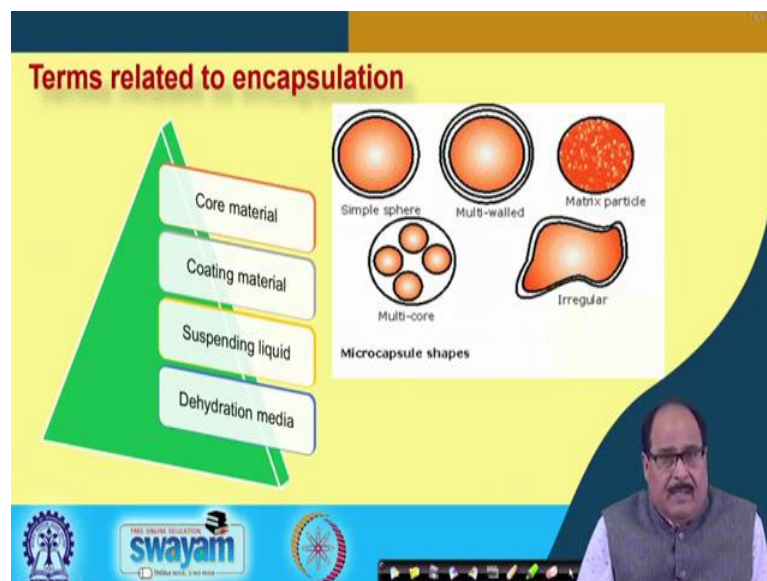
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There is some active ingredient. This is subjected to a suitable process technology or process called microencapsulation in which a coating of the material is applied over it and finally, a coated particle (microencapsulated particle) is obtained where these ingredients are protected.



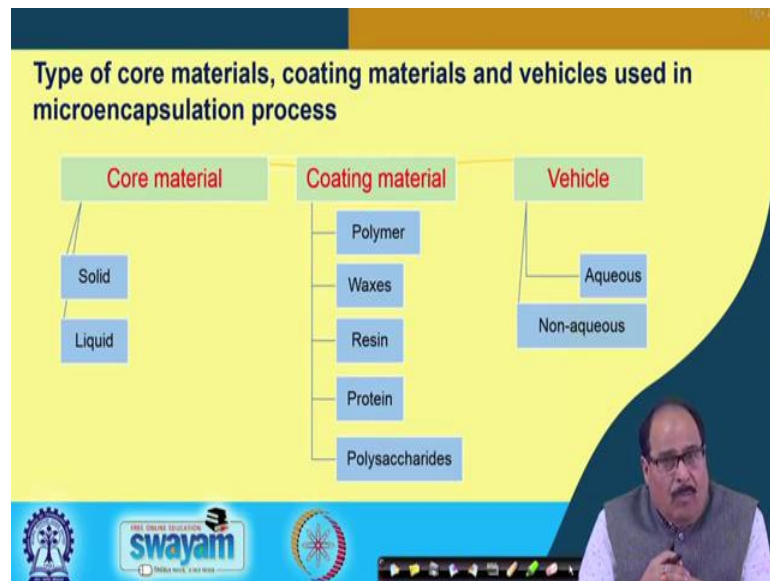
Encapsulation provides stability and protection to the materials, enhances acceptability, improves flavor and other properties of the product. It may even be used for controlled release or targeted nutrition delivery or for immobilization purposes etc. So, encapsulation technology provides lot of benefits to the food processing industries as well as consumers.



Different terms are related to the microencapsulation. There are some core materials and on this core material some coating is applied. For application of the coating, there should be some suspending liquid or vehicle through which coating is applied. Finally, dehydration media i.e. the solutions or vehicle which are used to apply the liquid is

removed or evaporated from the material so that the coating which is formed on the core material becomes rigid, it provides the structure and desired properties.

Depending upon the different types of processes and materials used, the microcapsules of various shapes and sizes can be formed. Different shapes include simple sphere, multi walled microcapsules, matrix particles, irregular shape microcapsules or multi core microcapsules.



The core material used in the microencapsulation process might be a solid or a liquid material. Coating materials generally used are polymers, waxes, resins, protein or polysaccharides. The vehicle to be used for microencapsulation may be an aqueous or even non-aqueous material. So, both aqueous and non-aqueous vehicles can be used for this purpose.

