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**MICROENCAPSULATION TECHNIQUES APPLICABLE TO FOOD FLAVOURS
RESEARCH AND DEVELOPMENT: A COMPREHENSIVE REVIEW****Vedpal Yadav^{1*}, Alka Sharma² and S K Singh³**

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Received on: 1st October, 2014Accepted on: 23rd May, 2015**ABSTRACT**

This systematic review sought to determine the current state of the literature on the microencapsulation of flavours. Different wall matrix materials and flavours encapsulated by different techniques are discussed in detail. Though no classification of methods used for microencapsulation is perfect even then an effort has been made to classify the methods between physical processes and chemical processes. The advantages and disadvantages of both types of methods is being highlighted. A future application of microencapsulation process is highlighted for various flavours.

Keywords: Microencapsulation; Microcapsule; Encapsulation; Flavours; Coacervation**INTRODUCTION****METHODS AND DISCUSSION****FLAVOUR**

Flavour is the combination of odor and taste. Some describe total flavor as including textural (temperature, coolness, etc.) attributes, color, and sound, in other words, the total sensory experience (The Dictionary of Flavors, 1998) Flavor is the sensation produced by a material in the mouth, perceived principally by the senses of taste and smell, and also by the general tactile and temperature receptors in the mouth. Flavor also denotes the sum of the characteristics of the material which produces that sensation (Min,2010).

MICROENCAPSULATION

Microencapsulation is defined as the process of surrounding or enveloping one substance within another substance on a very small scale, yielding capsules ranging from less than one micron to several hundred microns in size. Microcapsules may be spherically shaped, with a continuous wall surrounding the core, while others are asymmetrically and variably shaped, with a quantity of smaller droplets of core material embedded throughout the microcapsule. All three states of matter (solids, liquids, and gases) may be microencapsulated. This allows liquid and gas phase materials to be handled more easily as solids. There are almost limitless applications for

microencapsulated material (Bokovic,1996, Cocero et al,2009 Gibbset al,1999, Malay et al,2007, Pather et al,2008, Tan,2005). Although no officially approved definition of a microcapsule exists, most workers use the term microcapsule to describe particles with diameters between 1 and 1000 mm that contain a desired ingredient of some sort. Particles smaller than 1 mm are called nanoparticles; particles greater than 1000 mm can be called microgranules or macrocapsules. The microencapsulated product can be delivered as a: dry, free-flowing powder, as slurry, or in the form of a wet filter cake. For larger particle sizes there are granules and beads (Microtek Laboratories,2011)

PURPOSE OF MICROENCAPSULATION

Purpose of microencapsulation is to:

IMMOBILIZE OR ENTRAP- limit contact between parts of a system till the time, when the microcapsule is ruptured.**PROTECTION-** Natural ingredients, essential oils, vitamins and supplements can be encapsulated to protect them from the deteriorating effects of the environment or for optimal release and performance in the final product (Microtek Laboratories,2012). Fragile ingredients, such as many vitamins, unsaturated lipids or phytochemicals, may need protection from oxygen, light or other environmental insults to prevent degradation. Probiotics need protection

from digestive acids and enzymes in order to reach the colon unharmed to colonize there. Many industrial enzymes are sold in encapsulated form to avoid allergic reactions in the workplaces using them.

CONTROLLED RELEASE- Allows pharmaceuticals to be released in serum or tissues with predefined kinetics. Can speed or limit release of entrapped actives, be they pharmaceuticals, nutrients, flavors or aromas. Encapsulation can retard evaporation of volatile ingredients such as fragrance oils. This can dramatically increase the useful life of fragrance-based products such as room freshener sprays, shampoos, deodorizing sprays, etc.

ENHANCE MIXING AND FLOW- Allows homogeneous incorporation of a small amount of liquid into a large volume of dry material and coats sticky materials to create free flowing powders (such as encapsulated honey or other syrups).

CREATE NEW INGREDIENT FUNCTIONS AND BEHAVIOR- Regulating catalytic or other chemical activity by controlling membrane or microcapsule permeability (Arshady et al,1993, Poncelet,2007, Wrick,2008)

MASKING- Encapsulation can help mask odors (9).

IMPROVED HANDLING PROPERTIES- Emollients used in skincare and hair care products can be encapsulated for added product features and to improve handling properties. Sticky components of products or difficult to handle ingredients can be encapsulated to improve handling properties.

