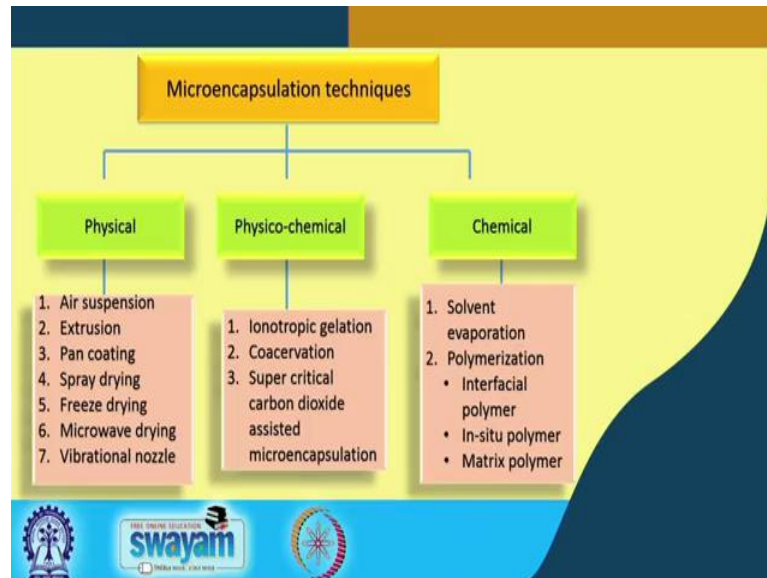


**Novel Technologies for Food Processing and Shelf Life Extension**  
**Prof. Hari Niwas Mishra**  
**Department of Agricultural and Food Engineering**  
**Indian Institute of Technology, Kharagpur**

**Lecture – 38**  
**Microencapsulation ( Part 2 )**

This is the 2nd part of lecture on microencapsulation.



In the earlier part of this topic, the basic principles of microencapsulation and the physical and physico chemical methods of microencapsulation were discussed. Now in today's lecture, the chemical methods of microencapsulation like solvent, evaporation and polymerization will be studied. Further, the applications of this technology in food processing or in different food products will be elaborated.

### Solvent evaporation

**Step 1 :** Formation of a solution/ dispersion of the core material into an aqueous solvent phase.

**Step 2 :** Emulsification of the core material into an aqueous phase containing a suitable stabilizer, thus, forming a o/w emulsion.

**Step 3 :** Removal of the solvent from the dispersed phase by extraction or evaporation leading to precipitation and formation of the microspheres.

primary emulsion (w/o)

oil phase + aqueous phase → homogenized → immediately injected → homogenized

immediately injected → double emulsion (w/o/w)

DCM DCM

hardened microspheres was collected, washed and lyophilized

continuously stirred 'hardening tank'

swayam

## Solvent evaporation

The solvent evaporation methods includes 3 step. First of all there is a formation of solution or dispersion of the core material into an aqueous solvent phase. This is followed by emulsification of the core material into an aqueous phase containing a suitable stabilizer and therefore, this results in the formation of a water in oil emulsion or oil in water emulsion. After this, in the third step, the solvent is removed from the dispersed phase either by extraction or evaporation and this leads to precipitation and formation of microspheres.

### Polymerization

- It occurs in the continuous phase rather than on both sides of the interface between the core material and the continuous phase.
- The microcapsule formation process is conducted by the addition of oil in a melamine–formaldehyde resin solution and a sonication process to emulsify the oil in the aqueous phase.
- The resin is then added under stirring and adjustment of the emulsion pH to acidic range is done.
- The microcapsule shell formation occurs due to the reaction of melamine with formaldehyde at the interface of oil droplets, producing a cross-linked film of melamine–formaldehyde polymer.