Different coating materials which are used for microencapsulation

<u>Water soluble resin</u>	<u>Water insoluble resin</u>	<u>Wax and lipid</u>	<u>Proteins</u>
<input type="checkbox"/> Gelatin	<input type="checkbox"/> Ethyl cellulose	<input type="checkbox"/> Paraffin	<input type="checkbox"/> Skim milk protein
<input type="checkbox"/> Gum arabic	<input type="checkbox"/> Polyethylene	<input type="checkbox"/> Carnauba wax	<input type="checkbox"/> Milk protein isolates
<input type="checkbox"/> PVP	<input type="checkbox"/> Poly methacrylate	<input type="checkbox"/> Bees wax	<input type="checkbox"/> Soy protein isolates
<input type="checkbox"/> CMC	<input type="checkbox"/> Cellulose nitrate	<input type="checkbox"/> Stearic acid	<input type="checkbox"/> Pea protein
<input type="checkbox"/> Methyl cellulose	<input type="checkbox"/> Silicon	<input type="checkbox"/> Stearyl alcohol	
<input type="checkbox"/> Arabino galactan			
<input type="checkbox"/> Polyvinyl acrylate			
<input type="checkbox"/> Polyacrylic acid			

Different coating materials which are commonly used for microencapsulation process include water soluble resins like gelatin, gum arabic, carboxymethyl cellulose, methyl cellulose etc. Water insoluble resins are ethyl cellulose, polyethylene, polymethyl crylate etc. Wax and lipids like paraffin, carnauba wax, bees wax, stearic acid etc. can also be used as coating material. Or even different proteins like skim milk protein, milk protein isolates, soya protein isolates, pea proteins etc. are used for this purpose.

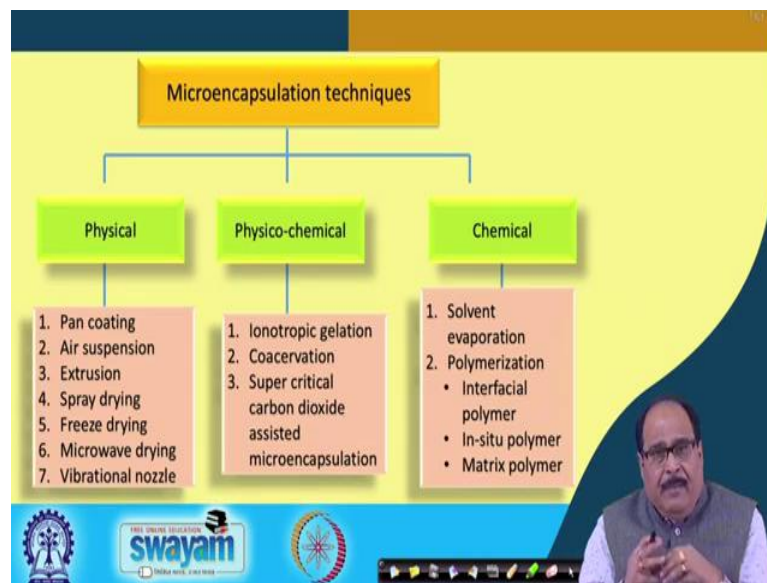
Formulation considerations

Core materials	<ul style="list-style-type: none"> The solid core can be mixture of active constituents, stabilizers, diluents, excipients and release-rate retardants or accelerators.
Coating/ Wall materials	<ul style="list-style-type: none"> Compatible and non-reactive with the core material. Provide desired coating properties like strength, flexibility, impermeability, optical properties, non-hygroscopicity, tastelessness, and stability. Economic food-grade substance.

There are certain points to be considered while deciding the core material as well as coating or wall materials. For example, the solid core materials, can be mixture of active constituents, stabilizers, diluents, excipient and release rate retardants or accelerators etc.

So, core material either alone or in combination with various function improving constituents like stabilizer, diluents, etc.

As far as the coating and wall materials are concerned they should be comfortable and non reactive with the core material, they should provide desired coating properties like strength, flexibility, impermeability, optical properties, non-hygroscopicity, tastelessness as well as stability to the materials and more importantly they should be economic food grade substances.