EXTENSION OF THE PRODUCT'S USEFUL LIFE AND EFFECTIVENESS- Active ingredients such as insect and animal repellents can be encapsulated to allow for slow diffusion from the microcapsule to extend the product's useful life and effectiveness.

PRODUCT DIFFERENTIATION- From a marketing standpoint, encapsulation offers advantages such as allowing for product differentiation, creation of "new and improved" and/or premium products, and generation of entirely new product lines.

Microencapsulation of key ingredients in personal care products can dramatically extend their effectiveness and greatly improve their handling properties. With a variety of encapsulation release technologies available, including temperature, mechanical rupture (rubbing, pressure, impact), controlled release, or no release at all - the possibilities are endless(Microtek Laboratories,2012)

Key drivers of food microencapsulation market dynamics are increased shelf life, retention or masking of taste and color, enhanced bio availability, controlled release function, emergence of functional/ nutrition foods, children's foods market.

Microcapsules can be classified on three basic categories according to their morphology as follows:

- 1- Mononuclear (core-shell) microcapsules contain the shell around the core.
- 2- Polynuclear capsules have many cores enclosed within the shell.
- 3- Matrix encapsulation in which the core material is distributed homogeneously into the shell material.

In addition to these three basic morphologies, microcapsules can also be mononuclear with multiple shells, or they may form clusters of microcapsules (13).

MICROENCAPSULATION PROCESS

Microencapsulation involves basically three things:

1. Material to be encapsulated or core.
2. Material in which to be encapsulated or wall matrix material.
3. Microencapsulation techniques to be employed.

MATERIAL TO BE ENCAPSULATED OR CORE

Many terms have been used to describe the contents of a microcapsule: active agent, actives, core material, fill, internal phase, nucleus, and payload(Hui,2003).Core materials include flavours, antimicrobial agents, nutraceutical and therapeutic actives, vitamins, minerals, antioxidants, colours, acids, alkalis, buffers, sweeteners, nutrients, enzymes, cross-linking agents, yeasts, chemical leavening agents, and so on(Lakkis,2007).Most liquid food flavorings are volatile and chemically unstable in presence of air, light, moisture and light temperatures. Microencapsulation has become an attractive approach to transform liquid food flavorings into stable and free flowing powders which are easy to handle and incorporate into a dry food system. The diluted state is also more convenient to mix the flavor materials normally with solid foods(Bhandari et al 1992). Volatile products, citral and linalyl acetate have been successfully microencapsulated using spray drying (Bhandari et al 1992). Mint oil (Zhong et al,2009), Polyunsaturated Fatty Acids (PUFA)(Serfert,2009) is used in some studies as core material. Limonene and menthol powder (Segolene et.al., 2009), Lemongrass essential oil (Leimann et.al., 2009) and Medium-chain triglyceride oil (MCT, Lumulse CC-33K) (Leclercq et.al., 2009) are microencapsulated and studied for their efficacy.

MATERIAL IN WHICH TO BE ENCAPSULATED OR WALL MATRIX MATERIAL

Many terms have also been used to describe the material from which capsules are formed: carrier, coating, membrane, shell, or wall (Encyclopedia of Food Sciences and Nutrition, 2003). Proteins, polysaccharides and semi synthetic cellulose derivatives are widely used biopolymers used in food emulsions to control their texture, microstructure and stability.

The coating material should be capable of forming a film that is cohesive with the core material; be chemically compatible and nonreactive with the core material; and provide the desired coating properties, such as strength, flexibility, impermeability, optical properties, and stability. The coating materials used in micro encapsulation methods are amenable, to some extent, to in situ modification (Bandhavi et.al., 2013).

The typical coating properties such as cohesiveness, permeability, moisture sorption, solubility, stability and clarity must be considered in the selection of the proper microcapsule coating material.

Coating material properties are:

- Stabilization of core material.
- Inert toward active ingredients.
- Controlled release under specific conditions.
- Film-forming, pliable, tasteless, stable.
- Non-hygroscopic, no high viscosity, economical.
- Soluble in an aqueous media or solvent, or melting.
- The coating can be flexible, brittle, hard, thin etc.