Step 1: Melamine-formaldehyde precondensate and copolymer in water

Step 2: Pre-crosslinked

Step 3: Emulsion by agitation

Step 4: Wall formation

Oil

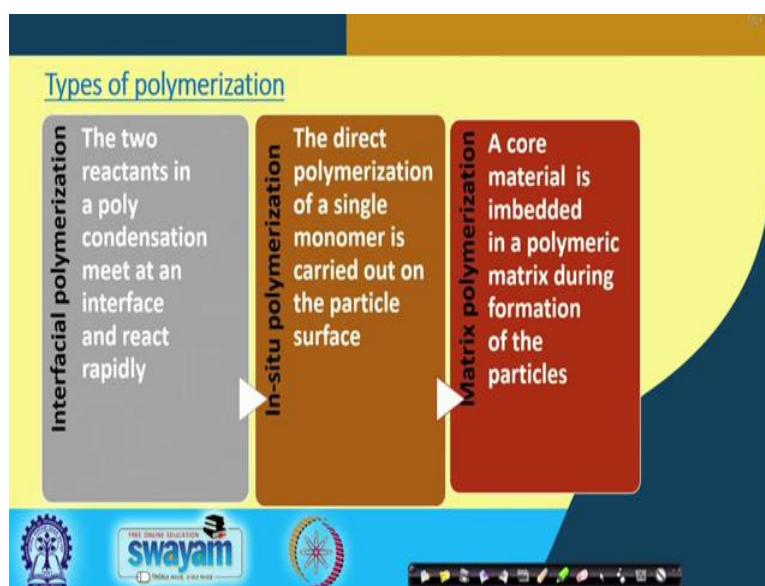
Melamine-formaldehyde

swayam

## Polymerization

The polymerization process occurs in the continuous phase rather than on both sides of the interface between the core material and the continuous phase. The micro capsule formation process is conducted by the addition of oil in a melamine-formaldehyde resin solution and a sonication process to emulsify the oil in the aqueous phase. The resin is then added into the emulsion under continuous stirring and finally, the adjustment of the emulsion pH is done to the acidic range. After this, the micro capsule shell formation occurs due to the reaction of melamine with formaldehyde at the interface of oil droplets producing a cross linked film of melamine-formaldehyde polymer.

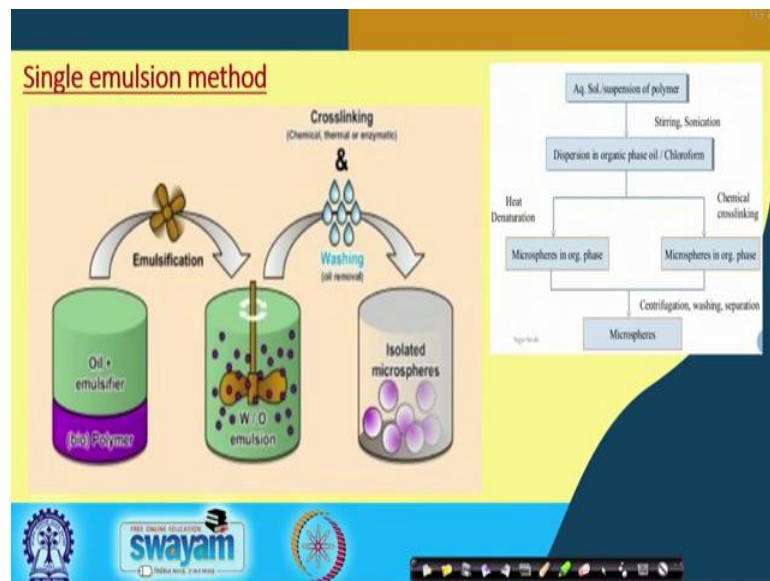
The whole process of polymerization is schematically or pictorially presented in the figure. The solution of melamine-formaldehyde is a pre condensed form and co polymer added in water has a pre cross-linked polymer. Then oil is added step by step and it is agitated to form the emulsion, then the wall formation takes place and finally, rigidization of the wall is encapsulated or coated with melamine formaldehyde membrane.



## Types of polymerization

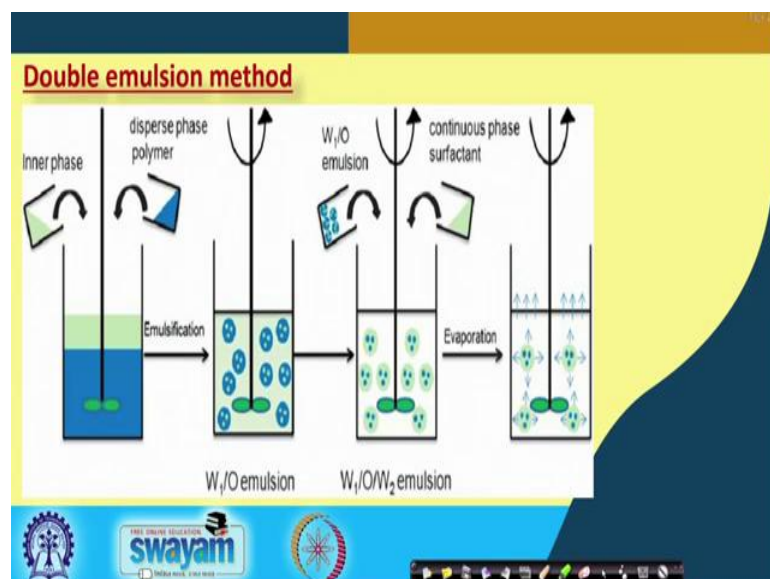
The polymerization can be conducted in 3 ways; one the interfacial polymerization, in-situ polymerization or matrix polymerization. In interfacial polymerization, 2 reactants in a poly condensation meet at an interface and react directly. In the case of in-situ polymerization, there is a direct polymerization of single monomer and this is carried out