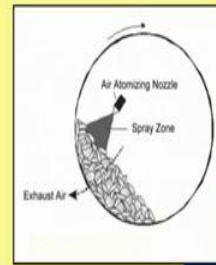


Microencapsulation techniques can be broadly classified into three major categories; physical, physico-chemical and purely chemical processes.

The physical methods of microencapsulation include pan coating, air suspension, extrusion, spray drying, freeze drying, microwave drying, vibrational nozzle and so on. The physico-chemical processes include ionotropic gelation, coacervation or super critical carbon dioxide assisted microencapsulation. The purely chemical methods include solvent evaporation and polymerization.

Pan coating

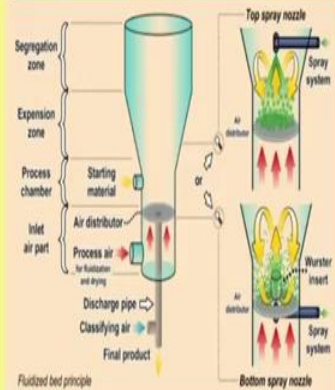
- Oldest industrial procedure for forming small, coated particles or tablets.
- Solid particles greater than $600\ \mu$ in size are generally considered essential for effective coating.
- The particles are tumbled in a pan or other device while the coating material is applied slowly.
- Coating solution is sprayed on the solid core material.
- Usually warm air is passed over the coated material as the coating is being applied which evaporates the solvent.



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Air suspension (Wurster process)



- ✓ The fine core materials are suspended in a vertical current of air and sprayed with the coating material.
- ✓ After evaporation of solvent, a layer of encapsulating material is deposited on core.
- ✓ Gives improved control and flexibility as compared to pan coating.

Disadvantage
Agglomeration of the particles to some larger size is normally achieved.



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Microencapsulation through extrusion

The core material, dispersed in molten carbohydrate, is pressed through one or more dies into a bath of cold, dehydrating liquid.

Melt injection

- The wall material solidifies forming an encapsulating matrix to entrap the core material.
- The granules are recovered by filtration or centrifugation.
- The residual solvent is removed by air-drying or vacuum-drying.

Melt extrusion Similar to that of melt-injection.

- The major difference is that it is a vertical screw-less process with surface-washed particles, while the melt injection is a horizontal screw process with particles that are not surface-washed.

Centrifugal extrusion Consists of a concentric feed tube through which wall and core materials are pumped separately to the nozzles mounted on the outer surface of the device.

- Core material flows through the center tube, and
- Wall material flows through the outer tube.

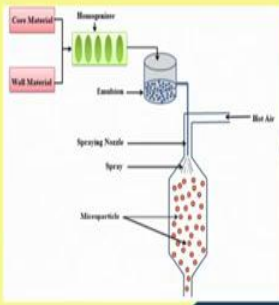
Microencapsulation through extrusion

There are three variations in this case; melt injection, melt extrusion and centrifugal extrusion. In the melt injection, the core material is dispersed in molten carbohydrates, is pressed through one or more dies into a bath of cold dehydrating liquid. The wall material solidifies forming an encapsulating matrix to interrupt the core material. The granules are recovered by filtration or centrifugation and the residual solvent is removed by air drying or vacuum drying. The melt extrusion method is similar to that of the melt injection method. The major difference is that the melt extrusion method is a vertical screw-less process whereas, the melt injection is a horizontal screw process. In the melt extrusion process, they contain surface washed particles whereas; in the melt injection process, the particles are not surface washed.

The third type of the extrusion technology for microencapsulation is centrifugal extrusion. It consists of a concentric feed tube through which the wall and core materials are pumped separately to the nozzle mounted on the outer surface of the device. The core material flows through the center tube and the wall material flows through the outer tube. Vacuum drying process is used to provide rigidity to the microcapsules.

Spray drying

- It is the most commonly used encapsulation method in the food industry.
- The process is economical and flexible.
- It produces particles of good quality.
- The process involves three basic steps
 - ✓ Preparation of a dispersion or emulsion to be processed,
 - ✓ Homogenization of the dispersion, and
 - ✓ Atomization of the mass into the drying chamber.
- Spray dried ingredients typically have a very small particle size (generally less than 100 μm) which makes them highly soluble.