Examples of coating materials:

- Water soluble resins: Gelatin, Gum Arabic, Starch, Polyvinylpyrrolidone, Carboxymethylcellulose, Hydroxyethylcellulose, Methylcellulose, Arabinogalactan, Polyvinyl alcohol, Polyacrylic acid
- Water insoluble resins: Ethylcellulose, Polyethylene, Polymethacrylate, Polyamide (Nylon), Poly (Ethylene Vinyl acetate), cellulose nitrate, Silicones, Poly lactideco glycolide.
- Waxes and lipids: Paraffin, Carnauba, Spermaceti, Beeswax, Stearic acid, Stearyl alcohol, Glyceryl stearates.
- Enteric resins: Shellac, Cellulose acetate phthalate, Zein.

Polysaccharides are mainly added to enhance the viscosity and to stabilise the system, while proteins can form networks and have emulsification and foaming properties (Moschakis et.al., 2010; Dickinson, 2003).

Powdered WPI Bipro®, Sodium azide and chitosan are used as wall material for encapsulation successfully (Moschakis et.al., 2010). Gelatin 250 Bloom strength, 20 mesh, type A, provided by PB-Leiner (Davenport, IA, USA) and gum acacia (Acacia seyal, FT powder, TIC Gums, Belcamp, MD, USA) were used as wall materials in coacervation (19). Waxy maize starches (24), soybean protein isolate (SPI) (25) and soybean protein isolate (SPI)/pectin (Mendanha et.al., 2009) were used as wall materials in the formation of microcapsules.

In developing or modifying an encapsulation system, it is very helpful to look at the available wall materials that one may have for use. The following materials should be useful in developing a microencapsulation system using coacervation: Acacia (Gum Arabic), Carob Bean Gum, Carrageenan, Ethyl cellulose, Food Starch (Modified), Guar Gum, Hydroxypropyl Cellulose, Hydroxypropyl Methyl cellulose, Locust Bean Gum, Methyl cellulose, Methyl Ethyl Cellulose, Potassium Alginate, Potassium Citrate, Potassium Polymetaphosphate, Shellac (Bleached), Sodium Alginate, Sodium Carboxymethylcellulose, Sodium Citrate, Sodium Polyphosphates (Glassy), Sodium Trimetaphosphate, Tragacanth, White Shellac, Xanthan Gum, etc. (Versic, 2010).

MICROENCAPSULATION TECHNIQUES TO BE EMPLOYED

Encapsulation of food ingredients into coating materials can be achieved by several methods. The selection of the microencapsulation process is governed by the properties (physical and chemical) of core and coating materials and the intended application of food ingredients (Desai, 2010; Barbosa et.al., 2005)(28, 29).

Many microencapsulation processes exist. Some are based exclusively on physical phenomena. Some utilize polymerization reactions to produce a capsule shell. Others combine physical and chemical phenomena. Because there are so many encapsulation techniques, it is logical to make an effort to attempt to categorize or classify them in some manner, thereby providing a means of identifying the concepts on which various encapsulation technologies are based (Encyclopedia of Food Sciences and Nutrition, 2003).

Microencapsulation processes are usually categorized into two groupings: chemical processes and mechanical or physical processes. These labels can, however, be somewhat misleading, as some processes classified as mechanical might involve or even rely upon a chemical reaction, and some chemical techniques rely solely on physical events. A clearer indication as to which category an encapsulation method belongs is whether or not the capsules are produced in a tank or reactor containing liquid, as in chemical processes, as opposed to mechanical or physical processes, which employ a gas phase as part of the encapsulation and rely chiefly on commercially available devices and equipment to generate microcapsules.

CHEMICAL METHODS

COACERVATION OR COMPLEX COACERVATION

Capsules for carbonless paper and for many other applications are produced by a chemical technique called complex coacervation. This method of encapsulation takes advantage of the reaction of aqueous solutions of cationic and anionic polymers such as gelatin and gum arabic. The polymers form a concentrated phase called the complex coacervate. The coacervate exists in equilibrium with a dilute supernatant phase. As water-immiscible core material is introduced into the system, thin films of the polymer coacervate coat the dispersed droplets of core material. The thin films are then solidified to make the capsules harvestable.

