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Core-Shell Micro/Nanocapsules: From Encapsulation to Applications

Eslam Elkalla ^a, Sumera Khizar ^a, Mohamad Tarhini ^a, Nouredine Lebaz ^b,
Nadia Zine^a, Nicole Jaffrezic-Renault^a, Abdelhamid Errachid ^a, Abdelhamid
Elaissari^a,*

^a *Univ Lyon, University Claude Bernard Lyon-1, CNRS, ISA-UMR 5280, F-69622 Lyon, France*; ^b *Univ Lyon, University Claude Bernard Lyon-1, CNRS, LAGEPP UMR-5007, Villeurbanne, France*

*Corresponding author. ISA, University Claude Bernard Lyon-1, 5 rue de La Doua, Villeurbanne 69100, France. Email address: abdelhamid.elaissari@univ-lyon1.fr

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ABSTRACT

Encapsulation is the way to wrap or coat one substance as a core inside another tiny substance known as a shell at micro and nano scale for protecting the active ingredients from the exterior environment. A lot of active substances such as flavors, enzymes, drugs, pesticides, vitamins, in addition to catalysts being effectively encapsulated within capsules consisting of different natural as well as synthetic polymers comprising poly(methacrylate), poly(ethylene glycol), cellulose, poly(lactide), poly(styrene), gelatin, poly(lactide-co-glycolide)s, and acacia. The developed capsules release the enclosed substance conveniently and in time through numerous mechanisms, reliant on ultimate use of final products. Such technology is important for a number of fields counting food, pharmaceutical, cosmetics, agriculture, and textile industries. The present review focuses on the most important and high-efficiency methods for manufacturing micro/nanocapsules and their several applications in our life.

Keywords: encapsulation; micro/nanocapsules; pharmacy; textile; cosmetics; agriculture

Introduction

Recently the advancement of multifunctional polymer-based micro/nanocapsules with controlled physical and chemical properties is center for an immense investigation in life as well as material sciences. Particularly in the biomedical field, the design of innovative polymeric composites with organic/inorganic materials as micro/nanocapsules for early diagnosis in addition to active therapeutic delivery that indicate most progressing zones in science (Deng et al., 2020). Microcapsules are particles with dimension in between 0.1 μm and 100 μm), whereas nanocapsules have diameter between 1nm and 100nm, comprising of solid or liquid core along with a polymer shell. Incredible research about synthesis of distinct micro/nanocapsules has been made aimed at obtaining preferred features, for instance shape, size, composition, and functionalization (Musyanovych and Landfester, 2014).

New developments concerning micro/nanocapsules having the ability to carry and release susceptible constituents become a significant issue in pharmacological

industries, cosmetics, food along with agriculture, marine, and chemical industry (Sun et al., 2018). A lot of work has been done to protect enclosed molecules improving their constancy, attaining higher efficacy, as well as increase bioavailability (Ozturk et al., 2015). Encapsulation of liquids allows fabrication of liquid droplets in the core surrounded by a tiny material sphere (the shell) with specific geometry, size, kinetic and physical properties which lead to producing capsules. There are three types of encapsulation that have been reported extensively (Trojanowska et al., 2017). In brief, the ideal definition of the encapsulation process is the entrapment of a liquid form inside a dispersed material for its conservation, immobilization, protection, controlled release, structuration, and functionalization (Su et al., 2015). During their synthesis, different methods are used (for instance ionic gelation, polymerization, coacervation, and dispersal of preformed polymers) influencing their absolute characteristics (Bielski et al., 2020).

Approaches based on emulsion are extensively considered to encapsulate payloads inside capsules (Koroleva et al., 2016). There are several benefits of encapsulation of liquids. The first advantage is the isolation and protection of the incompatible materials: cover and isolates the liquid core from the surrounding environment and external harsh effects; e.g. in the stomach and digestive system for protection from enzymes effects, prevent the effect of oxidation, moisture especially in the food industry, etc. (Vijeth et al., 2019). Another advantage is increasing the interval period to protect the liquid which is encapsulated until achieving the appropriate conditions. Moreover, transforming liquid forms to solid ones can be achieved simply by moving in the surface area without direct contact with the surrounding effects which can induce a negative effect on the targeted function of this liquid (Bielski et al., 2020). Furthermore, encapsulation leads to enhancing the activity and stability of the encapsulated material and protecting it from oxidation or suppression due to any possible reaction with the environment around. It also leads to masking and covering the odor and taste. Finally, a controlled sustained release of the active materials can be achieved which regulates the effect of the encapsulated liquid (Water, Oil) (Jiang et al., 2020).

Above the aforementioned benefits, polymer micro/nanocapsules gained attention besides possessing great potential for biological application in agriculture, delivery of drug, textile, plus food industries. In the present review, we focus on the state of art for the encapsulation of liquids through different methods from the oldest

one to the more recent one, and more effective and accurate method to produce capsules of liquids with controlled size and properties, including their possible usages in pharmacological, cosmetics, in addition to food productions. Micro/nanocapsules can be produced in many structural forms (Single-core, multi-core, multi-core, irregular) by different techniques described in this manuscript and have many utilizable characteristics for many applications. But herein we only focused on core-shell morphology of micro/nanocapsules. The foremost benefits of this morphology over other morphologies are (a) shell decreases the reactivity of core, (b) thermal stability of the particles can easily be modified, (c) enhances the overall dispersibility and stability of core, (d) shell provide further opportunity to modify or functionalize surface of the nanoparticles, and (e) improves oxidative stability (Chen et al., 2020). Because of varied composition, modifiable performances, as well as synergistic impact among constituents, micro/nanocapsules with core-shell morphology are extensively used in various field recently. The core-shell micro/nanocapsules are of great interest because they exhibit exceptional properties rising from combining core and shell material and their geometry. Therefore, they have found wide-range applicability in fields such as biomedicine, electrical and semiconducting materials, and catalysts (Nomoev et al., 2015). Additionally among several morphologies, core-shell nanoparticles are most promising and appropriate for biomedical applications for instance bioimaging, drug delivery, gene delivery, and sensors attributed to their morphology, better dispersibility, bio-functionalization, and optical property (Chatterjee et al., 2014). First, chemical, physico-chemical, and physic-mechanical methods to prepare capsules are described in this review. The second part provides information about the applications of these micro/nanocapsules in pharmacy, agriculture, food, and cosmetics industries. Finally, the challenges, as well as the future perspectives of micro/nanocapsules applications are highlighted.

Encapsulation technologies for the fabrication of micro/nanocapsules

Encapsulation being an active field of research developed roughly 65 years ago, it offers innovative technologies, fabrication routes, in addition pioneering applications (Miladi et al., 2016). The prepared capsules showed diverse morphologies depending on the fabrication material, though synthetic method also has substantial influence on ultimate product (Trojanowska et al., 2017). Generally, the encapsulation process depends on the formation of a stabilized emulsion system and this will be explained in detail in this review. These processes make use of hydrophobic and hydrophilic liquids e.g. volatile

compounds, like medications or fragrances that can be encapsulated for more protection and conservation for applications that need sustained release effect, and DNA, proteins, peptides, and other active compounds to target specific sites in the body (Suganya and Anuradha, 2017). Encapsulation processes are classified by chemical, physico-chemical, and physico-mechanical methods presented in Figure 1 and described below (Trojanowska et al., 2017).

Chemical methods

These methods include the fabrication of a micro/nano sphere with several polymerization reactions. Monomers or prepolymers are the preparatory materials in these methods. Additional division of methods are described briefly below:

Interfacial polymerization

Mostly, it is also known as the interfacial polycondensation method used to generate capsules, ultrathin layers, and fibers. It involves a polymerization reaction between 2 immiscible dissimilar phases in liquid state (aqueous and oil phase). The capsule formation by this method can be achieved when the boundary between phases is droplet surface regarding emulsion. The emulsion is categorized as water-in-oil (w/o), oil-in-water (o/w), and oil-in-oil (o/o). A polycondensation of a hydrophilic monomer and a hydrophobic monomer occurs at the interface by a surface-active initiator that is dissolved in one of the phases (Raaijmakers and Benes, 2016). Figure 2 provides an example of an interfacial polymerization at the surface of an emulsion. The given method catches active components in microcapsules, with mean diameter varying from nano to some hundred microns.

Recently, it has many applications because of its higher yield, reasonable besides rapid synthesis, in addition to well-regulated release. It is direct and commonly used technique to produce capsules (Ricardo et al., 2021). Another benefit concerning this method is the production of homogeneous particles with easily controllable size. In addition, it allows customizability in the nature and dimensions of the shell that can offer high protection of the liquid core. Interfacial polymerization is a comparatively easy, flexible, and economical method; thus, it is an effective method for encapsulation. However, the procedure is still not understood well, for instance catalyst, temperature, surfactant, and active component impact is not clear (Mooranian et al., 2015). Encapsulation by this method is used in agriculture, pharmacy, cosmetics as well as in many other applications such as energy storage/transfer industries (Zhang and

Rochefort, 2012).

Primarily an impressive polymer collection have been investigated in this process that includes polyurethanes, polyamides, polyureas, polyanilines, polyimides, as well as polycarbonates (Michiel J.T.Raaijmakers, 2016). Micro/nanocapsules with various materials for shell and active components being explored in different applications are summarized in Table 1.

Recently microcapsules had been synthesized via this process involving methylene diphenyl diisocyanate and chitosan oligosaccharide (COS) soluble in oil and water, respectively. Results showed controlled release of material encapsulated as core. Hence, they represent favorable candidates to be used for precise release regarding growth regulators, pesticides, as well as fertilizer (Yu et al., 2021). Interfacial polymerization was used to prepare microcapsules of polyurea possessing oxyfluorfen herbicide. A reaction occurs between ethylene diamine and toluene diisocyanate. Capsules consuming N-N-dimethyldecanamide, a green solvent utilized as agents to emulsify and medium to carry in addition to high molecular weight lignin as core. They indicated uniform along with persistent release of lignin. Such capsules are safer for usage onto paddy crops (Rao et al., 2021). Inverse mini-emulsion was utilized to prepare cross-linked nanocapsules of starch encapsulating sulforhodamine 101, hydrophilic dye. Shell permeability was estimated via degradation of amylase (enzyme) through regulating chemical factors throughout crosslinking reaction (Steinmacher et al., 2017).

In-situ polymerization

It is an extensive reaction mixture that includes suspension, emulsion, and dispersion polymerization (Peng et al., 2020). These monomers/prepolymers exist in single phase, as compared to interfacial reaction in which each liquid phase comprises no less than 1 responsive monomer (Bakry et al., 2016a). Suspension, pearl or bead polymerization involves water insoluble monomer or monomers dispersed as droplets of liquid by means of steric stabilizers. The system is kept under vigorous stirring throughout reaction generating capsules of polymer as per discrete solid phase. Additionally, initiators solvable in liquid phase of monomer are employed in this process. However, as for emulsion polymerization, addition of monomer to solution containing core element with surfactant is drop-wise to fabricate micro/nanocapsules (Vauthier, 2016). In dispersion polymerization all initiator, monomer, also dispersant exist within one batch for producing nanocapsules with well-defined characteristics.

M-PCM (microencapsulated phase change material) was produced with mixture of eutectic and MF (melamine formaldehyde) as core and shell, respectively by this method. The developed microcapsules have uniform morphology of surface, sphere-shaped with a mean size of 10.41 μm (B et al., 2020). Similarly, M-PCMs sequences produced through in-situ polymerization by 1-dodecanol encapsulation in CNFs (cellulose nanofibers), modified with MF (melamine-formaldehyde) resins. They are favorable for thermo regulating uses (Wu et al., 2020). Reduction- reactive CPT (camptothecin) nanocapsules were developed through integrating in-situ polymerization with nanoprecipitation employing polymeric ligand and disulfide bond having crosslinker. These nanocapsules present an active intracellular drug delivery strategy for anticancer therapy (Song et al., 2018). A novel M-PCM, along organic composites as core and polymethylmethacrylate as shell, synthesized via this polymerization with 2–3 μm average diameter (Han et al., 2017).

Nanoprecipitation

It is also called as interfacial deposition or solvent displacement method. It is an effective technique for the fabrication of capsules that contain an aqueous core protected and coated with a polymeric shell (Miladi et al., 2016). In general, this process consists of dissolving the shell material, polymer for example, and a proper surfactant into the solvent that can be organic or aqueous (phase 1), then adding another phase that is miscible with phase 1, however it is bad solvent for polymer (phase 2). By mixing the two phases, the solubility of the polymer in the mixture will be reduced, leading to its precipitation in the form of particles under controlled agitation. This method was used for the encapsulation of various active molecules ranging from small molecules to biological molecules (Tarhini et al., 2020, Martinez Rivas et al., 2017). Indeed, this method was also used to enclose liquid core for instance essential oils (Lammari et al., 2020).

In case of liquid encapsulation, the polymer and active ingredient have been exclusively dispersed in organic phase, and not in aqueous phase (the non-solvent). Solvent is usually organic and non-solvent is water (Lammari et al., 2020). The continuous adding of oil phase to aqueous phase keeping magnetic stirring on, leads to polymer deposition on oil/water interface after transposition of organic solvent (Deng et al., 2020). Hydrophobicity of the oils prevents its dispersal from interior core to outside phase. Organic solvent can be eliminated by evaporating at appropriate temperature or by using rotavapor that produces the nanocapsules particles suspension in the water, as

the polymer is the shell that surrounds the core containing the oil.

Nanoprecipitation was used to prepare a polymeric nanosystem with insecticidal activity. Polycaprolactone (shell material), rosemary oil (insecticidal agent), and a non-ionic surfactant were dissolved into acetone (solvent phase). Then it is added to aqueous phase in a controlled manner, oil-loaded polymeric nanocapsules were obtained (Khoobdel et al., 2017). Similarly, a recent study shows the use of nanoprecipitation to produce palmarosa essential oil-loaded polycaprolactone nanocapsules as an antioxidant and antimicrobial agent (Jummes et al., 2020). The nanoprecipitation process is gaining more attraction due to its ease and flexibility. With slight modifications in the method, different kinds of vectors can be obtained. The formulations and the particle size of suspensions (submicron in range) obtained via nanoprecipitation process was summarized in Table 1, using various active ingredients, polymers and solvents to target specific applications such as inks, cosmetics, diagnosis, pharmaceutical industry.