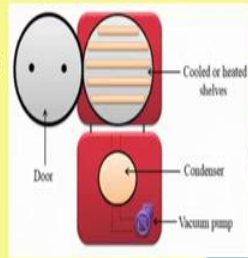
The diagram illustrates the spray drying process. It starts with 'Core Material' and 'Wall Material' being combined in a 'Homogenizer'. The mixture then goes to a 'Tank' and then to a 'Spraying Nozzle' where it is atomized into a 'Spray'. This spray enters a drying chamber where 'Hot Air' is used to dry the particles, resulting in 'Microcapsules'.

Spray drying: It is the most commonly used encapsulation method in food industry. The process is economical and flexible. It produces particles of good quality. The process involves three basic steps like, preparation of a dispersion or emulsion which is to be processed it is followed by homogenization of the dispersion and finally, atomization of the mass into the drying chamber.

Spray dried ingredients typically have a very small particle size generally less than 100 μm or so and which makes them highly soluble. Spray technology technology in fact, produces microcapsules of good solubility.

Freeze drying

- It is also known as lyophilization or cryodesiccation.
- Before drying, the oil is dissolved in water and frozen (between -90°C and -40°C).
- Then the surrounding pressure is reduced and enough heat is added to allow the frozen water in the material to sublime directly from the solid phase to the gas phase.
- Freeze-dried materials seem to have the maximum retention of volatile compounds in comparison to that of spray drying.

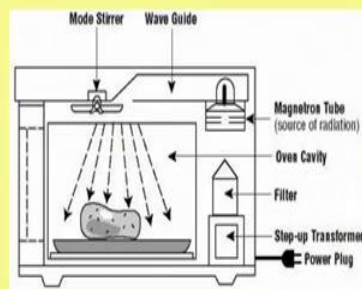


Freeze drying

Freeze drying is another good technology for microencapsulation of the heat sensitive materials. It is also known as lyophilization or cryodesiccation. So, before freeze drying the essential oils or such other materials are first frozen and depending upon the type of the material the freezing temperature may range between -90 to -40°C . After the material is frozen, the surrounding pressure is reduced in the set up and enough heat is added to allow frozen water in the material to sublime directly from the solid phase to the gas phase. These freeze dried materials seem to have the maximum retention of volatile compounds in comparison to those of the spray drying materials.

Microwave drying

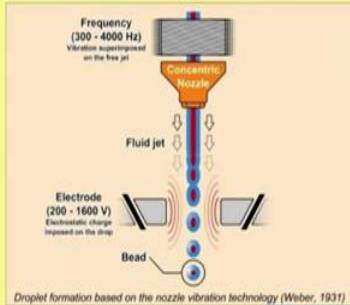
- It is based on a unique volumetric heating mode and internal vapour generation facilitated by electromagnetic radiation of 2450 MHz .
- Heating from interior of a food product leads to the buildup of internal vapor pressure.
- This drives the moisture out of the product.
- A significant reduction in drying time is accompanied by an improvement in product quality.
- Beneficial for temperature sensitive products.



Microwave drying is another technology for microencapsulation which is based on the unique volumetric heating mode and internal vapour generation facilitated by electromagnetic radiation of 2450 MHz. Heating from the interior of a food product leads to the buildup of internal vapour pressure and this drives the moisture out of the product. A significant reduction in drying time is accompanied by an improvement in product quality. It is particularly beneficial and desired for encapsulation of the temperature sensitive products.

Vibrational nozzle

- This is based on Rayleigh instability.
- A fluid stream of liquid core and shell materials is pumped through concentric tubes and forms droplets under the influence of vibration.
- To guarantee production of uniform beads and to avoid large size distributions due to coalescence effects during the flight, the droplets pass through an electrostatic field to be charged.
- As a result these droplets do not hit each other during the flight.



The diagram illustrates the process of droplet formation using a vibrational nozzle. It shows a 'Concentric Nozzle' at the top, which produces a 'Fluid jet'. This jet passes through an 'Electrode' (200 - 1600 V) where an 'Electrostatic charge' is imposed on the drop. The resulting 'Bead' is shown at the bottom. The process is based on Rayleigh instability, with a frequency of 300 - 4000 Hz applied to the nozzle. A reference is given: 'Droplet formation based on the nozzle vibration technology (Weber, 1931)'.