INTERFACIAL POLYMERIZATION (IFP)

Interfacial polymerization (IFP) is another chemical method of microencapsulation. This technique is characterized by wall formation via the rapid polymerization of monomers at the surface of the droplets or particles of dispersed core material. A multifunctional monomer is dissolved in the core material, and this solution is dispersed in an aqueous phase. A reactant to the monomer is added to the aqueous phase, and polymerization quickly ensues at the surfaces of the core droplets, forming the capsule walls. IFP can be used to prepare bigger microcapsules, but most commercial IFP processes produce smaller capsules in the 20-30 micron

diameter range for herbicides and pesticide uses, or even smaller 3-6 micron diameter range for carbonless paper ink.

POLYMER-POLYMER INCOMPATIBILITY OR PHASE SEPARATION

Polymer-polymer incompatibility, also called phase separation, is generally grouped with other chemical encapsulation techniques, despite the fact that usually no actual chemical reaction is involved in the process. This method utilizes two polymers that are soluble in a common solvent; yet do not mix with one another in the solution. The polymers form two separate phases, one rich in the polymer intended to form the capsule walls, the other rich in the incompatible polymer meant to induce the separation of the two phases.

The second polymer is not intended to be part of the finished microcapsule wall, although some may be caught inside the capsule shell and remain as an impurity.

IN SITU POLYMERIZATION

In situ polymerization is a chemical encapsulation technique very similar to interfacial polymerization. The distinguishing characteristic of *in situ* polymerization is that no reactants are included in the core material. All polymerization occurs in the continuous phase, rather than on both sides of the interface between the continuous phase and the core material, as in IFP.

Examples of this method include urea-formaldehyde (UF) and melamine formaldehyde (MF) encapsulation systems.

CENTRIFUGAL FORCE PROCESSES

Centrifugal force processes were developed in the 1940s to encapsulate fish oils and vitamins, protecting them from oxidation. In this method an oil and water emulsion is extruded through small holes in a cup rotating within an oil bath. The aqueous portion of the emulsion is rich in a water-soluble polymer, such as gelatin, that gels when cooled. The resulting droplets are cooled to form gelled polymer-matrix beads containing dispersed droplets of oil that are dried to isolate.

SUBMERGED NOZZLE PROCESSES

Similar in concept to centrifugal force processes, submerged nozzle processes produce microcapsules when the oil core material is extruded with gelatin through a two-fluid nozzle. The oil droplets are enveloped in gelatin as they are extruded through the nozzle. Then the capsules are cooled to gel the walls, before being collected and dried.

MECHANICAL OR PHYSICAL METHODS

SPRAY DRYING

Spray drying is a mechanical microencapsulation method developed in the 1930s. An emulsion is prepared by dispersing the core material, usually an oil or active ingredient immiscible with water; into a concentrated solution of wall material until the desired size of oil droplets are attained. The resultant emulsion is atomized into a spray of droplets by pumping the slurry through a

rotating disc into the heated compartment of a spray drier. There the water portion of the emulsion is evaporated, yielding dried capsules of variable shape containing scattered drops of core material. The capsules are collected through continuous discharge from the spray drying chamber. This method can also be used to dry small microencapsulated materials from aqueous slurry that are produced by chemical methods.

SPRAY CHILLING

Spray chilling, cooling, or congealing are variations of conventional spray-drying. In these cases, chilled air is used to solidify molten capsule shell material formulations rather than volatilize a solvent. Various fats, waxes, fatty alcohols, fatty acids, or combinations of these materials are the shell materials used. In such encapsulation procedures, the active agent is dispersed in a molten shell material with the aid of an emulsifier, if necessary. The resulting dispersion is atomized through heated nozzles into a cooling chamber analogous to that used in a spray drier. The shell material is solidified by cooling, thereby producing solid particles (Encyclopedia of Food Sciences and Nutrition, 2003).

PAN COATING

In pan coating, solid particles are mixed with a dry coating material and the temperature is raised so that the coating material melts and encloses the core particles, and then is solidified by cooling; or, the coating material can be gradually applied to core particles tumbling in a vessel rather than being wholly mixed with the core particles from the start of encapsulation.

FLUID BED COATING OR FLUIDIZED BED COATING

Fluid bed coating, another mechanical encapsulation method, is restricted to encapsulation of solid core materials, including liquids absorbed into porous solids. This technique is used extensively to encapsulate pharmaceuticals. Solid particles to be encapsulated are suspended on a jet of air and then covered by a spray of liquid coating material. The capsules are then moved to an area where their shells are solidified by cooling or solvent vaporization. The process of suspending, spraying, and cooling is repeated until the capsules' walls are of the desired thickness.