on the particle surface. Matrix polymerization includes a core material embedded in a polymeric matrix during formation of the particles.



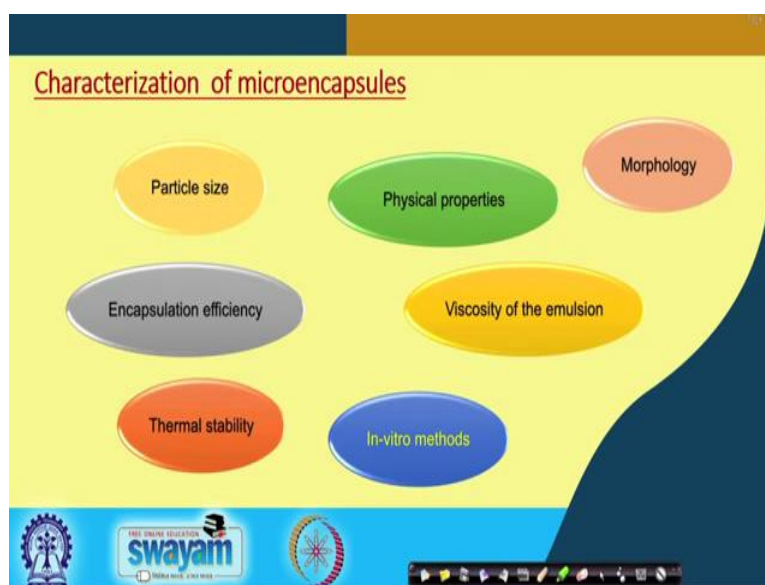
### Single emulsion method

The process can be done either in single emulsion method or in double emulsion method. In the single emulsion method, (see picture) oil and polymer are mixed using proper sonication or mixing and water in oil emulsion is formed. This water in oil emulsion is added with the cross-linking agents. By heating the denaturation occurs or by the process of chemical cross-linking microspheres are formed in the organic phase. Once the microspheres are formed, they are isolated either by centrifugation, washing and separation processes.



## **Double emulsion method**

In the double emulsion method, (see picture) the inner phase and dispersed polymer phase are mixed together to emulsify it and in this process, water and oil emulsion is formed. The water in oil emulsion is added with the continuous phase having surfactant by stirring etc., the suspension or emulsion is formed. So, the double emulsion water-oil-water once formed and using appropriate methods of evaporation, heating etc. the solvents are evaporated and finally, the rigid microspheres or microcapsules are formed.




## **Characterization of microencapsules**



They include determination of particle size, physical properties of the microcapsules, encapsulation efficiency, viscosity of emulsion, thermal stability of microcapsules, morphologies, and in-vitro studies. It is a very important aspect that the microcapsules would be properly characterized to ensure the purpose of microencapsulation.



**Application of microencapsulation technology in food processing**



- Adding ingredients to food products to improve nutritional value can compromise their taste, colour, texture and aroma.
- Sometimes they slowly degrade and lose their activity, or become hazardous by oxidation reactions.
- Microencapsulation offers promising solutions to these problems.
  - ✓ EPA and DHA levels in milk can be increased without any adverse effects on its flavour by the incorporation of microencapsulated fish oils.
  - ✓ Milk can be fortified with vitamin D entrapped in liposome.





THE ONLINE SOLUTION **swayam**  
विद्यया ऽर्द्धो ज्ञानेन चतुर्विधः

**Application of microencapsulation technology in food processing**

- Adding ingredients to food products to improve nutritional value can compromise their taste, colour, texture and aroma.
- Sometimes they slowly degrade and lose their activity, or become hazardous by oxidation reactions.
- Microencapsulation offers promising solutions to these problems.
  - ✓ EPA and DHA levels in milk can be increased without any adverse effects on its flavour by the incorporation of microencapsulated fish oils; the flavor of the milk may get some sort of fishy flavor.
  - ✓ Milk can be fortified with vitamin D entrapped in liposome.