Frequency (300 - 4000 Hz)
Vibration superimposed on the flow jet

Concentric Nozzle

Fluid jet

Electrode (200 - 1600 V)
Electrostatic charge imposed on the drop

Bead

Droplet formation based on the nozzle vibration technology (Weber, 1931)

The vibrational nozzle technology is based upon the Rayleigh instability in this process. A fluid stream of liquid core and shell material is pumped through the concentric tubes and forms droplets under the influence of vibration.

To guarantee production of uniform beads and to avoid large size distributions due to the coalescence effects during the flight, the droplets are passed through the electrostatic field to be charged. As a result these droplets do not hit each other during the flight and the separate individual droplets or microcapsules are formed.



Ionotropic gelation (polyelectrolyte complexation): This technique involves interaction of a cation or an anion with an ionic polymer to generate a highly cross linked structure.

Generally, sodium alginate is used which is first dissolved in water, then in this solution of sodium alginate in water, polymers are added with continuous stirring. These drugs are dispersed properly they are placed on magnetic stirrer and the ionic solution is added drop wise through needle which results into the ionotropic gelations or microspheres are formed.

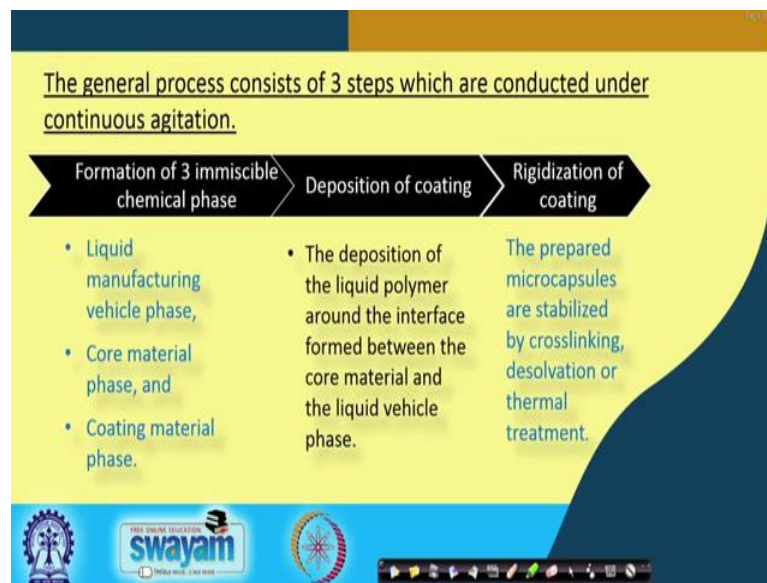
Coacervation

- Coacervation is a unique type of electrostatically driven liquid-liquid phase separation resulting from association of oppositely charged macro-ions.
- The term 'coacervate' is sometimes used to refer to spherical aggregates of colloidal droplets held together by hydrophobic forces.
- Two methods for coacervation are
 - (i) Simple coacervation, and (ii) Complex coacervation.
- The mechanism of microcapsule formation in both processes is identical, except for the way in which the phase separation is carried out.
- In simple coacervation a desolvent agent is added for phase separation whereas the complex coacervation involves complexation between two oppositely charged polymers.