Double emulsification method

Multiple emulsions methods as double or multiple emulsions can efficiently encapsulate liquid inside shell (Leister and Karbstein, 2020). This technique is better because of its relative simplicity, suitable to control parameters of process, and capability for producing capsules using simple apparatus (Sutthapitaksakul and Sriamornsak, 2019, Ayoub et al., 2011). This formulation method, together with emulsion diffusion and nanoprecipitation technique has been utilized for preparing micro/nanocapsules. They possess an extensive variety of uses in food, cosmetics, in addition to pharmaceutical industry (Iqbal et al., 2015). Numerous biopharmaceuticals as well as pharmaceuticals having dissimilar physical and chemical characteristics are formulated in micro/nanoparticles using this technique (Giri et al., 2013).

Double emulsions can be w/o/w having dispersed oily globules enclosing little water drops or o/w/o along aqueous dispersed phase comprising of small oil droplets (Mudrić et al., 2019). Typically, such emulsions consist of two-step process and have potentially small particle size besides narrower distribution of size in addition to extraordinary efficiencies of encapsulation (Ding et al., 2019). Though, w/o/w is most commonly investigated system as favorable carrier vehicle for drug in pharmaceuticals (Safari et al., 2018). In such process, first aqueous medium comprising active constituents is dispersed into organic medium (i.e., ethyl acetate, ethanol, dichloromethane in which biodegradable polyesters, for instance PCL, PLA, and PLGA are dissolved) to make primary emulsion namely w/o emulsion, After that, primary

emulsion was added into secondary aqueous phase containing a stabilizing agent to assist and formulate w/o/w emulsion. Afterwards, this emulsion was evaporated to remove organic phase out of dispersed medium. It is insolubility point, and as a result, polymer forms a hard shell encapsulating the active material (Panigrahi et al., 2021). For encapsulating liquid inside polymer shell, solvent should be evaporated through rotavapor or else via simply stirring at ambient temperature which doesn't lead to evaporation of the internal phase (Sutthapitaksakul and Sriamornsak, 2019). Micro/nanocapsules are collected after ultracentrifugation, followed by lyophilization for storage. High energy methods like high-speed homogenizer or ultra-sonicator are normally used to make primary emulsion (Sorelli et al., 2017). The secondary emulsion is preferred to be prepared by low-energy method because high speed could break the internal primary emulsion, resulting in only o/w emulsion (Sorelli et al., 2017).

Few double emulsion formulations along with particle size were summarized in Table 1, encapsulating different active molecules inside polymers to target specific applications.

A double emulsion was established to encapsulate BSA (Bovine Serum Albumin-a model protein) consuming poly (ϵ -caprolactone) (PCL) as polymer along with surfactant polyvinyl alcohol (PVA) for encapsulating viral proteins (Martinez et al., 2017). PLGA (Poly (D,L-lactic-co-glycolic acid)) is mainly used to regulate release regarding nicardipine hydrochloride (NCH) in in vitro drug delivery applications. w/o/w emulsion had been utilized for formulating microcapsules of PLGA loaded with nicardipine hydrochloride (NCH). These particles present stimulating alternating system for drug delivery, particularly using NCH in biomedical applications (Soomherun et al., 2017). In another work, same method was used to encapsulate carboplatin (CPT) in microcapsules comprising PET-DLA (poly(ethylene terephthalate-ethylene dilinoleate)) copolymer having average size of 18.5 μm . These microcapsules verified effectiveness towards SK-OV-3 (ovarian adenocarcinoma) human cells to treat ovary cancer (Cymbaluk-Ploska et al., 2019). Multifunctional PLGA-based nanocarriers were obtained by w/o/w evaporation to encapsulate VP (verteporfin), a hydrophobic porphyrin photosensitizing dye, along CisPt (cisplatin), a hydrophilic cytostatic drug in small dose. These had been investigated for bioimaging and anticancer treatment (Bazylińska et al., 2019).

Physico-chemical methods

In physico-chemical methods, a chemical reaction takes place through several physical

transformations for shaping capsules. Some of these methods are described below.

Coacervation/phase separation

It is first process based on technology of encapsulation. In this an active reagent is dispersed in solution of polymer homogenously. Agglomerates (coacervates) of colloidal polymer are made on external surface of droplet of active reagent via stimulating coacervation. This method is primarily utilized for polyelectrolytes, monomer, or else polymers having cross-linkers as function groups to fabricate capsules (Deng et al., 2020). Coacervation methods are divided into two subgroups, i) simple coacervation (It follows the desolvation or salting out of designated polymer use for preparation of microcapsules) and ii) complex coacervation (Accomplished via complexation of polyelectrolytes (two or more) having opposite charges) (Trojanowska et al., 2017).

Coacervation process has three steps. First is development of 3 chemical immiscible phases (i) liquid as vehicle, (ii) core, and (iii) coating material. Core element is dissolved in coating solution of polymer that is immiscible within liquid. The procedure was started by changing one or more parameters of the system, such as altering solution temperature containing polymer, addition of salt, non-solvent, incompatible polymer, or persuading inter polymer interactions. Second stage is polymer deposition as coating on core through mixing of coating and core material inside liquid vehicle physically. Polymer gets adsorbed between interface of core and liquid phase. The last step involves hardening of coating using cross-linking, thermal, or desolvation methods, for developing self-sustained micro/nanocapsules. The bi-layered microencapsules enclosing anthocyanin in *Passiflora edulis* shells (APESs) were synthesized through complex coacervation. Gum Arabic along gelatin were used as materials for first shell materials and gum Arabic having nano-SiO₂ served as material for second shell to increase core stability (Sun et al., 2021). Different antimicrobial capsules with essential oils had been prepared by complex coacervation having antibacterial reactivity towards Gram (negative) and Gram (positive) bacteria (López et al., 2019). Microcapsules loaded with thyme oil being synthesized through complex coacervation were investigated for impact of oil and shell material concentration on yield of microencapsules, distribution in size, and loading capacity. The antimicrobial reactivity regarding developed microcapsules in addition to microcapsule-treated fabric was estimated. These antimicrobial textile were used in medicinal applications for instance wound dressings and bandages (Senem Karagonlu, 2018). The synthesis of

flavor nanocapsules, resistant to heat, containing jasmine essential oil was accomplished by gum arabic and gelatin dependent complex coacervation (Lv et al., 2014). By the same process complex coacervates were synthesized using CMTG (carboxymethyl tara gum) along with gelatin A. They were consumed as materials for shell to encapsulate VD3 (vitamin D3). These nanocapsules have enhanced constancy besides sustained release in digestion (Santos et al., 2021).

Layer by layer (LBL)

LBL method depends upon self-assembly of polyelectrolytes having opposite charges on external surface of colloids. This traditional method is alternate deposition or adsorption of polyelectrolytes with opposite charges (electrostatic attraction) for the formation of a layer or multilayer know as a shell that surrounded and protected the colloidal, oil, or liquid core (Figure 3) (Guzman et al., 2017).

The multi-layer shell can offer several advantages to the system. It can regulate the delivery of active constituent out of core, it can offer better protection of the core from environmental factors, and it can be tweaked to gain responsiveness to multiple changes that can occur during the administration. In addition, these layers can be functionalized in order to add more features to the system. It can protect it during circulation, and it can increase its specificity by combining with ligands specific to the target site (Lengert et al., 2020). LBL assemblage is captivating method used as templates for coating to fabricate micro/nanocapsules. LBL polymeric micro/nanocapsules have many substantial benefits in mechanical effectiveness as well allows to regulate shell thickness, composition, density, diameter, plus permeability (Zan et al., 2015).

The motivating potency of LBL assembly concerns opposite interactions, such as hydrogen bonding, electrostatic attraction, or covalent bonding among layers. Strong attraction between polyelectrolytes having positive and negative charges, layering materials, and layers numbers regulate structural and other capsule properties, for instance biocompatibility, stability, as well as biodegradability (Timin et al., 2017). Addition of a polyelectrolyte layer around a liquid core is done by its adsorption on an opposite charge ionic surfactant. However, use of classical water-soluble surfactants result in forming complex consisting of surfactant and polyelectrolyte and hinder layer formation. Therefore, lipophilic ionic surfactants should be used in this method (Guzman et al., 2017).

Microcapsules were prepared by the LBL method utilizing SLN (solid lipid

nanoparticles) for shell that has negative charged and nanoparticles of gold along with CS (chitosan) that served as shell having positive charge. Ibuprofen and AA (ascorbic acid) were encapsulated inside the shell. The developed capsules are multi-functional, improved efficiency of encapsulation, plus discrete partitions for trapping several drugs (Kalaycioglu and Aydogan, 2020). Microcapsules of tebuconazole, pectin and chitosan, abbreviated as Tebuc@PCN@P@C, had been made through this method. They exhibited exceptional role in PDT (photodynamic therapy) as well as revealed substantial prospective to be used as effective system for delivery in formulations regarding pesticides (Tang et al., 2019). Nanocapsules based on polysaccharides were synthesized utilizing furcellaran with chitosan by LBL method. Consuming doxorubicin- DOX (model drug), they displayed tremendous properties for loading and releasing drug, affected by stability and pH along with exceptional compatibility against eukaryotic species (Milosavljevic et al., 2020). Nanocapsules were fabricated by LbL deposition using anionic CMS (carboxymethyl starch) and cationic QAS (quaternary ammonium starch) on colloidal particles of BSA. They were exploited in oral delivery regarding proteins to colon (Zhang et al., 2017).

Gelation method

It is founded upon droplet formation possessing liquid in core surrounded by shell with important gelling properties. This is an acute method employed to encapsulate enzymes, active complexes or molecules, lipids, and cells among other methods. It is precisely considered as economical due to no requirement of organic solvents innovative apparatus, or else higher temperatures. Simply, by gellifying the outer layer of the droplets, the inner layer will be trapped, and its release will depend on the degradation of the outer gel. Such systems are ideal for controlled drug delivery and release.

The gelation process takes place under trivial environments since powerful stirring, higher temperatures, and organic nature of solvents results in decaying of enclosed compounds. A disadvantage is easy dispersion and quick release of encapsulant if they are hydrophilic or low-molecular-weight, regardless of pH, through matrix of gel (de Moura et al., 2019). It is simple to treat and provides increased stability to the encapsulated bioactive compounds through this process. Particulate gels formed by this process possess numerous practices, comprising structuring, strengthening, as well as texturizing compounds for food webs. They have possibility for improving acceptability regarding product. They are able to alter morphology along with size, permitting precise release concerning active compounds in agronomic,

pharmacological, in addition to foodstuff products (Mohammadalinejad and Kurek, 2021).

The formation of an outer gel layer requires the use of polymers with outstanding gelling properties. A biopolymer, alginate holds unparalleled gelling features that permit simple formation of gel in nontoxic besides insignificant settings (Leong et al., 2016). The gelation of alginate shells is usually accompanied by the use of calcium ions. Depending on the solubility and migration of calcium, gelation can be partitioned into different methods internal, , external, inverse, interfacial, plus interrupted multi-stage gelation (Martins et al., 2017). Microcapsules of chitosan encapsulate essential oil (Figure 4) to examine their releasing property. They displayed an operative essential oil release for controlling growth of fungus and bacteria compared with microspheres of chitosan/k-carrageenan (Raza et al., 2020, Souza et al., 2019).

External gelation

In this method, oil-in-alginate solution emulsion droplets are dispersed in an aqueous CaCl_2 solution. The Ca ions migrate from the aqueous solution towards the droplets. Ca ions will then crosslink the alginate within the droplet forming a gel that entraps the oil core. Ca^{2+} migrates from the outside to the inside; the crosslinking degree of the surface alginate is higher than the inner alginate, resulting in particle formation having thicker outer surface along loose interior.

Several oils can be encapsulated with the external gelation method, oils of canola, sunflower, linseed, olive, lemongrass, and turmeric with many applications for pharmaceutical, food, and cosmetics applications (Maes et al., 2019).

Internal gelation

In this method, oil-in-alginate solution emulsions droplets are supplemented with water-insoluble CaCO_3 . The droplets are then added to an external oil phase containing acetic acid. On acetic acid decomposition, protons out of external oily medium will diffuse to drops which reduce its pH and lead to the breaking of CaCO_3 into calcium and CO_2 . Ca ions will then crosslink the alginate layer internally, leading to the formation of polynuclear capsules, and the presence of CO_2 will lead to the creation of cavities within the alginate matrix. Such scheme goes through huge problems such as organic solvent usage that leads to the necessity of an extensive washing step (Martins et al., 2017).

Inverse gelation

In this method, oil/calcium chloride emulsion is added to an outer alginate solution

phase. The calcium ions will migrate from the droplet to the outer alginate phase, leading to the formation of a uniform cross-linked alginate membrane and a core-shell structure. Active encapsulation by inverse gelation method was first used to obtain liquid-core-shell capsule by adding drops from sucrose solution/ calcium chloride and proteins (active) inside alginate solution bath (Leong et al., 2016).

Inverse gelation method for oil encapsulation is simple technique and does not require huge equipment to obtain capsules, and little parameters compared to other methods such as vibrating jet and microfluidic. By inverse gelation, there is a variety of oil that can be encapsulated (sunflower, linalool, volatile compounds, dyes, and enzymes) and applied to the agriculture and cosmetics industries (Pateiro et al., 2021).

Interfacial gelation

An innovative process for gelation happened at liquids interface that are immiscible had also been reported (Leong et al., 2015). A cargo of oil emulsified in aqueous solution containing nanoparticles of insoluble CaCO_3 . These nanoparticles assembled themselves at interface for formulating pickering o/w emulsion. This prepared emulsion being permitted to phase separation before upper layer (rich in oil drops) had been collected and then re-dissolved in sol of alginate. Addition of acetic acid to emulsion brings nanoparticles dissolution at o/w edge. Ions of Ca^{2+} discharged and bonded to alginate at o/w boundary upon dissolution, lead towards formation of unbroken membrane surrounding core made up of oil, not like previous gelation mechanisms. Thus, such process was designated 'interfacial gelation' (Leong et al., 2016).