WURSTER PROCESS

The fluid bed coating or fluidized bed coating process is known as the Wurster process when the spray nozzle is located at the bottom of the fluidized bed of particles. Both fluidized bed coating and the Wurster process are variations of the pan coating method.

CENTRIFUGAL EXTRUSION PROCESS

Centrifugal extrusion processes generally produce capsules of a larger size, from 250 microns up to a few millimeters in diameter. The core and the shell materials, which should be immiscible with one another, are pushed through a spinning two-fluid nozzle. This movement forms an unbroken rope which naturally splits into round droplets

directly after clearing the nozzle. The continuous walls of these droplets are solidified either by cooling or by a gelling bath, depending on the composition and properties of the coating material.

DESOLVATION OR LIQUID EXTRACTION ENCAPSULATION TECHNOLOGY

Desolvation encapsulation processes consist of dissolving a shell formulation in a finite amount of water, thereby forming a concentrated solution of shell material. Core material is dispersed in this solution with the aid of a surfactant. The dispersion produced can be extruded or atomized directly into a desolvation bath that solidifies the shell formulation by extracting the solvent used to dissolve the shell material from the spray droplets. Alternately, the core material/shell dispersion can be extruded or atomized into a vessel that contains excess spinning solvent. The spinning bath produces droplets of core/shell formulation that are subsequently solidified in a desolvation bath (14).

MELT EXTRUSION

A melt extrusion encapsulation process involves dispersing a core material in a molten shell formulation at 85-125°C with or without the aid of a surfactant. Once formed, the dispersion is extruded as filaments into a relatively cool environment that solidifies the extruded mass. The receiving environment can be a gas phase or a tank that contains a suitable solvent. If extruded into a gas phase, the cooled mass is simply broken up into particles and used. If extruded into a solvent, the solvent simultaneously cools and removes unencapsulated or free core material from the filaments. The solidified product is subsequently dried and broken up to yield particles with the multinuclear structure. The particles are glass matrices loaded with dispersed core material. Flavour loaded particles produced in this manner have an excellent resistance to oxidation during storage (Encyclopedia of Food Sciences and Nutrition, 2003).

SUSPENDED NOZZLES

A number of workers have produced microcapsules by ejecting droplets that contain a core and shell material from suspended nozzles into a gas phase, usually air. The droplets pass through a gas phase until the capsule shell is solidified by cooling, or they fall into a curing bath where they are gelled and subsequently harvested (Encyclopedia of Food Sciences and Nutrition, 2003).

ROTATIONAL SUSPENSION SEPARATION OR SPINNING DISK METHOD

Another mechanical encapsulation process is rotational suspension separation, or the spinning disk method. The internal phase is dispersed into the liquid wall material and the mixture is advanced onto a turning disk. Droplets of pure shell material are thrown off of the rim of the disk along with discrete particles of core material enclosed in a skin of shell material. After having been solidified by cooling, the microcapsules are collected separately from the particles of shell material.

The range of applicability using the encapsulation methods described above is extremely wide, and tends to

overlap (Barbosa et.al., 2005; Pothakamury, 1995 and Vilstrup, 2001). For instance, one may exclude certain processes and favor others based on the characteristics of the material to be encapsulated, release methods and patterns, health and safety issues, or economic concerns (Barbosa et.al., 2005 and Poncelet, 2006).

RELEASE MECHANISM

In most cases, food components concentrated inside microcapsules are released as the food is consumed or during a food preparation step. Release is achieved by destroying the integrity of the microcapsule shell. This is done by dissolving it in water, melting it, or mechanically rupturing it. There are cases where no release of core material is desired until after the food has been ingested and is present in the digestive system. Perhaps, the intent is for the core material carried by a microcapsule never to be released. In these latter situations, the capsule shell must remain intact throughout the food preparation and ingestion steps (14).

CONCLUSION

Although all the techniques discussed have some advantages over the other techniques even then categorical the most appropriate technologies are adopted commercially like spray drying to the fullest. For maximum loading of flavours complex coacervation scored best but commercially it has not been widely accepted as chemicals and time involved are more. There is ample scope of using two or three different techniques in complementation to each other as per the requirement and convenience and a lot of work needs to be done on different combinations of newly developed and traditional wall matrix materials.

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