- ✓ Microencapsulated iron ingredients can prevent off flavor development and maintain bioavailability.
- ✓ Probiotic bacterial cells encapsulated in calcium alginate are provided protection in fermented frozen dairy desserts.
- ✓ The objectionable tastes and aroma of popular nutritional ingredients like soy extracts, bitter herbs and  $\omega$ -3 oils, can be masked by microencapsulation.
- ✓ Microencapsulation of  $\beta$ -galactosidase in liposomes can be used to act in-vivo but protect from acting on lactose during storage.
- ✓ Ascorbic acid entrapped in liposome together with vitamin E is used for protection of emulsion-type foods.



THE ONLINE SOLUTION **swayam**  
विद्यया ऽर्द्धो ज्ञानेन चतुर्विधः

- ✓ Microencapsulated iron ingredients can prevent off flavor development and maintain bioavailability.
- ✓ Probiotic bacterial cells encapsulated in calcium alginate are provided protection in fermented frozen dairy desserts.
- ✓ The objectionable tastes and aroma of popular nutritional ingredients like soy extracts, bitter herbs and  $\omega$ -3 oils, can be masked by microencapsulation.
- ✓ Microencapsulation of  $\beta$ -galactosidase in liposomes can be used to act in-vivo but protect from acting on lactose during storage.
- ✓ Ascorbic acid entrapped in liposome together with vitamin E is used for protection of emulsion-type foods.

✓ Hygroscopic properties of materials like sodium chloride, etc. may be reduced by microencapsulation.

✓ Fluid-bed encapsulated salt is used in meats to prevent development of rancidity, as well as premature set due to myofibrillar binding.

✓ Among all existing microencapsulation methods, molecular inclusion of flavour volatiles in  $\beta$ -cyclodextrin molecules is the most effective method for protecting the aromas.

✓ Ascorbic acid is added to bread to improve its quality. The encapsulated form can protect this acid from water and oxygen in the bread which causes degradation.

- ✓ Hygroscopic properties of materials like sodium chloride, etc. may be reduced by microencapsulation.
- ✓ Fluid-bed encapsulated salt is used in meats to prevent development of rancidity, as well as premature set due to myofibrillar binding.
- ✓ Among all existing microencapsulation methods, molecular inclusion of flavour volatiles in  $\beta$ -cyclodextrin molecules is the most effective method for protecting the aromas.
- ✓ Ascorbic acid is added to bread to improve its quality. The encapsulated form can protect this acid from water and oxygen in the bread which causes degradation.

No.	Category of food ingredients	Examples	Preferred mode of encapsulation	Applications
1	Acidulants	Lactic acid, glucono- $\delta$ -lactone, vitamin C, acetic acid, potassium sorbate, sorbic acid, calcium propionate, and sodium chloride	Fluidized-bed coating, extrusion	<ol style="list-style-type: none"> <li>Used to assist in the development of color and flavor in meat emulsions, dry sausage products, uncooked processed meats, and meat containing products.</li> <li>Baking industry use stable acids and baking soda in wet and dry mixes to control the release of carbon dioxide during processing and subsequent baking.</li> </ol>
2	Flavoring agents	Citrus oil, mint oils, onion oils, garlic oils, spice oleoresins	Inclusion complexation, extrusion, centrifugal extrusion, spray-drying	<ol style="list-style-type: none"> <li>To transform liquid flavorings into stable and free flowing powders, which are easier to handle and incorporate into a dry food system.</li> </ol>

**Encapsulated food ingredients and their application in food industry**



The category of various food ingredients suitable or preferred methods of their encapsulation and their use in different food processes are shown in table. For example the preferred method for encapsulation of acidulants like lactic acid, vitamin C, acetic acid or such other acidulants or the fluidized bread coating or extrusion is tabulated. The coated acidulants are used to assist in the development of color and flavor in meat emulsions, dry sausage products, meat containing products, etc. The baking industry use stable acids and baking soda in wet and dry mixes to control the release of carbon dioxide during processing and subsequent baking.