Interrupted multi-stage gelation

A hydrogel multi-membrane having onion-like configuration including Ca-alginate can be produced through interrupted multi-stage gelation (Ladet et al., 2008). It was developed via interference and repetitions of mechanism concerning external gelation. In this method, layers of membrane consecutively designed from external to internal core of template made of alcogel. Concisely, alginate alcogel dipped alternately in solution of calcium chloride and water. Ions of Ca^{2+} inside solution of calcium chloride get transmitted across boundary of template afore crosslinking as well as making membrane layer of gel. Then process of gelation get stopped or interrupted via eliminating gel template out of solution of calcium. Then template is submerged into water allowing void creation between gel and solution of alginate. For forming second gel membrane, template is immersed again in solution of calcium. Through repetition of this procedure a number of times, multi-membrane of hydrogel along inter-membrane

voids were produced. This procedure is more accurately named interrupted multi-stage external gelation, in which 'external' states cross-linking trend, from external to the internal template core (He et al., 2020).

Physico-mechanical methods

These techniques have no polymerization process. Polymers are preliminary substances and therefore mostly only the shape formation occurs. Few methods have been summarized below.

Spray drying

This process eliminates liquid out of bulk wet constituents. Previously, people dried food and other substances in hot air or smoke for storage. Later, drying methods via spraying or vacuum were established. Then for drying different set-ups like vacuum-freeze dryer or drum dryer had been designed (Idakiev et al., 2017). In recent times, spray drying is used broadly in pharmacological, cosmetic, and food industries aimed at fabricating useful foods, medicines, and also nutraceuticals. This method to encapsulate is vastly favored because of commercial benefits comparing with all encapsulating procedures. Such method improves handling of products as well as increases oxidative constancy through guarding bioactive composites. It includes numerous parameters like temperatures of inlet and outlet valves, entire solid constituent, and material used as shell, which considerably influence quality of ultimate product (Mohammed et al., 2020). During this method, liquid part (suspension, emulsion, else solution) make drops forcefully with the help of spray nozzle or atomizer. The droplets get dried using hot air, after those solid capsules are accumulated and stored.

It was explored for production of encapsulated particles using subtilisin, model protein, having range in micro to nano scale for oral protein administration. Spherical, smooth-surfaced capsules ranged from 2.2 to 4.5 μm , were synthesized from alginate/protein feed solutions (Erdinc and Neufeld, 2011). Peppermint oil was encapsulated via complex coacervation utilizing a combination of bovine serum albumin (BSA) and gum Acacia (GA). The capsules were then chemically crosslinked by a novel method based on oxidized starch and were afterward spray-dried to examine stability and payload retention under various conditions (Glomm et al., 2021). Encapsulating oil aromas in spray-dried form has been extensively employed in food production, as it is reasonable method to be utilized for nonstop process as well as making of worthy products (Mohammed et al., 2020).

Yet, one drawback of this method is the loss of substances having low-boiling

point during drying in addition to capsule aggregation lead to loss of product due to adhering of material to wall of internal chamber (Trojanowska et al., 2017).

Phase inversion precipitation

This method is developed to encapsulate proteins, small molecules, as well as genes in micro/nanocapsules. This technique includes transfer of mass in addition to separation of phases. It happens when polymer in droplet form gets interacted with non-solvent, as a result polymer get precipitated (Letícia Braz and Ahmed, 2017). It is typically isothermal, ternary system that comprises 3 constituents: solvent, polymer, and non-solvent. Procedure initiates via dispersing polymer into solvent, then casting to desired shape that can be a drop or film. After that dipping of solution inside bath of non-solvent stimulates interchange between non-solvent and solvent molecules. This transfer allows polymer precipitation resulting in membrane having porous structure (Azagury et al., 2018). An exciting benefit of micro/nanocapsules prepared by this technique is tailored morphology significant for systems of adsorption/separation/transport aimed at application regarding environmental and physiological control (Duraikkannu et al., 2021). Solvent interchange leads to phase separation that determines the final morphology of capsules (Liu et al., 2021).

Photosensitive membranes and microcapsules established on poly (α -methylstilbenesebacate-co- α -methylstilbeneisophthalate) were synthesized by this process. The developed capsules have chloroform or concentrated vanillin in chloroform inside core. Outer surface of capsules is uniformly dense before irradiation with UV. When microcapsules were irradiated, morphology of surface altered significantly and release of vanillin get started (Bogdanowicz et al., 2013). Microencapsulation utilizing polymers that are biodegradable are suitable for systems employed in cell and drug delivery. CRL2522 (Human fibroblast cells) microcapsules had been synthesized by cross-linking emulsion method followed by phase inversion with 2 % Tween 80 surfactant. Emulsion cross-linking along with phase inversion microcapsules synthesis resulted in diverse kinds of capsules, clinically established for transporting enclosed mammalian cells for treating future disease (Chaemsawang et al., 2018). Microcapsules of PSf (Polysulfone) are developing substitute because of having higher mechanical constancy besides exceptional capacity for encapsulation.

Polysulfone microcapsules of $30 \pm 10 \mu\text{m}$ of diameter were synthesized having various morphologies by this method. Solution of PSf was exposed to precipitation bath. 3 solvents N,N-dimethylacetamide, N,N-dimethylformamide, and N-methyl-2-

pyrrolidone were evaluated. Baths for precipitation possess water, mixed with solvent or pure. Developed microcapsules exterior were porous in addition to showing macrovoids with sponge-like configuration (Panisello et al., 2013). Microcapsules of PSf/vanillin were also synthesized by this method. Vanillin encapsulation protects until its use besides providing everlasting fragrance (Peña et al., 2012).

Microfluidic method

Traditional microencapsulation approaches have some drawbacks such as specific manipulation of morphology, distribution of size, composition, as well as porosity of developed particles (Li et al., 2018). Consequently, the scaling-up of such procedures has been largely restricted. On the other hand, recent studies have proposed that encapsulation via microfluidic is capable for producing capsules with monodispersity along with accurate control on ratio of core to shell and efficacy for encapsulation. This is due to uniform reaction atmosphere, prospect of reagent addition on exact space and time intervals, as well as probability of decreasing reagents quantity essential for manufacturing (Souza and Al-Tabbaa, 2018). Microfluidic methods have also been developed as an influential device for achieving scaled-up and economic exploration regarding single cells. Microcapsules possibly synthesized by microfluidic employment for encapsulating single cells aimed at detailed and accurate investigation. The microfluidic approach is novel to amount practical and genomic heterogeneity about growth, division, apoptosis, and metabolism concerned to cells (Chen and Lin, 2019). The first studies of the flows in the micro-nano scale were in the 1800s, the method was started to be discussed deeply in the 1970s. Microfluidic technique based on PDMS (polydimethylsiloxane) materials was developed in the early 2000s (Bhushan, 2017). In comparison with the traditional production methods, the microfluidic process enhanced micro/nanocapsules features, in particular; increasing the encapsulation efficiency and controlling shape and size of capsules formed. A microfluidic method rooted in liquid flow through very tiny tubes with the diameter ranging from 5-500 μm to nanometer scale that fabricates micro/nanocapsules with high quality and required characters. PDMS and glass capillary are the most common materials used in microfluidic devices, other materials are also used such as; silicon, paper, and polytetrafluorethylene (Luo et al., 2017, Zhu and Wang, 2016). The employment of such devices to develop

microcapsules has been valuable as a consequence of their small dimension, minimal sample reagents, economical, nontoxic procedure, besides eco-friendly as shown in Figure 5.

Many dominant phenomena have been observed in the microfluidic process like; Laminar flow, diffusion, rapid heating-cooling, surface tension, electrokinetics, geometric control of two-phase flow, and nanoscale (having or involving dimensions of less than 100 nanometres) phenomena (Bhushan, 2017). Currently, expansion in microfluidics systems provides fast and precise production of monodisperse capsules intended for manifold uses. In process of microfluidic encapsulation technique, drug with polymer were solvated in organic medium. After dissolving, it was mixed in the aqueous phase for forming an emulsion. Then an organic solvent was removed because in biomedical applications the organic phase has no benefit, else it is harmful. There are several methods for solvent removal such as lyophilisation, evaporation, precipitation, reverse extraction, and dialysis (Ma et al., 2020b). The stirring process parallel with the solidification process increases the formation of polydisperse microparticles (Zhu et al., 2019).

In microfluidic methods reactants streamed via channels having size of micrometer and combined at junction that controls dispersity and size of drop accurately. A shear force is generated at mixing point between immiscible liquids, causing formation of droplet. This force tuned through changing comparative rates of regarding input streams with size of channels that controls droplet size. Hence, desired shaped of capsules possibly be produced continuously with multi-functionality having diameter of 10 to 50 microns (Vaccaro et al., 2014). The developed microcapsules have consumption in self-care industry for preventing possible threats regarding nanoparticles. Additionally, such approaches need very little quantity of material, completes in less reaction intervals with less energy consumption. There is also minimization of waste along with cost-effective continual process (Thorne et al., 2019).

Microfluidic is one of the most modern technologies for liquid encapsulation. By using this method for creating droplets, the first capillary was used for aqueous phase flux inserted into another capillary which contains oil phase (continuous phase). This method depends on the emulsion formation technique (o/w, w/o, o/w/o, or w/o/w) with controlling the size of capsules. The

droplet dimension is determined by aqueous phase (viscosity and density), flux factors (rates of flow regarding continuous and dispersed phases), capillary geometry, as well as reactants characteristics (Liu et al., 2017). The oil, polymer, and surfactant have vital influence on polymerization and thickness of shell of resulting microcapsules.

The microfluidic method has several important advantages with comparison to other conventional methods for producing capsules of liquids at micro or nano scale such as; fabricating small capsules with controlled size with high quality of properties for the droplets required, providing a high impact in the chemistry and biology fields, reducing the overall reaction time scale and reducing the quantity of the chemical compounds as well as the overall cost of the production process. However, the disadvantage of this method is the low output of the fabricated capsules in comparison with the other liquid encapsulation methods (Ding et al., 2016).

Nowadays, microfluidic has large applications in many fields as; physics, chemistry, biology, environmental sciences, engineering, pharmaceutical (drug discovery and drug delivery), and medical diagnostics (Ganesan et al., 2018). Some of the commercial microfluidic applications include inkjets, bioprinting, micro valves, passive valves, check valves, capillary valves, magnetic and electrostatic valves, rotary and micro pumps. Microfluidics has a high effect and participation in other fields (artificial organs, biopharmaceutical production, droplets in food and cosmetics and continuous health monitoring) (Mandrachia and Tripodo, 2020, Pessi et al., 2014).

An easy microfluidic technique was developed to encapsulate reagents in one step along variable quantities in double emulsion (w/o/w) droplets. Capillary with theta-bent tube being used for fabricating uniform drops, encapsulating different volume of reagents, adjusted through tuning of rates of flow in both channels of tube. Comparative ratio of volume concerning encapsulated reactants got to 1:20. These droplets get transformed into microcapsules holding outstanding prolonged energy. It can be further used for acid/base reaction as well as for redox reaction catalyzed by enzymes to detect glucose (Hou et al., 2017).

Berberine-loaded chitosan/sodium tripolyphosphate nanoparticles (CSNPs) having 130 to 200 nm mean size being synthesized via different microfluidic processes. Homogeneous and uninterrupted fabrication regarding

CSNPs as carrier for drug was assisted through this strategy for drug delivery applications (Farahani et al., 2021). Simple procedure to enclose fish oil utilizing microfluidic device has been established. It was working in constant flow that allowed precise regulation of particle size. Encapsulated capsules are of sphere shape having diameters from 50-250 nm. Such microfluidics incorporating encapsulation through w/o/w was single step without any use of organic solutions, compared with conservative practices involving numerous steps as well as the use of organic solvents. Moreover, large particles with size of about 2 to 4 μm can also be synthesized through this device which is inefficient for food industry (He et al., 2019). Versatile microfluidic method was used to encapsulate PCMs possessing tunable thermal properties. The developed microencapsulated PCMs (MEPCMs) are highly monodisperse with high efficiency for encapsulation, capacity to store energy, reliability, thermal stability, as well as rates of heat discharging and charging. Droplet-based fabrication through microfluidic unlocks different avenues to develop versatile MEPCMs holding exceptional thermal properties, therefore promoting their uses (Han et al., 2020). Uniform microcapsules of alginate were produced by economical microfluidics. Copper sulfate, being used as cross-linker, to encapsulate enzymes, laccase. Copper is consumed because of mechanical properties, high selectivity of sodium alginates for Cu^{2+} ions, antibacterial activity, and likely participation in numerous biochemical activities. Furthermore, it is vital nutrient beneficial for agronomy, wound dressings, in addition to wastewater managements. Device has been designed and tested for varied procedure for evaluating impact on distribution in size and shape along with efficacy for encapsulation (Campana et al., 2020). A microfluidic chip was used to generate limonene microcapsules enclosing monomer diisocyanate inside carrier liquid having NaCl and SDS (sodium dodecyl sulfate). By methodically changing flow rate of oil, sequence of uniform microcapsules of polyurea was manufactured. Comparative rates of flow producing one droplets stream had been directed for reducing particle aggregation at polymerization stage. Though, it is also possible to make small and large sized drops via broadening used flow rates range (Figure 6) from 20–26 μm was the measured mean diameters of droplets through analyzing by video. The rate of flow regarding Qd (dispersed phase) changed from 1 to 5 $\mu\text{L}/\text{min}$. There is an increase in of droplet diameter as Qd increases. Thus sustainable microcapsules

can be produced by manipulating the tunability along with the size control to be used in healthcare, marketable formulations, as well as sustainability and energy applications (Thorne et al., 2019).