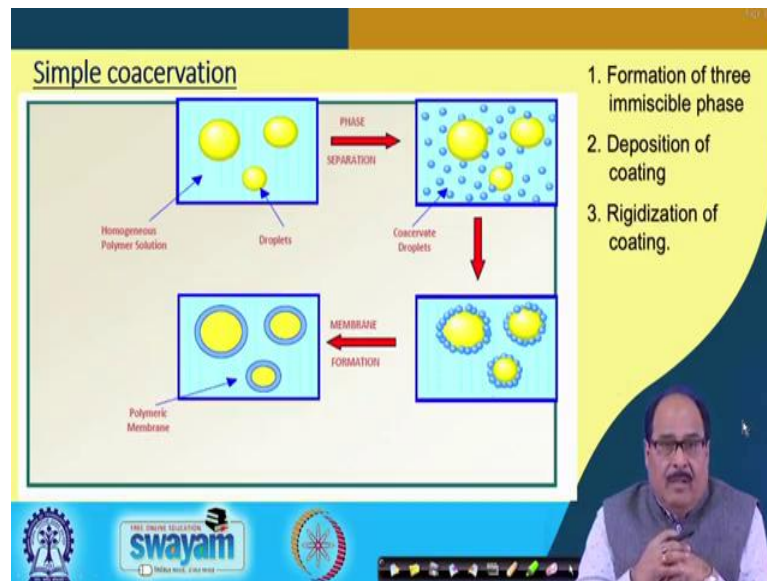
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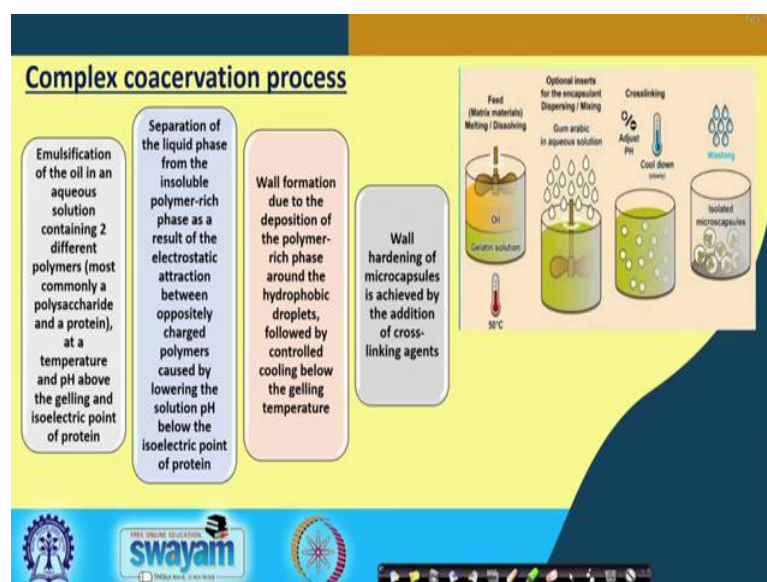
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The general process consists of three steps which are conducted under continuous agitations and the first step includes the formation of three immiscible chemical phase, maybe a liquid manufacturing vehicle phase, core material phase and the coating material phase. Then the second step is the deposition of coating where the liquid polymer is deposited around the interface formed between the core material and the liquid vehicle phase. And, finally, rigidization of the coating takes place where the prepared microcapsules are stabilized by cross linking desolvation or thermal treatment.



In this picture, the process of simple coacervation can be seen. There are three stages for formation of three immiscible phase deposition of coating and rigidization of the coating material. First, there are some droplets and these core material are put into a homogeneous polymer solution. Then it results into the phase separation process where the coacervate droplets of the polymer solutions are formed, and this coacervate droplets, they now get assembled on the core material slowly and slowly and finally, the membrane formation takes place. So, in this way the microcapsules with the desired characteristics and strength are formed.



Complex coacervation process: In the first step, the emulsification of the oil in an aqueous solution containing two different polymers generally polysaccharides and a

protein are emulsified at a temperature and pH above the gelling point and isoelectric point of protein. The second step is the separation of the liquid phase from the insoluble polymer rich phase as a result of the electrostatic attraction between oppositely charged polymers caused by lowering the solution pH below the isoelectric point of protein. The third step is the wall formation due to the deposition of the polymer rich phase around the hydrophobic droplets followed by controlled cooling below the gelling temperature. And finally in the fourth step, then wall hardening i.e. the rigidization of the coated materials results into the wall hardening of the microcapsules is achieved by the addition of cross linking agents.

Super critical fluid technology

- The system consists of a high-pressure stainless steel impregnation vessel, a temperature-controlled bath, a magnetic stirring plate, a pressure transducer, and a high-pressure CO₂ liquid pump.
- The impregnation cell is fed with a fixed amount of essential oils/core material at the bottom.
- Wall materials are placed in a stainless mesh elevated from the bottom by a support.
- After loading the oils and the wall materials, the impregnation cell is immersed in the waterbath at less than 80 °C and CO₂ is then fed into the cell until the desired pressure is achieved in order to ensure the solubilization of active ingredients in supercritical CO₂.

The diagram illustrates the experimental setup for supercritical fluid technology. It features a central stainless steel impregnation vessel containing oil and wall materials, which is submerged in a thermostatic water bath. The system is equipped with a CO₂ liquid pump, a pressure transducer, and a magnetic stirrer. The vessel is connected to a CO₂ inlet and a CO₂ outlet. The water bath is controlled by a temperature controller.

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