The acidulants and other ingredients; flavoring ingredients like, citrus oil, milt oils, onion oil, garlic oil, etc.; the preferred method for their encapsulation is inclusion complexation, extrusion, centrifugal extrusion, spray drying, etc. And these are used to transform liquid flavorings into stable and free flowing powders which are easier to handle and incorporate in a dry food system.



3	Sweeteners	Sugars, nutritive or artificial sugars: aspartame	Cocrystallization, fluidized-bed coating	1. To reduce the hygroscopicity, improve flowability, and prolong sweetness perception.
4	Colorants	Annatto, $\beta$ -carotene, turmeric	Extrusion, emulsion	1. Encapsulated colors are easier to handle and offer improved solubility, stability to oxidation, and control over stratification from dry blends.
5	Lipids	Fish oil, linolenic acid, rice brain oil, egg white powder, sardine oil, palmitic acid, seal blubber oil	Spray-drying, freeze-drying, vacuum-drying	1. To prevent oxidative degradation during processing and storage.
6	Vitamins and minerals	Fat-soluble: vitamin A, D, E, and K. Water-soluble: vitamin C, vitamin B <sub>1</sub> , vitamin B <sub>2</sub> , vitamin B <sub>6</sub> , vitamin B <sub>12</sub> , niacin, folic acid	Coacervation, inclusion complexation, spray-drying, liposome entrapment	1. To reduce off-flavors. 2. To permit time-release of nutrients. 3. To enhance the stability to extremes in temperature and moisture. 4. To reduce each nutrient interaction other ingredients.
7	Enzymes and microorganisms	Lipase, invertase, <i>Brevibacterium linens</i> , <i>Penicillium roqueforti</i>	Coacervation, spray method, liposome entrapment	1. To improve the stability. 2. To reduce the ripening time.

Sweeteners like, sugars, nutritive or artificial sugars like aspartame etc. are generally microencapsulated using co crystallization or fluidized bed coating processes and the microencapsulated forms of these sweeteners are used to reduce the hygroscopicity to improve flowability as well as to prolong sweetness perception.

Colorants like annatto, beta, carotene, turmeric etc. are microencapsulated using extrusion or emulsion methods and their encapsulated colors are easier to handle and offer improved solubility and stability to oxidation and control over stratification from dry blends. Lipids like, fish oil, linolenic acid, rice brain oil, egg white powders etc., are spray dried or freeze dried or vaccum dried to encapsulate them. And these encapsulated forms are used to prevent oxidative degradation during processing and storage. Similarly vitamins and minerals are encapsulated using coacervation process or inclusion complexation, spray drying and such other methods.

The microencapsulated vitamins and minerals like, vitamin fat soluble vitamins or water soluble vitamins and niacin, folic acids etc., are used to reduce off flavors to permit time release of nutrients to enhance the stability to extreme temperature and moisture. They are also used to reduce the nutrients interactions with other components or other ingredients. Enzymes and microorganisms are microencapsulated generally by coacervation method or spray drying method and these are used to improve the stability or to reduce the ripening times.

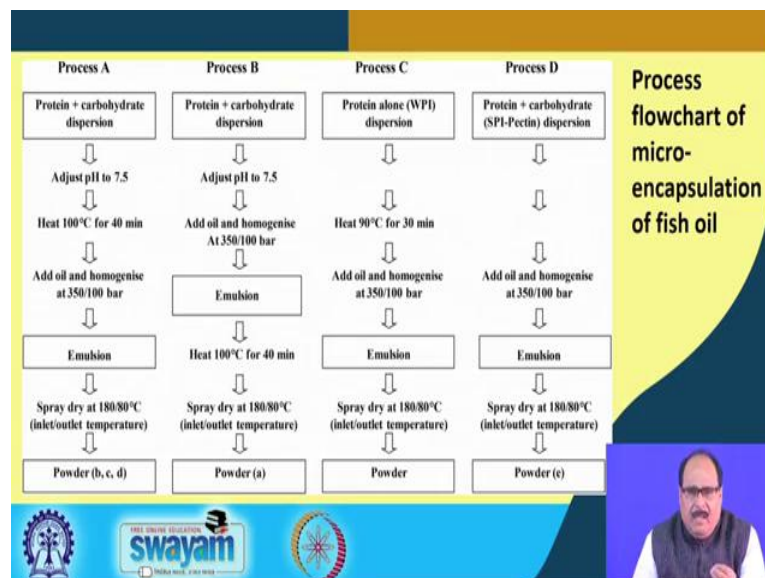
### Microencapsulation of fish oil

- Fish oil powder is produced by microencapsulating fish oil with micellar casein in the form of SMP using homogenization and spray drying.
- It had acceptable taste and modest shelf life of 3 weeks at 4 °C.
- It is successfully incorporated into number of food products including infant foods.