A simple and effective dual-coaxial capillary microfluidic device is fabricated to synthesize monodisperse spherical microcapsules of alginate by combining mutilated o/w/o drops on-chip with internal gelation process. This is done by the addition of water-insoluble nanoparticles of CaCO₃ to water phase, having sodium alginate along with acetic acid to external oil phase. The degrees of deformation in addition to inner droplet configuration concerning microcapsules were adapted by varying rate with which outer phase flow. Thickness of shell and diameter of core respecting sphere-shaped capsules were manipulated specifically by controlling rate of flow regarding fluids. These capsules encapsulate α -tocopherol and its release from microcapsules had been investigated inside PBS (phosphate buffer solution of pH 7.4) (Huang et al., 2021). A summary of micro/nanocapsules synthesized by microfluidic systems for a variety of biological applications is presented in Table 1.

There are advanced technologies used to fabricate particles such as supercritical fluid and 3D. The supercritical fluid technology for the encapsulation process has seen rapid development in recent years due to its numerous advantages and properties. The advantages include a high degree of homogeneity, low surface energy, improved stability, solubility in various compounds, inflammability, non-toxicity, and release characteristics, uniformity in size, and the ability to work with a wide range of materials (Ana p et al.,2013). The supercritical fluid technology has therefore provided a great opportunity to develop encapsulated materials that can be used in a variety of applications, such as food ingredients, pharmaceuticals, fragrances and flavorings, agrochemicals, nutraceuticals, and vaccines (mRNA) (Maria et al.,2009). Supercritical fluids can be used to encapsulate a variety of substances, including active pharmaceutical ingredients, vitamins, probiotics, flavors and colors. In addition, it enabled scientists and researchers to develop targeted drug delivery systems with improved accuracy, specificity, and reliability. This has resulted in enhanced safety and efficacy of the drug delivery systems, which in turn has led to improved outcomes for patients. Supercritical fluids (Carbon dioxide (CO₂), Water (H₂O), Methane (CH₄), Ethane (C₂H₆), Propane (C₃H₈), Ethylene (C₂H₄),

Propylene (C_3H_6), Acetone (C_3H_6O), Nitrous oxide (N_2O), and Ethanol (C_2H_5OH), we obtained using this technology are held at or above its critical temperature and pressure, have properties of both a liquid and a gas. They are considered as green alternative in drug delivery and gene delivery applications (Kenji Mishima.,2008). 3D technology for the encapsulation process has also revolutionized many industries, allowing manufacturers to produce more complex products in less time and with better accuracy. Several different types of 3D technologies are used in the encapsulation process, including 3D printing, additive manufacturing, and laser sintering. The benefits of using 3D technology in the encapsulation process include higher accuracy, faster production times, and more complex designs. With the implementation of 3D technology, manufacturers can produce products with greater accuracy and precision in a much shorter period of time than they would be able to do with more traditional methods. Additionally, the use of 3D technology allows manufacturers to create more complex designs that would be impossible to achieve using conventional manufacturing processes (Ong Ernest E.S et al.,2012). In today's modern world show that the feasibility and capability of using as such 3D design technologies in manufacturing various food products like protein snack has been greatly improved and thus represented an important avenue to boost a food product market in the future.

Applications

Micro/nanocapsules have been tremendously desired for various uses in which protection of active constituents by encapsulation, the confined reaction of chemicals, and delivery of active molecules to specific target along with orderly release are needed (Galogahi et al., 2020). Few examples of their applications comprise agriculture, food industry, cosmetics, the constructing self-healing materials, and pharmaceuticals (Deng et al., 2020). Recent advancement in the production of capsules provides effectual enclosing besides transport concerned with hydrophobic composites. It protects them against severe surroundings, and initiated release. Several methods have been used to manufacture capsules consisting of a shell and liquid core, every method has specific criteria, chemical and physical properties that tend to be applied in different fields. In recent years, there has been considerable advancement in the field of microencapsulation aimed at different biomedical applications. Some recent examples have been summarized in this review focuses on the latest advances in biomedical

applications particularly in drug delivery. There has been a lot of work related to the preparation of microcapsules with tailored structures for bio-medical applications (Ghiman et al., 2022). Through the use of microencapsulation technologies, it is possible to develop devices with high efficiency and biocompatibility that can be used for the delivery of a wide range of bioactive molecules into different organs or tissues for the treatment of various medical conditions. This review highlights the most important applications of microcapsules in biomedical applications and emphasizes the importance of tailoring the microcapsule structure to achieve specific desired properties. We also emphasize the impact of new research on advances in microencapsulation techniques in food industry, drug delivery and biomedical applications. According to the European Commission, European Medicines Agency (EMA) and United States Food and Drug Administration (FDA), rigorous testing protocols and standards have been set regarding food and drug safety and their relevant regulatory issues, which help to speed up the process of bringing new food products and drugs to market in both Europe and the US. All food manufacturers must comply with certain standards when producing and labeling products to ensure the safety and well-being of consumers. This technology is widely used in the pharmaceutical industry for delivering drugs to their target sites in the human body through the sustained release of the drug from the carrier over a long period of time. There is a broad range of applications in drug delivery systems, including oral drug delivery systems, transdermal delivery systems, injectable drug delivery systems, and ocular drug delivery systems (Salerno et al., 2022). Significant numbers of encapsulation uses are in biomedical along with pharmaceutical sectors. The developed capsules possess incredible prospective to establish fields related to cell biology, delivery carriers, as well as biosensors. Even though numerous ideas are discovered in past years, however there are several pioneering purposes in addition to innovations need exploration. In following section, some uses of micro/nanocapsules were elaborated.

Drug delivery

Currently, well-regulated systems for drug delivery have fascinated scientists in comparison with customary uncontrollable release of drug. These controlled methods hold significant influence to enhance therapy and pharmacological characteristics regarding drugs taken. Through fabricating capsules at micro and nano scales, it is possible to deliver drugs accurately to the targeted site. Such systems being exceptionally vital aimed at delivery of protein, genes, drugs, and vaccines to particular

sites. The polymeric wall of micro/nanocapsules is ideal for conjugation to ligands for targeting, resulting in selective destruction of cancerous cells, immune or any additional cells on interaction (Richards et al., 2017). Nanocapsule modification through particular bioactive molecules evolved as effective treatment approach to treat cancer by accumulation of drug at targeted sites. Thus, this strategy enhances therapy effects as well as reduces side-effects over healthy organs and tissues (Yameen et al., 2014). Few examples of drugs encapsulated via different techniques are given in Table 2.

The LBL approach was used to form multilayer polyelectrolyte nanocapsule using chitosan (CS-polycation) along κ -carrageenan (κ -CAR-polyanion) encapsulating diflunisal (DF). For this purpose nanoemulsion was first prepared by dissolving dodecyltrimethylammonium chloride (DTAC) in ethanol, olive oil, and acetone. This organic solution was added to water medium. Blend instantly converts milky resulting in formation of drops of nano size, attributed to ethanol/ acetone diffusion to water phase. Ethanol, acetone, along with water portion gets vaporized producing an aqueous nanoemulsion (NE). The prepared NE was dipped alternatively in CS and κ -CAR to obtain preferred number of layers. The *in-vitro* release behavior of DF was investigated. (Figure 7) shows profiles for release of DF that depends on time, from the NE and nanocapsules having core of oil with single, double, triple, and tetra layers of polyelectrolyte at 37 °C and 7.4 pH. 95% of encapsulated DF was discharged from NE. Nanocapsules with single, double, triple, and tetra layers delivered 85 %, 78%, 59%, and 53%, respectively. The obtained release profiles showed that shell of polyelectrolyte served as obstacle for DF release. Hence NE coated by biopolymer and encapsulating DF displayed improved cumulative release features than naked NE. This is beneficial for integrating lipophilic materials in food, agrarian or pharmacological fields (Rochin-Wong et al., 2018).

Alginate nanoparticles were synthesized having protein as an agent intended for oral delivery in gnotobiotic European sea bass larvae using the gelation method. Being a delivery system for fish, alginate was able to offer protection of the encapsulated protein in sea water, while enhancing resistance to disease as well as decrease death rate in larviculture (Yaacob et al., 2017). In another study, the gelation method was used to produce turmeric oil- and lemon- grass oil-loaded chitosan-alginate nanocapsules. It was found these capsules offer a high encapsulation efficiency at around 86%, a sustained release for 48 hours, and high cell viability *in-vitro* indicating their non-toxicity (Natrajan et al., 2015). Several other studies were done to prove the controlled

release behavior of liquid-encapsulating carriers. PMMA polymer was used for the delivery of O₂ to cells and tissues. A W/O emulsion of H₂O₂-H₂O and PMMA was formed and the *in-vitro* profile for release presented 74% H₂O₂ release within 24 hours (Farris et al., 2016).

Another model for delivery of drug was designed aimed at controllable as well as persistent release regarding NSAIDs (nonsteroidal anti-inflammatory drugs). Three different NSAIDs had been encapsulated (i.e., NAP, IBU, CEL equal to model drugs) in polymer microcapsule. Drug encapsulation in these microcapsules has been for avoiding any possible interactions in between drugs. The examples of polymers which are biocompatible, FDA-approved, and biodegradable are PLLA (poly (L-lactide)), PCL (poly (caprolactone)), and PLGA (poly (D,L-lactide-co-glycolide)). An antacid CIM (Cimetidine) is histamine H₂-receptor antagonist being loaded in adapted microcapsule form is extensively utilized for inhibiting secretion of gastric juice. The release capabilities of these drugs were investigated. The results presented persistent NAP and IBU release from PLGA and PLLA nanoparticles, afore their encapsulation in hollow and large PLGA/PCL and PLGA microcapsules. Such microcapsules revealed constant NSAIDs release in PBS further than thirty days. Progressive CIM release following NSAIDs inside SGF (stimulated gastric fluid) been perceived from adapted microcapsules. CIM release is quick and instant, following late and nonstop NSAIDs release. Thus this is a promising system for regulating NSAIDs delivery for fighting against inflammation diseases in addition to protecting against probable gastrointestinal side-effects caused by overdosing of NSAIDs (Baek et al., 2017). Novel polymeric nanocapsules were prepared using pullulan as a stabilizer encapsulating beclomethasone dipropionate (BD). BD is used as per drug model owing therapeutic prospective. BD is corticosteroid medication generally consumed as anti-inflammatory factor for topical therapy regarding asthma and rhinitis. This formulation had been investigated for *in-vitro* release of drug. Results presented controlled release of BD from capsules compared with drug solution in ethanol. Free drug of about 99.59% was released within 24-hrs, whereas 40.48% out of nanocapsules at same time. This showed that nanocapsules prolong release mechanism and might be deliberated as favorable drug vehicles (Figure 8) (Janaïne Micheli Chassot, 2016).

Cosmetics

During past few years, personal care and beauty industry have been turned into billion-

dollar global business along substantial progress within evolving markets. Generally, there is a growing concern about natural formulations for cosmetic. It demands fresh reformulated products consuming bioactive and botanical constituents, counting aromas and fragrances, contributing towards beauty, health, plus wellness. As per EU (European Union), Cosmetics Regulation, products used as cosmetic are defined like “any substance or mixture used for external parts of the human body (epidermis, hair system, nails, lips, and external genital organs), teeth and mucous membranes of the oral cavity for cleaning them, perfuming them, changing their appearance and/or correcting body odors and/or protecting them or keeping them in good condition” (Perinelli et al., 2020). Usage of emergent encapsulation technologies gives advance and efficient properties, therefore surplus value to cosmetics. Precisely, encapsulation skills find increasing advantages in cosmetics and personal care markets, such as; increase stability, increase efficiency and reduce toxicity (Casanova and Santos, 2016).

Fragrance constituents usually consumed in cosmetics are proposed to be applied on skin and hair for releasing pleasant odors. EOs are being valued elements for cosmetic fields. They are added to natural origin products for enhancing product fragrance. Floral EOs, for instance tuberose, rose, gardenia, narcissus, *Lavandula officinalis*, and jasmine are utmost famous aromatic constituents utilized in cosmetics (de Barros Fernandes et al., 2014). Numerous works presented that EOs (e.g. *Pteronia incana*, *Artemisia afra*, and *Rosmarinus officinalis*) possibly served as preserving constituents of cosmetics, because of antimicrobial characteristics towards variety of bacteria strains (Bakkali et al., 2008).

Cosmetic industry has benefited from the liquid encapsulating system. Microencapsulation represents an effective strategy for enhancing stability, effectiveness, and permeability regarding many cosmetics (for instance perfumes) in addition to self-care (body and hand wash, toothpaste, etc.) goods. Additionally, effective formulation concerning micro/nanocapsules must consider various problems, namely amphiphilicity related to volatile composites and obtaining homogenous microcapsules with controllable materials and thickness of shell (Carvalho et al., 2016). Table 2 displays some examples related to coating elements utilized in encapsulation of ingredients having beautifying significance.

The influence of encapsulated virgin oil of coconut on skin had also been investigated. Oil loaded within nanoparticles of solid lipid with 600 nm diameter was added into a base lotion. It was found that the lotion with particles significantly

increases skin hydration and elasticity comparing to the lotion with the non-encapsulated coconut oil (Noor, 2013). The same effect was also observed when rosemary essential oil was encapsulated within similar particles (Montenegro et al., 2017). More recently, dermal photo-protecting nanoparticles were created using lipid carriers as vehicles of a combination of Diosmin and several essential oils. It was found that the encapsulation of the diosmin/essential oil combination improves the antioxidant and antiphotogeing effect comparing to diosmin-loaded particles. Furthermore, microencapsulation of thyme oil is one of the examples of the application in the cosmetic field as shown in Figure 9 that show shows the release kinetics for thymol during the first hour and after 5 days period. It is observed in Figure 9 (A,B) that the diffusion coefficient is $1.39 \times 10^{-15} \text{ m}^2/\text{s}$ for thymol, the polar component, in first hour. Furthermore, for the apolar component, p-cymene, the diffusion coefficient for the first hour of release is lower than that obtained for thymol. This behavior is in accordance with the previous results observed by Wischke and Schwendeman, where the release differences were attributed to the distinct hydrophobic characteristics of the two compound polymeric materials. Microencapsulation by coacervation of thyme oil with PLA is presented as an example. Summarizing, microencapsulation efficiency depends on several variables, namely the chemical nature of the core, properties of the shell/matrix material, the hydrophobic character of the used surfactants, as well as of the chosen microencapsulation technique. In fact, the generated microcapsules in what concerns size, shape and stability, is affected by the used coacervation process conditions, being particularly relevant the chosen surfactant. In the case of the focused process (encapsulation of thyme oil with PLA) higher encapsulation efficiency for apolar compounds of thyme oil was verified. The best encapsulation results were achieved with Tergitol 15-S-9 (HLB of 13.3): 80% for the apolar compounds and 54% for the apolar ones. The release profile of thymol (major polar compound of thyme oil) from the PLA microcapsules can be explained by a diffusion mechanism, in accordance with the developed model and was found to be in good agreement with the experimental measurements. Figure 9 (C,D) shows optical and cryogenic scanning electron microscopy images of PLA microcapsules with thyme oil reported by Martins et al. Images obtained through optical microscopy demonstrate that droplets of thyme oil have been individually encapsulated as spherical particles without noticeable agglomeration. On the other hand, the Cryogenic Scanning Electron Microscopy (Cryo-SEM) image confirmed the rough surface of PLA microcapsules with some visible

pinholes, cracks and pores. The presence of essential oils within the system increases the anti-erythematous and anti-wrinkling effect, and therefore offers a better photoprotective agent (Kamel et al., 2017). Complex coacervation–emulsion polymerization technique was used to synthesize apple aroma microcapsules consuming tetradecylallyldimethylammonium bromide with sodium alginate as materials for shell. Acquired microcapsules possess core-shell morphology and are sphere-shaped with diameter ranged from twenty to fifty micrometers. They are thermally stable to 110 °C. Aroma of about 10.8 % gets released after 100 hrs from optimized formulation. There is an increase in the release of aroma when microcapsules are under pressure, resulting in prospective uses in cosmetics (Song and Chen, 2018). A micro system established on proteins of keratin and zein to release fragrances in hair. Menthol and linalool were employed as typical fragrances. Keratin/zein microcapsules being fabricated by two methods: (a) First nanoparticles of zein were prepared followed by deposition of keratin on surface via electrostatic interactions (b) precipitation of zein along keratin to form microcapsules. Microcapsules form a film on hair, release fragrance thus enhancing degree of hydration as well as mechanical features related to hair. The established system showed substantial probability towards progress of self-care cosmetic products for hair permitting modifications relying upon particular sensual inclinations (Tinoco et al., 2021).

Textile industry

The use of encapsulation techniques brings the opportunity to produce novel products with countless benefits in comparison to traditional textile products. The micro/nanocapsules presented invaluable aspects to fabrics and garments, particularly improved constancy as well as precise delivery regarding active composite(s). A large range regarding marketable products based on microencapsulation materialized in textile sector in 1990s, whereas still numerous prospective products are under exploration and progress phase (Siler-Marinkovic et al., 2006). Common samples are textiles having long lasting fragrances, T-shirts possessing microcapsules for absorbing UV-rays or thermo-adaptable dyes, ski suits, uniforms for military forces encapsulating insecticide, thermal controlled carrier seats, and gloves.

The micro/nanocapsules encapsulating fragrances and aromas are consumed for years for finishing procedures used for textiles. Essential oils and perfumes are been supplemented within ties, scarves, lingerie, in addition to additional garments. They are

also been used for home-based textiles, for instance curtains, sofa covers, as well as cushions aimed at aromatherapy. These aromas and oils are applied directly onto textiles though, rare attraction of oil or aroma onto fabric along with chemical instability restrict textile performance (Perinelli et al., 2020). By encapsulating fragrances, they are preserved and delivered for longer period. Furthermore, feeling of fragrances encapsulated inside fabric is kept after a number of washings; hence, the desirability of the product to the consumers is enhanced (Zhao et al., 2016).

Thus, encapsulation improves the sustained period regarding aroma perceptions because of precise release of fragrance. Aimed at this, some fabrics are treated by encapsulating aromas and fragrances, like silk, cotton, in addition to synthetic textiles (polyester or polyamide) (Bruyninckx and Dusselier, 2019). Interfacial free-radical polymerization was used to fabricate microcapsules of poly (1,4-butanediol dimethacrylate) encapsulating DPO (dementholized peppermint oil) in core. The prepared fragrance microcapsules are sphere-shaped shape and possess exceptional thermal constancy at temperature lower than 100 °C. They efficiency for encapsulation is 91%. Moreover, these microcapsules with fragrance overlaid on cotton textile exhibited better washing strength ever after fifteen rounds. This is advantageous in future to be used in everyday requirements in addition to textiles (Zhao et al., 2016).

The microencapsulation of citronella oil through complex coacervation use gum Arabic biopolymer and gelatin for coating. *In-vitro* citronella release out of microcapsules coated onto fabric has been examined and results revealed the holding and releasing of fragrances for long duration because of interaction of fabric with water (Bezerra et al., 2019). All-aqueous electro-spraying technique was proposed for producing complex nanocapsules comprising fragrance/2-hydroxypropyl- β -cyclodextrin along renewed silk fibroin having efficiency for aroma encapsulation larger than 90% inside matrixes. The prepared silk nanocomposites can preserve the volatile cargo more effectively and displayed ideal kinetics for release of zero-order. A slow rate of release was observed on alcohol treatment. Additionally, one benefit is permission of direct layering of nanoparticles carrying fragrance onto silk textiles throughout electro-spraying process (Ye et al., 2019). The retention of aromas and fragrances, particularly later to washing and rubbing, rely onto micro/nanocapsules diffusion inside textile pores in the process of finishing. Sequence concerning micro/nanocapsules, having size comparable to spacing between pores present in cotton fabric were fabricated via encapsulating citronella oil in chitosan shell through microemulsion methodology. Vacuum

impregnation was used to apply these micro/nanocapsules onto textile. The retaining of aroma on cotton processed by nanocapsules ((28.84%) had been greater when compared with similar textiles done by microcapsules (1.55%) even after washing 10 times. It might be due to better penetration of nanocapsules inside the textile pores ((Medeiros et al., 2011).

Microcapsules with well-defined geometry regarding gum Arabic and gelatin encapsulating methyl salicylate in core were acquired through complex coacervation. Later they were assimilated in cotton (100%), Jersey and polyamide (100%) textiles. Controlled *in-vitro* methyl salicylate release out of polyamide and cotton and from samples containing cotton (100%) and polyamide (100%) modified by microcapsules has been studied. The effect relating to temperature gets clear after exposing samples to washing. There is a prominent release at higher temperature (50 °C) as compared to the lower temperature (37 °C). It indicates base of releasing mechanism is subjective to matrix of textile in addition to microcapsule durability during washing. (Figure 10) presents *in-vitro* profiles of release for samples of polyamide and cotton. From each sample, release of methyl salicylate gets complete nearly within sixty minutes, approaching about 98%. However, microcapsule-modified samples, release occurs in 2 parts: an initial fast release called burst effect, following gradual increase till equilibrium is reached. In this maximum methyl salicylate get released after two-fifty and three-fifty minutes for cotton and polyamide, respectively. The first release step was due to the methyl salicylate release stacked on substrate surface of textile; this part of compound has not been encapsulated. Hence above-mentioned facts, functionalization concerning cotton and polyamide substrates are deliberated as favorable substitute aimed at progress regarding practical textile. They are contributing to customer well-being because of microencapsulation as well as durability (Mendes et al., 2021).

The encapsulation of fragrances and aromas were also used to fabricate “smart textiles” having additional functional properties e.g. UV shield, antibacterial, moisturizing as well as skin therapy, regulation of temperature concerned to body, in addition to insect repellency, dependent upon role of encapsulated aromas, fragrances, or EOs (Romagnoli et al., 2020). An easy and green process has been established to fabricate multifunctional fabric employing hybrid silica/cellulose microcapsules. Synthesis of microcapsules encapsulating Lavender oil as fragrance takes place in single step through emulsion diffusion process. They have core-shell morphology holding UV absorbers with silica shell grafted by methacrylic acid. Afterwards they were added to

resins of waterborne polysiloxane for making multifunctional coatings for fabric. This fabric displayed slow release of oil in controlled manner, keeping thirty percent fragrance even after ninety days (Chen et al., 2020). Release of aroma from cotton fabrics incorporated with nanocapsules in controllable way possesses durability for washing in addition to enduring fragrance deprived of adhesives. They were synthesized using electrostatic-adsorption with *in-situ* immobilization methodology. This method delivers eco-friendly approach to prepare nanocapsules holding prospective uses in area regarding practical fragrant domiciliary, perfumed medicinal care textiles, progressive efficient materials, outfit products, etc. (Ma et al., 2020a).

Cosmetotextile is another perception based on releasing superficial ingredient to skin of humans. Cosmetotextiles sector is full of potential and demands resilient necessity as regards smashing together diverse industries for instance cosmetic, textile, herbal pharmaceuticals, amongst all others (Alvarez et al., 2017). Thus cosmetic ingredients can also be incorporated inside the cotton fibers of clothes that get transferred after contact with the skin to impart some cosmetic benefits at an adequate dose (Mamta et al., 2017).

Methanol is mostly used as a cooling agent that provides a freshening sensation. Menthol was encapsulated inside nanocapsules having lipid core through nanoprecipitation and investigated for skin usage. There is a fast penetration of nanocapsules inside layers of skin, especially via trans-epidermal absorption ways besides hair follicles. Hence, established capsules enhanced water solubility and thermal stability regarding menthol. They are also alternate preference in cosmetotextiles along quick skin absorption characteristics (Yingngam et al., 2019). Table 2 summarizes the current research on micro/nanoencapsulation as regards flavors and fragrances aimed at textile uses.

Food industry

Encapsulation is positive way to provides core constituents a shielding along with better stability. As compared to pharmaceutical applications; processing of food involves complex necessities aimed at safety as well as hygiene. This demands nutrition in addition to quality maintenance. Customized design with economical, effective, also green synthesis regarding microcapsules been immediately desired for meeting demands concerned to diverse handling or else storage situations (Yang et al., 2020a).

Encapsulation provides several benefits that find their use in the food packaging,

preservation, and safety sectors (Pradhan et al., 2015).

An interesting illustration is flavors encapsulation inside chewing gums which get released by chewing, yeast encapsulation which is released at a time of baking, and probiotic bacteria protection by encapsulation in gastric environment that get released in small intestines (Galogahi et al., 2020). The use of the liquid-loaded system in the food industry focuses mostly on anti-bacterial agents for the long-term preservation of food products. Essential oils are heavily used for such purpose due to their great anti-bacterial activity, good taste, and their natural origins. Some of the important polymers used for microencapsulation with various characteristics are given in Table 3.

Lavandin essential oil-loaded particles were produced and influence of concentration of oil and carrier on anti-bacterial properties was tested. Lecithin particles showed a better activity compared to starch particles, while the encapsulated essential oil generally possesses a higher antibacterial activity than free oil (Varona et al., 2013). Essential oil (EO) of clove was entrapped inside nanoparticles of chitosan consuming gelation technique. It was found that encapsulation was able to increase antimicrobial and antioxidative characteristics of EO (Hadidi et al., 2020). Another study discusses the encapsulation of lavender essential oil within double-layered microcapsules. The system is consisting of an oil core, chitosan and sodium alginate for external layer along with β -cyclodextrin for internal layer. System yields 80% efficiency for encapsulation besides continual release of drug. Essential oil fragrance and the introduced cotton fabric lasted for several months, suggesting the feasibility of such a system for food and insect prevention purposes (Zhang et al., 2020). Microencapsulated garden cress seed oil was added to protect α -linolenic acid to avoid oxidation in biscuits and prolong their life (Umesha et al., 2015). Furthermore, cyclodextrin encapsulated capsules maintained flavour of strawberry against environmental pressure that is significant for food sector to produce high-quality products (Pellicer et al., 2019). Unique Syringa EO microencapsulation (SEOM) had been carried out by inclusion technique. The microcapsules have 39.52% efficiency of encapsulation in addition to 11.54% capacity of loading. *In-vitro* caryophyllene, eugenol, and humulene release from microcapsules was investigated. 1-MCP treatments and/or SEOM preserved fruit stiffness, acidity, and also inhibited its decay and release of ethylene. SEOM also stimulated biosynthesis of terpenoid and ester along with inhibiting production of aldehyde in fruit, Prunus Persia, at the time of storage (Yang et al., 2019). Figure 11 shows a clear example of microencapsulation of mustard (*Sinapis alba*) seed essential oil by complex

coacervation and thermogravimetric analysis (TGA) curve of microcapsules under different hardening conditions. Figure 11 (A) illustrates the curves after different periods of cross-linking, with increasing stability with time. It could be observed that the entire decomposition process corresponds to the temperature range of 250-450 °C (the weight loss at temperatures lower than 150 °C was attributed to the water content in the microcapsules). Because the cross-linking reaction was incomplete at 2 h and 4 h, the drastic weight loss occurred at 250 °C, and thus the microcapsule hardening effectiveness was poor. An incremental increase in the cross-linking time of 8 h or 12 h resulted in a higher structural stability because the intermolecular cross-linking density in gelatin increased with time, improving microcapsule stability until the entire gelatin molecules reacted. As the TGA curve showed no obvious change when the cross-linking time increased from 8 h to 12 h, it could be concluded that 8 h was the optimum cross-linking time for the hardened stage. Figure 11 (B) showed TGA curves with different cross-linking pH values, indicating that the cross-linking reactions of microcapsules were pH dependent. When gelatin was the substrate, genipin exhibited an optimal activity at pH 10.0, ensuring that the microcapsules were sufficiently hardened during cross-linking. Under neutral and weak basic conditions, the decomposition process of the genipin cross-linked microcapsules started at 250 °C. This was because under neutral and weak basic conditions genipin and amino group-containing polymer, such as gelatin, formed cross-linked networks by short chains of cross-linking bridges. However, as the basic concentration increased, genipin would undergo a self-polymerization reaction prior to cross-linking with gelatin. Therefore, the gelatin-arabic gum microcapsules cross-linked with genipin under a relatively strong base consisted of long chains of cross-link bridges. However, the stability of the microcapsules was reduced when the pH was increased to 12, which might account for too strong alkaline conditions leading to microcapsules prone to swelling. with SEM micrographs of microencapsulated mustard seed oil that show micrographs of microencapsulated mustard seed oil. Figure 11(C) representatives of complete microcapsules without the cross-linking process. Figure 11(D): representatives of complete microcapsules with the cross-linking process. (Peng et al., 2014).