### Microencapsulation of fish oil

- Fish oil powder is produced by microencapsulating fish oil with micellar casein in the form of SMP using homogenization and spray drying. It is easier to be used in different products.
- It had acceptable taste and modest shelf life of 3 weeks at 4 °C.
- It is successfully incorporated into number of food products including infant foods.



In this slide, the process flow chart for different methods of microencapsulation of fish oil is presented which can be read and understood.

### Microencapsulation of polyphenols

- Polyphenols from natural products such as grape seed, soybean, oak, etc. are extracted using standard methods.
- The phenolic extracts are then mixed with wall materials like polysaccharides, proteins, gums to form a solution.
- The solution is then spray dried to form microcapsules.

THE OPEN SOURCE Swayam

### Microencapsulation of polyphenols

The polyphenols have very good antioxidant properties, functionality and in many foods many a time they are required to be added. But most of the polyphenols have bitter taste, so, when added to the food materials etc., they impart sensory profile. So, these polyphenols if they are encapsulated and then used in the encapsulated forms, then their interaction with other food components can be avoided.

From the natural products like grape seed, soybean, oak etc., the polyphenols may be extracted using standard methods and then these extracted polyphenols are mixed with wall materials like polysaccharides, proteins, gums etc., to form solution. The solution is then spray dried to form microcapsules.

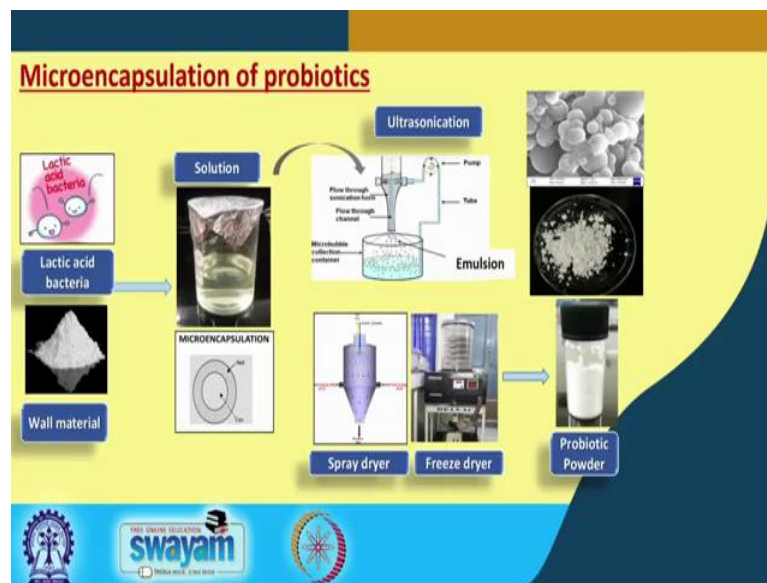
### Microencapsulation of high PUFA containing edible oils

THE OPEN SOURCE Swayam

## Microencapsulation of high PUFA containing edible oils

In our lab, some work in microencapsulation of edible oils has been done; different oil blends like, sunflower oil and sesame oil in different proportions were taken and conditions were optimized to have high content of PUFA as well as more antioxidants. Oils alone or in combination (see picture) were emulsified with the wall materials and with suitable aqueous medium, then oil-in-water emulsion was formed. This oil and water emulsion was dried using spray drying as well as microwave drying and good quality free flowing oil powder was obtained.

Spray drying yielded a good free flowing powder and the microwave drying also was found to be equally better process. The oil entrapped in the microencapsules was comparatively found more in the microwave drying process than that of the spray drying process.



## Microencapsulation of probiotics

Lactic acid bacteria are mixed with suitable wall materials i.e. the sources of the wall materials may be carbohydrate sources, maltodextrin or some protein sources. Aqueous medium is used for their emulsification and 2 methods are used; one is spray drying and another freeze drying; probiotic powder is obtained.