Agriculture

It is one of the vital as well as established sector reasons being yielding and delivering basic stuff used in feed in addition to food services. Sustainable growth regarding

agronomy leans on original and inventive practices along with the usage of chemicals particularly fertilizers or pesticides. The advancement concerning advanced agronomic system along consumption of smart engineered nanotools possibly is an exceptional approach for making breakaway in an agrarian field. The use of nanomaterials not only also decreases or removes effect concerning contemporary farming on atmosphere but also enhances worth in addition to yield quantity (Liu and Lal, 2015).

Encapsulation technology has been employed for developing better products to deliver wanted chemicals for targeting biologic route. This holds huge potential for plant sciences aimed at food production as well as environmental protection (Ram et al., 2014). The application of micro/nanocapsules in the agricultural sector becomes a significant cause towards ecological expansions. In areas of food and agronomy, such technology turned out to be beneficial for resources supervision regarding agriculture, mechanisms of delivering drug in plants in addition to helping in preserving soil fertility (Sabir et al., 2014). In the agriculture industry, micro/nanoencapsulation is principally appropriate to shield active components responsive to light, oxygen, moisture, degradation besides insoluble, volatile, or reactive substances that leads to high yield crop production. Nonetheless, one imperative purpose of encapsulation of active ingredient is controllable release regarding active molecules to reduce its side effects.

Recently formulation of encapsulated pesticide in nanocapsules holds slow and gradual releasing characteristics as well as improved specificity, solubility, stability, and permeability such as; the effect of release rate of azadirachtin from the beads loaded with different amounts of neem oil nanoemulsion, coated with starch and PEG is presented in Figure. 12. It was observed that the higher the amount of nanoemulsion loaded the higher the release rate. The release rate of 8% of neem oil nanoemulsion loaded beads has shown 100% release in the 24th hour. In case of 4% and 1% neem oil nanoemulsion loaded beads, they showed only 98.4% and 95.8% release after 24th hour. 100% release rate was obtained with in small concentration of nanoemulsion itself which makes this process more effective. Beads loaded with different amounts of neem oil nanoemulsion (1%, 4%, and 8%) were selected for starch and PEG coating, since in all cases they showed effective release. It is clear that the neem Aza- A release rate is reduced significantly by starch and PEG coating, which is consistent with the results of the swelling study. The effect of release rate of Aza-A from the beads coated with starch was found to be more effective compared to PEG. The release of azadirachtin from starch coated beads were particularly less compared to PEG coated ones. The neem

Aza-A cumulative release in case of 8% neem oil nanoemulsion loaded beads without coating exposed for 6, 12, 18 and 24 h in aqueous medium was 44.2%, 66.8%, 79.4% and 100% respectively. But when same beads were coated with starch in the same condition, the release was observed to be 31.6, 40.4, 51.6 and 70.6 respectively. In case of PEG coated ones; it was 41.6%, 54.2%, 66.8% and 80.6% respectively. It should be noted that with coating of starch and PEG, the bead matrix becomes more compact and rigid, resulting in reduction in rate of diffusion of neem Aza-A through swollen beads. Starch coated beads showed decreased rate of diffusion compared to PEG coated ones. Scanning electron microscopic image showed that the beads formed were almost spherical in shape with smooth surface. The mean particle size was 1.21 mm. After the coating of beads with starch and PEG, their diameter drastically increased to 1.32 mm and 1.34 mm and had a smoother bead surface (Jayakumar et al., 2012). Nanoencapsulated pesticides formulation lessens pesticides dose along its exposure to human beings, revealing environmentally friendly nature. Furthermore, it redirects good quality about biodegradable components to protect crops. Though insufficient data on synthetic mechanism is available; so no appropriate statistics regarding cost-benefit checkups of micro/nanoencapsulation complexes hang up their uses for delivery pesticides. Systematic investigation contributed major as well as crucial figures meant for technocrats to utilize encapsulation methods with provision for pesticide. These properties are primarily attained by preservation of encapsulated components from untimely decomposition or else growing their efficiency to control pest for long duration (Nuruzzaman et al., 2016, Younas et al., 2020). Table 3 summarizes a few nanocarrier systems based on encapsulation of active molecules for agriculture applications.

Alginate/chitosan nanoparticles encapsulating the herbicide paraquat had been synthesized against contamination faced in underground besides surface hydro systems. The results showed that paraquat encapsulation within nanoparticles of alginate/chitosan helps in reducing ecological influences and also in controllable chemical discharge. Developed system is simple to prepare, environmentally safer, and efficient that creates them to be beneficial substitute for agronomic usages (Silva Mdos et al., 2011). The two herbicides imazapic and imazapyr were encapsulated inside shell made up of chitosan/alginate (CS/ALG) and chitosan/tripolyphosphate (CS/TPP) in agriculture to control weed growth. The results displayed that fabricated nanocapsules have ability to encapsulate herbicides by more than 60% efficiencies. The developed nanocapsules

enhanced activity along with reducing effects on environment and human, that would be positive scheme aimed at maintainable agrarian production (Maruyama et al., 2016). Microencapsulation of LC (lambda-cyhalothrin), an extensively consumed pesticide enclosed inside shell of PUU (polyurethane-urea) was successfully done via interfacial polymerization. Microcapsule sizes were estimated to be in range of nanometers to micrometers (45) with shell thicknesses in between 512.3 to 724.5 nanometers in addition to maximum yield of 85.5%. These microcapsules were able to halt on leaves of peppermint for about eight weeks and actively functions against bugs present inside leaves of peppermint through this period (Yilmaz et al., 2021).

Conclusion

Encapsulation is both a science and an art that demands experience, capability, and the authority of a wide range of technologies. This review has reported various encapsulation techniques to fabricate micro/nanocapsules along with their diverse applications. Nanocapsules containing polymers contributing to systems used for delivery of drugs could increase payloads bioavailability besides attaining persistent with precise delivery to target sites. Microencapsulation enclosing oils, food, and flavors improves encapsulation efficiency and extends products average life with the purpose of yielding good-quality food stuffs. In the field of cosmetics, microencapsulation has been developed for making items like antiperspirants, shampoos, and sprays, to improve their stability or bioavailability. Microencapsulation methods bring opportunity to fabricate unique products possessing several benefits when compared with customary fabrics. Encapsulation into microcapsules ensures the protection, controlled release of active agents with higher efficiency, and eco-friendly sustainable plant growth.

Despite the significant development of new-fangled yields, space is still available in place of more exploration for scaling up this technology for large-scale applications. However, more consideration with research is required for solving unsettled issues and for escalating their employments. Foremost issue is bringing of these smart capsules from hypothetical investigation to industrial scale. The microfluidic approach appears beneficial for overcoming problems related to orthodox procedures, along with disadvantages. Further research is desirable to advance the current practices in addition to improve innovative devices and schemes aimed at amplifying micro/nanocapsules capacity.

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Table 1. Summary of the studies of nano/micro capsules with various shell and core materials synthesized by interfacial polymerization, nanoprecipitation, double emulsion and microfluidic methods that contain different active substances for various applications, indicated with average diameter, PDI (Polydispersity index) and zeta potential.

Capsules	Shell material	Core material	Average diameter	PDI & zeta potential (mv)	Applications	Encapsulation technique	Ref
Polyamide microcapsules*	Polyamine	Poly acrylic gel, amino acid, enzymes, antibody	50-200 μm	(0.2)&(+40.2 \pm 0.8)	Extraction of metal cations, pharmaceutical applications	interfacial polymerization	(Laguecir et al., 2002, Yang et al., 2020b, Hussain et al., 2018)
Polyurethane* microcapsules	Polyurethane	Aromatic ether, vitamins, essential oils, antimicrobials, and/or antibiotics	0.15-5 μm	(0.24 \pm 0.01)&(+6.9 \pm 0.7)	Cosmetotextile applications, drug delivery	interfacial polymerization	(Ben Abdelkader et al., 2019, Valdes et al., 2018)
Polyurethane-urea* microcapsules	Polyurethane-urea	Dyes, bioactive compounds	1-500 μm	(0.21 \pm 0.02) & (-28.2 \pm 0.7)	Textile and food industries	interfacial polymerization	(Granato et al., 2019, Ozkan et al., 2019)
Polythiouretthane* microcapsules	Polythiouretthane	Self-healing agents	2-20 μm	(0.15 \pm 0.02) & (+6.9 \pm 0.7)	Self-healing systems	interfacial polymerization	(Szmechtyk et al., 2017)
Polyurethane* nanocapsules	Polyurethane	Bioactive molecules or drugs	248-261nm	(0.17 \pm 0.1)&(-8.9 \pm 0.3)	Enzyme triggered drug release, imaging	interfacial polymerization	(Pramanik et al., 2018)
Polyurea* nanocapsules	Polyurea	Liquid magnetic nanoparticles	5-20 nm	(0.19 \pm 0.01)&(-8.7 \pm 1.5)	Medicine, biotechnology, agriculture, magnetic resonance imaging (MRI), catalysis, besides additional industrial	interfacial polymerization	(Natour et al., 2019)

						and environmental applications		
Polyaniline* nanocapsules	polyaniline	Self-healing materials	400-700 nm	(0.14) & (-36)	Controlled release	interfacial polymerization	(Dolatkhah and Wilson, 2020)	
Polyamide* nanocapsules	Polyamide	Antimicrobial, anti-inflammatory, and antioxidant agents	50-100 nm	(0.08 ± 0.01) & (-6.6 ± 0.6)	Biomedicine, active packaging, and cosmetics	interfacial polymerization	(Marturano et al., 2018, Deng et al., 2020)	
PLGA	PLGA	Therapeutic Drugs for instance, antibiotics, chemotherapy, anti-inflammatory, antiseptic, proteins, and antioxidant drugs	5-2000 nm	~0.30 & (-18.7 mV ± 6.1)	Bacterial infection, anti cancer drug delivery, in addition to gene therapy	nanoprecipitation	(Swider et al., 2018, Zhi et al., 2021)	
PCL, PLA	PCL, PLA	Fluorescent marker (organic dye or quantum dot), cytostatic drugs, essential oils (rosemary, oregano)	160-180 nm	(0.16 & 0.019) & (-27.2 & -6)	Bioimaging, agriculture, textile	nanoprecipitation	(Drozdek and Bazylińska, 2016, Lammari et al., 2020)	
Amphiphilic block copolymers (PCL-PEG-PCL, triblock, PCL-PEO-PPO-PEO-PCL, pentablock)	Amphiphilic block copolymers (PCL-PEG-PCL, triblock, PCL-PEO-PPO-PEO-PCL, pentablock)	Dyes	50 -200 nm	(/)	Drug delivery, imaging, skin treatment	nanoprecipitation	(Dias et al., 2019, Oh, 2011)	
Polyesters	Polyesters	Antioxidant agents	50-300 nm	(0.22) & (-4.11)	Food and agricultural Industries	nanoprecipitation	(Barreras-Urbina et al., 2016)	
PLGA-PEG	PLGA-PEG	Chemotherapeutic agents	130-150 nm	(/)	Tumor diagnostic	nanoprecipitation	(Darwish and Bayoumi, 2020)	

Poly(ethylene glycol)*	Poly(ethylene glycol)	Gemcitabine hydrochloride	210-11 nm	(0.09 _ 0.01) & (~±30)	Clinical applications(treatment for different types of cancer, including bladder and breast cancer)	double emulsion	(Cosco et al., 2015)
Poly(L, lactide)*	Poly(L, lactide)						
Acetalated dextran	Acetalated dextran	Resiquimod (R848) Muramyl dipeptide (MDP)	~150 nm	(/) & (~±30)	Cancer treatment (treating inflammation)	double emulsion	(Passlick et al., 2018)
Poly(methacrylic acid)	Poly(methacrylic acid)	Doxorubicin Paclitaxel	184-10 nm	(/) & (+15.8 ± 0.42)	In vivo/In vitro cancer therapy(reatment of the blood, lymph system, bladder, breast, stomach, lungs, ovaries, thyroid, nerves, kidneys, bones, and soft tissues, including muscles and tendons)	double emulsion	(Chiang et al., 2014)
Polyvinyl alcohol*	Polyvinyl alcohol	Magnetic nanoparticles					
Poly(styrene)*	Poly(styrene)	Plasmid DNA PEGylated quantum dots Pyrene Doxorubicin	263-42 nm	(/)	Treating,cardiovascular ailments,cancer neurologicalsyndromes, and infective diseases	double emulsion	(Hu et al., 2012)
Poly(lactic acid-co-glycolic acid)	Poly(lactic acid-co-glycolic acid)	Bovine serum albumin	327 nm	(/)	Tissue engineering	double emulsion	(Yilgor et al., 2010)
Polycaprolactone	Polycaprolactone	Proteins	23–47 μm	(0.16 ± 0.019)&(-5.4 ± 0.057)	Improved properties for targetting, low porosity, 95% stability, monodispersity, and high protein encapsulation, 90% effeciency.	Microfluidic	(Pessi et al., 2014)
Polyamine	Polyamine	Self-healing and curing agents	10 μm	(/)	Self-healing systems and 90% performance, 95% stability, thermal stability,	Microfluidic	(Zhang et al., 2018)

					comparatively rapid kinetics for healing		
Polyurea *	Polyurea	Flavors, Aromas	10-50 µm	(/)	Cosmetic and Food industry, Long product mean-life, ability to deliver at right rate and time, moment, not dangerous for environs		
Alginate*	Alginate	Biocides	160 µm	(/)	Marine applications and building materials, Environment-friendly, monodispersity and High encapsulation efficiency 96%, improve biodistribution and bioavailability	Microfluidic	(Polenz et al., 2014)
PLGA	PLGA	Drugs, fluorescent dyes	50- 200 µm	(/)	Cosmetics, pharmaceuticals, and contrast imaging, Less side effects, controlled drug release kinetics, and 94% encapsulation efficiency.	Microfluidic	(Rezvantab and Keshavarz Moraveji, 2019, Operti et al., 2019)
PLA	PLA	Drugs, contrast agents	10-50 µm	(/)	Drug delivery system, Enhance drug efficacy, less drug toxicity	Microfluidic	(Watanabe et al., 2013)
Chitosan*	Chitosan	Drugs, genes, proteins,	200-600 µm	(0.17 ± 0.02)&(-8.17 ±0.1)	Sustained drug release, high cell adhesiveness, nontoxicity, and biodegradability	Microfluidic	(Yang et al., 2016)
Gelatin*	Gelatin	Drugs, enzymes	45 to 120 µm	(/)		Microfluidic	(Yeh et al., 2013, Sakai et al., 2011)

*; products commercially available.