### Mechanism of controlled release of ingredients

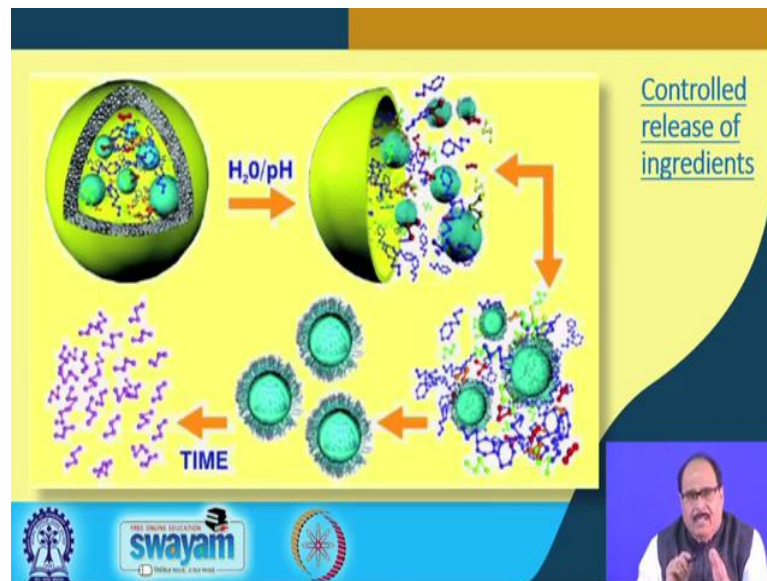
- The aim of a microencapsulation application is the isolation of the core from its surrounding, the wall must be ruptured at the time of use.
- Variety of release mechanisms have been proposed for microcapsules.
  - ✓ Pressure or shear stress
  - ✓ Melting the wall
  - ✓ Dissolving it under particular conditions, as in the case of an enteric drug coating
  - ✓ Solvent action
  - ✓ Enzyme attack
  - ✓ Chemical reaction
  - ✓ Hydrolysis or slow disintegration



### **Mechanism of controlled release of ingredients**

The bio actives or ingredients are coated with certain materials to provide them stability. Before they are used actually in the food system it has to be ensured that, this coating material is disintegrated or ruptured at the time of its use. So, variety of release mechanism or variety of methods have been proposed for breaking or rupturing of the wall of the microcapsules and they include pressure or shear stress, melting the wall by suitable method by thermal or such other methods, dissolving it under particular conditions as in the case of an enteric drug coating, solvent action enzyme attack, chemical reaction, hydrolysis or slow disintegration. These methods depending upon type of the micro capsule, wall material, core material, its sensitivity, its strength etc., either of these processes can be used to release the bioactives.





### Controlled release of ingredients

Some bio-actives are encapsulated and there is a hard rigid cell outside. There is a water or acidic condition etc., there may be water soluble material they can be dissolved in this acidic medium. The wall material is dissolved and finally, the active ingredients are released.

Advantages of microencapsulation technology

- Handling and flow properties can be improved by converting liquid to a solid encapsulated form.
- Hygroscopic materials can be protected from moisture.
- Various properties of active agents may be changed by encapsulation.
- Studies on the macrophage uptake of microspheres have demonstrated their potential in targeting drugs to pathogens residing intracellularly.
- Reliable means to deliver the drug to the target site with specificity.

### Advantages of microencapsulation technology

- Handling and flow properties can be improved by converting liquid to a solid encapsulated form.
- Hygroscopic materials can be protected from moisture.

- Various properties of active agents may be changed by encapsulation.
- Studies on the macrophage uptake of microspheres have demonstrated their potential in targeting drugs to pathogens residing intracellularly.
- Reliable means to deliver the drug to the target site with specificity.

The conversion of bio actives from liquid form to solid form improve their restorability, usability; even hygroscopic materials can be protected from moisture after being encapsulated. Various properties of active agents may be changed by microencapsulation. And studies on the macrophage uptake of microspheres have demonstrated their potential in targeting drugs to pathogens residing intracellularly. This is a reliable means to deliver the drug to the target site with specificity.



- The desired concentration can be maintained at the site of interest without untoward effects.
- The development of cyclodextrins has been a major breakthrough in microencapsulation technology; it provided good wall material for improving shelf-life, reducing volatility and protection of heat-labile substances.
- Due to encapsulation flavours are more stable after processing with microwave, heat, oven drying and frying.
- Liposomes have many benefits for the food industry including protection of materials until desired release or targeted delivery.

So, in nut shell, this microencapsulation technology has a wide ranging application in food processing industries. It is a very good and efficient method for improving the food functionality, characteristics and their usability.