Table 2. Examples of some different techniques used for microencapsulation of the drugs applied in different fields, such as; drug delivery, cosmetics and textile.

Drug	Coating material	Encapsulation Technique	Ref
Insulin, Doxorubicin*	Poly(butylcyanoacrylate), poly (alkyl cyanoacrylate),	Emulsion polymerization	(Mandal, 2020)
Enzymes, amino acid, antibody*	Microcapsules of polyamide	Interfacial polymerization	(Bah et al., 2020)
BSA*	PLGA (poly(DL-lactic-co-glycolic acid), PCL (Polycaprolactone)	Solvent extraction/ evaporation	(Swider et al., 2018)
Chlorothiazide	Protein (Albumin), polysaccharides	Suspension crosslinking	(Jiang et al., 2006)
Diphtheria toxoid Leuporelin acetate Tetanus toxoid	PLGA and poly lactic acid (PLA)	double emulsion method (w/o/w)	(Singh et al., 2006)
Melamine formaldehyde	Rosemary, Lavender, Sage	In situ polymerization	(Wei et al., 2020)
Chitosan*	Citronella	o/w emulsification	(Hsieh et al., 2006)
Ethylcellulose*	Rosemary, Lavender,	Phase separation	(Carvalho et al., 2016)
Gelatin*	Holy basil	Simple coacervation	(Sutaphanit and Chitprasert, 2014)
Alginate*	Clove, Thyme, Cinnamon	Emulsion extrusion	(Soliman et al., 2013)
Cellulose *derivatives/Polyvinyl alcohol (PVA)	Eugenol	Oil-in-water emulsion solvent evaporation	(Simões et al., 2020)
Crosslinked polymers	Model fragrances	Double-emulsion	(Stasse et al., 2020)
Polyurea Shell*	Thyme oil	Interfacial polymerization method	(Kudligi et al., 2019)
Starch/Glutaraldehyde	Aloe vera EO	Coacervation	(Fiedler et al., 2019)
Polyurethane/ β -CD	Neroline	Interfacial polycondensation	(Ben Abdelkader et al., 2019)
Copolymer of styrene/methyl methacrylate as core, poly(butyl acrylate) reformed via octamethylcyclotetrasiloxa	Jasmine EO	Two-stage emulsion polymerization	(Liu et al., 2018)

ne as shell
 Butyl Limonene Free-radical emulsion (Manfredini
 methacrylate/ethylene polymerization et al., 2020)
 glycol dimethacrylate

*; products commercially available.

Table 3. Summary of some of the important polymers used for microencapsulation with various characteristics active molecules for agriculture and food industry applications.

Polymers	Core material	Characteristics	Ref
Alginate,* chitosan/inulin, *chitosan/alginate, Chitosan/carboxymethyl cellulose,* Chitosan/sodium* tripolyphosphate,	Chitosan, Coriander (essential oil) Carotenoids Sodium caseinate	EO Increased degree of swelling, encapsulation efficiency and rate of release, Unaffected by to temperature and pH changes Release in a controlled manner and increased efficacy for encapsulation Increased thermal stability and encapsulation efficiency	(Dima et al., 2016) (Rutz et al., 2016) (Li et al., 2019)
Vegetable combined with dairy proteins	proteins		
Pullulan, carboxymethyl cellulose*	Tuna/peppermint oil, Tuna oil	Sphere-shaped microcapsules having uniform surface, better antioxidant ability, oxidation constancy	(Bakry et al., 2016b)
Inulin and chitosan*	Lactobacillus casei (L. casei)	Enhanced L. casei rate of survival	(Darjani et al., 2016)
Pectin and isolates of whey protein	Anthocyanins	Thermally stable, antioxidant	(Stănciuc et al., 2017)
Chitosan*	Agrochemicals and genetic material	Efficient gene delivery system, Controlled release of pesticides	(Kashya p et al., 2015, Maluin and Hussein, 2020)
Cellulose, starch, lignin*,	Herbicide, insecticide, and pesticide	Assisting the plant growth, growing plant production in dry regions	(Bhatia, 2020)
Alginate/Chitosan*	Biocontrol fungal agent	Increasing average life relevant to bio-control agents for fighting against diseases of plant and stimulate their growth	(Jurić et al., 2021)
Carbohydrates, Proteins*	Bacterial volatile organic compounds, pesticides	Improve plant growth and health	(Sharifi and Ryu, 2020,

Polyacetic acid- Insecticides and Eco-friendly, protect the plant against insects
polyethylene glycol- pesticides
polyacetic acid

Neri-
Badang
and
Chakrab
orty,
2019)
(Sun et
al., 2020,
Graily-
Moradi
and
Asgari
Lajayer,
2021)

*; products commercially available.

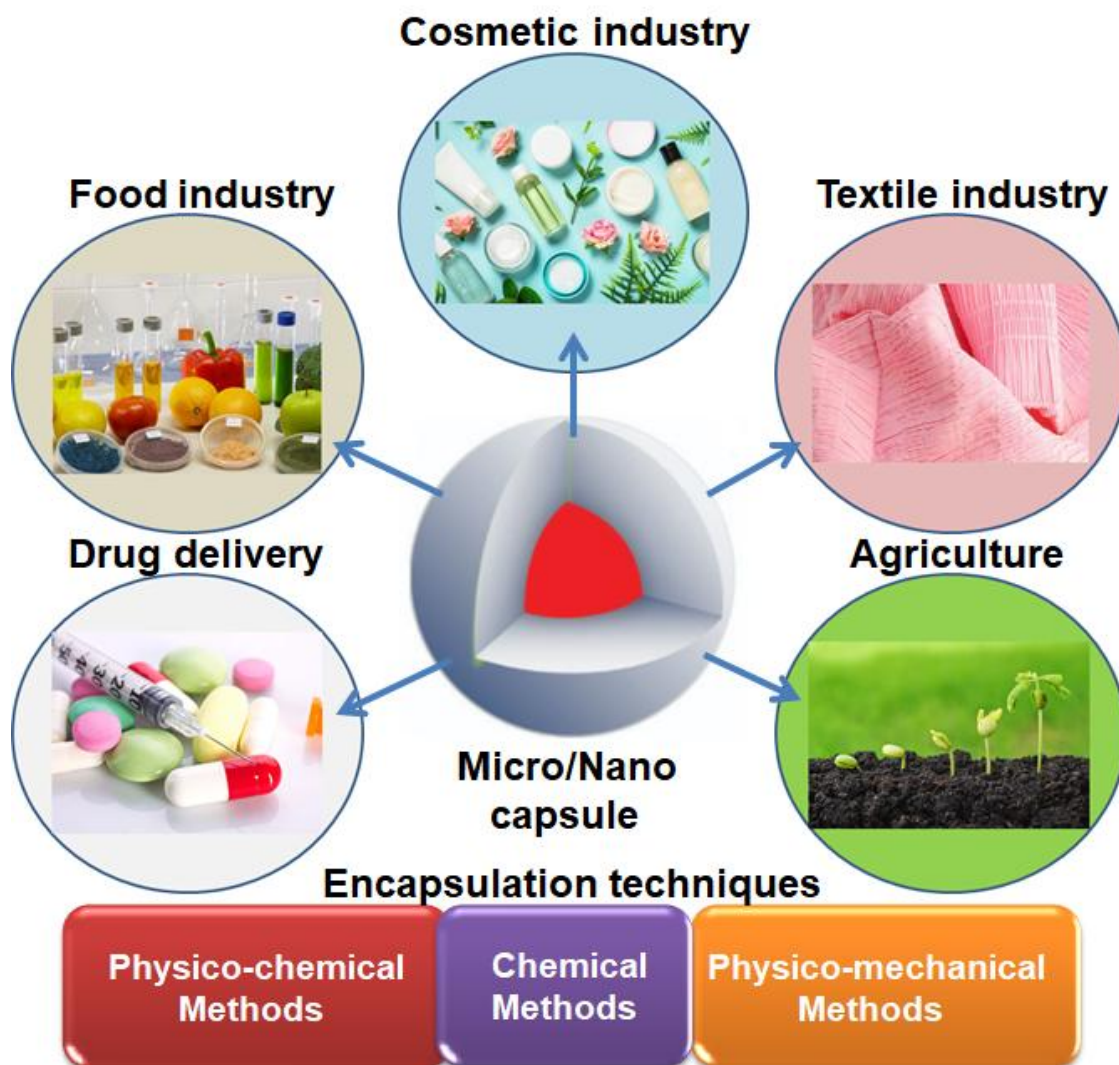


Figure 1. Schematics showing main techniques used for encapsulation in micro/nanocapsules with their applications in the field of drug delivery, agriculture, textile, cosmetics, and food industry.

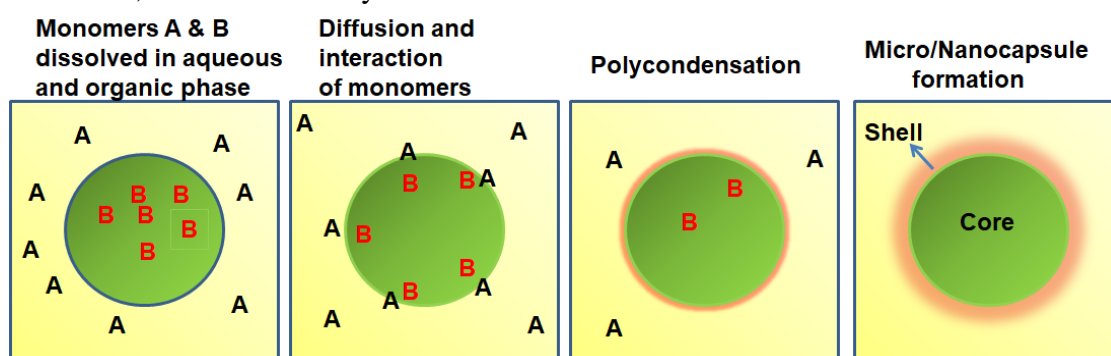


Figure 2. General representation of interfacial polymerization procedure which provides an example two monomers that react at the interface of a droplet, which lead to the formation of a capsule.

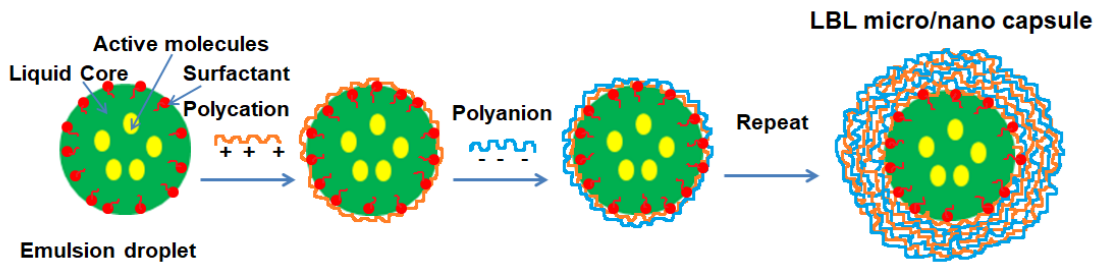


Figure 3. Diagram of micro/nano capsule formed via LBL technique. It illustrates alternate deposition or adsorption of polyelectrolytes with opposite charges through electrostatic attraction that results in a shell surrounding active material.

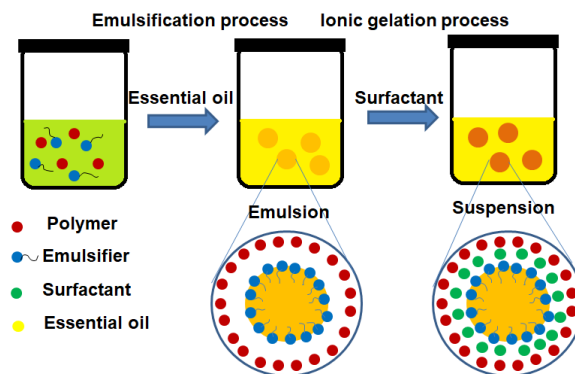


Figure 4. Microcapsules of polymer encapsulating of essential oil (EO) via ionic gelation process to examine their releasing property. In this process there is a formation of droplet possessing liquid (enzymes, active complexes or molecules, lipids, and cells) in core surrounded by shell with important gelling properties.

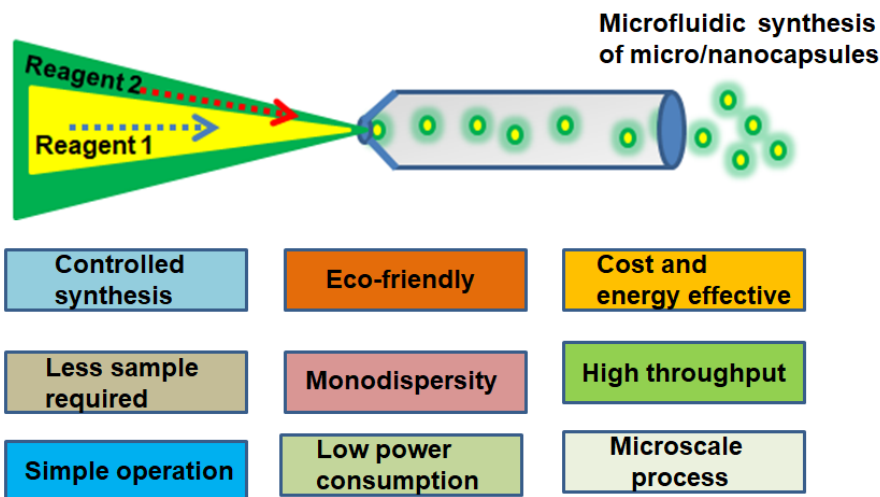


Figure 5. Schematics of microfluidics synthesis of micro/nanocapsules and their advantages. It is based on liquid flow through very tiny tubes with the diameter ranging from 5-500 μm to nanometer scale that fabricates micro/nanocapsules with high quality and required characters.

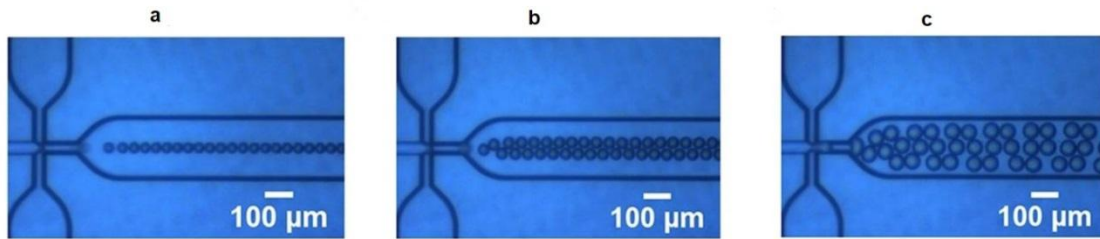


Figure 6. Droplets of limonene synthesized in microfluidic chip of controlled size (a–c) Optical microscopy o/w emulsions made at Qd (dispersed oil phase) and Qc (continuous water phase) of (a) Qd = 5 $\mu\text{L}/\text{min}$, Qc = 100 $\mu\text{L}/\text{min}$; (b) Qd = 10 $\mu\text{L}/\text{min}$, Qc = 100 $\mu\text{L}/\text{min}$ (c) Qd = 5 $\mu\text{L}/\text{min}$, Qc = 20 $\mu\text{L}/\text{min}$ (Redrawn from Thorne et al., 2019).

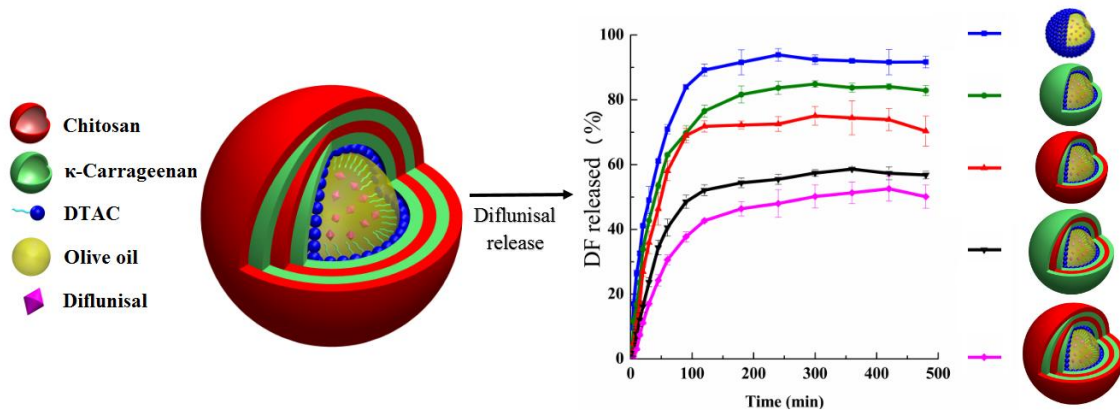


Figure 7. Profiles for release of DF(encapsulating difflunisal) that depends on time, from the NE and nanocapsules having core of oil with single, double, triple, and tetra layers of polyelectrolyte at 37 °C and 7.4 pH. 95% of encapsulated DF was discharged from NE. Nanocapsules with single, double, triple, and tetra layers delivered 85 %, 78%, 59%, and 53%, respectively (Redrawn from Rochin-Wong et al., 2018).

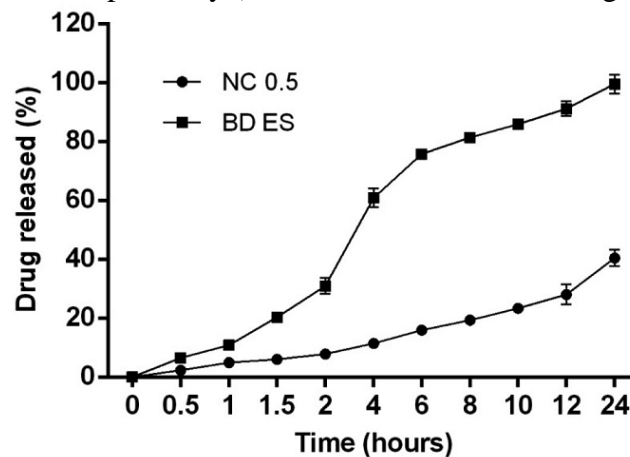


Figure 8. In-vitro profiles for release of BD from (NC) nanocapsules and BD-ES (ethanolic solution) via dialysis membrane technique(Redrawn from Janaíne Micheli Chassot, 2016).

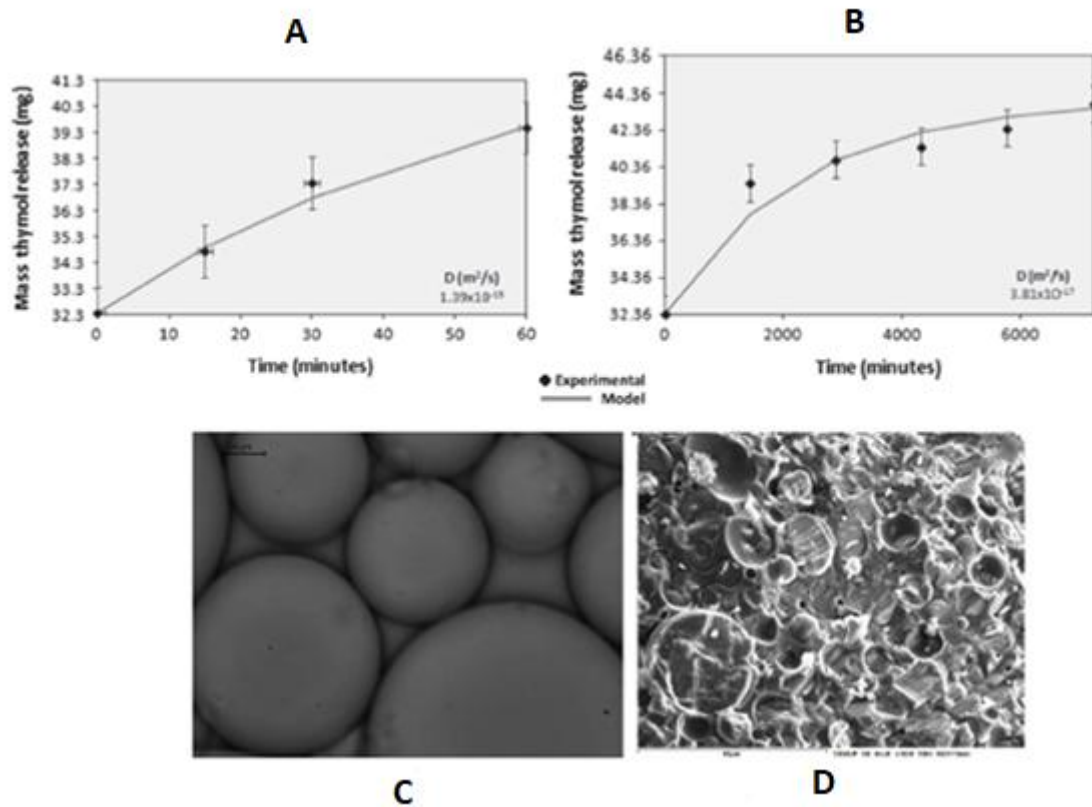


Figure 9. (A, B) Comparison between experimental and model results for thymol released from PLA microcapsules solution in first hour and for 5 days. Optical (C) and Cryo-SEM (D) microscopy images of PLA microcapsules with thyme oil for cosmetics applications (Redrawn from Martins et al., 2011).

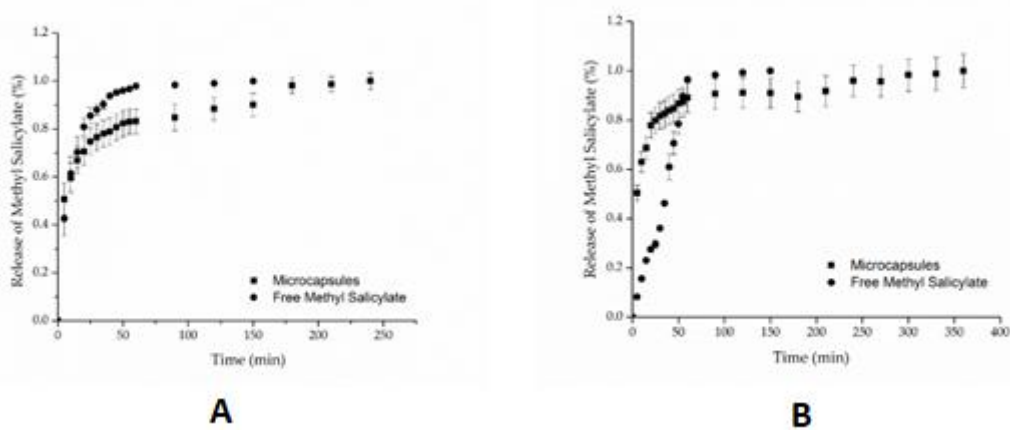


Figure 10. Controlled *in-vitro* methyl salicylate release out of polyamide and cotton and from samples containing (A) cotton (100%) and (B) polyamide (100%) modified by microcapsules has been studied. The effect relating to temperature gets clear after exposing samples to washing. There is a prominent release at higher temperature (50 °C) as compared to the lower temperature (37 °C) (Redrawn from Mendes et al., 2021),

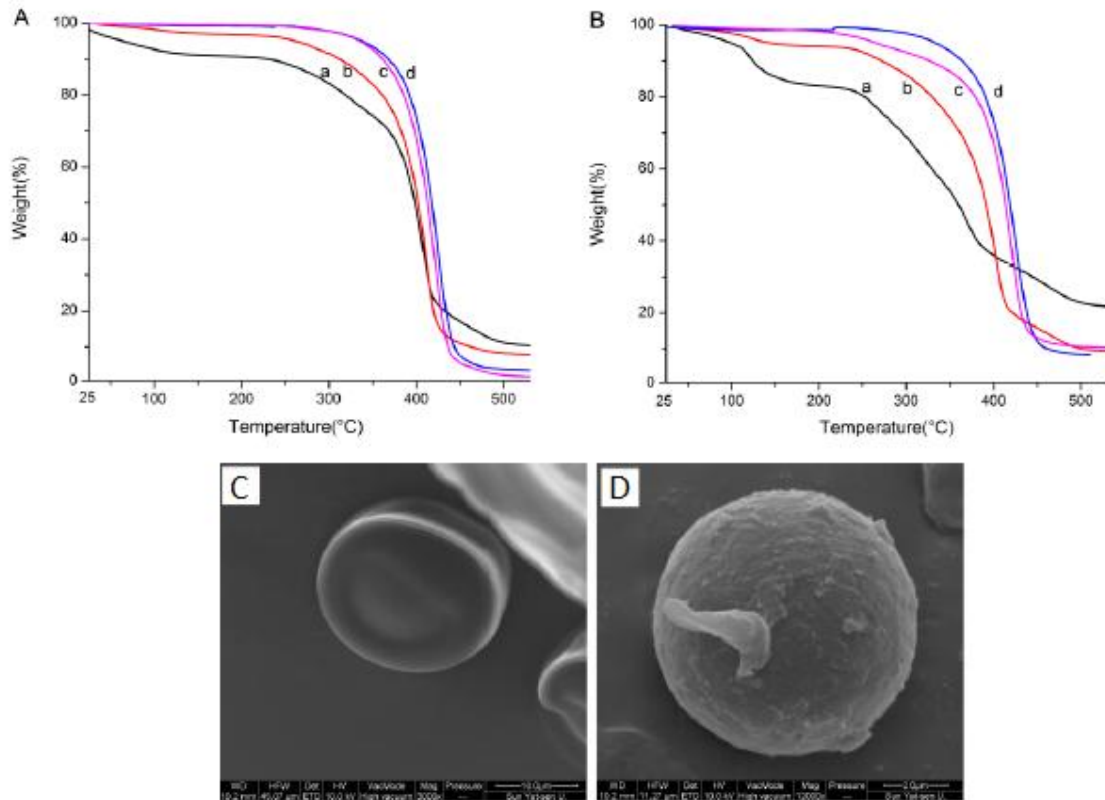


Figure 11. The thermogravimetric analysis (TGA) curve of microcapsules under different hardening conditions. (A) TGA curves of microcapsules hardened for different periods of time, (a) 2 h, (b) 4 h, (c) 8 h, (d) and 12 h, at pH 9.0, $T = 40\text{ }^{\circ}\text{C}$, 0.1 g genipin/g gelatin. (B) TGA curves of microcapsules hardened at different levels of pH, (a) pH 6.0, (b) pH 8.0, (c) pH 12.0, (d) and pH 10.0, at $t = 6\text{ h}$, $T = 40\text{ }^{\circ}\text{C}$, 0.1 g genipin/g gelatin. SEM micrographs of microencapsulated mustard seed oil. (C): Representatives of complete microcapsules without the cross-linking process. (D): Representatives of complete microcapsules with the cross-linking process for food applications (Redrawn from Peng et al., 2014).

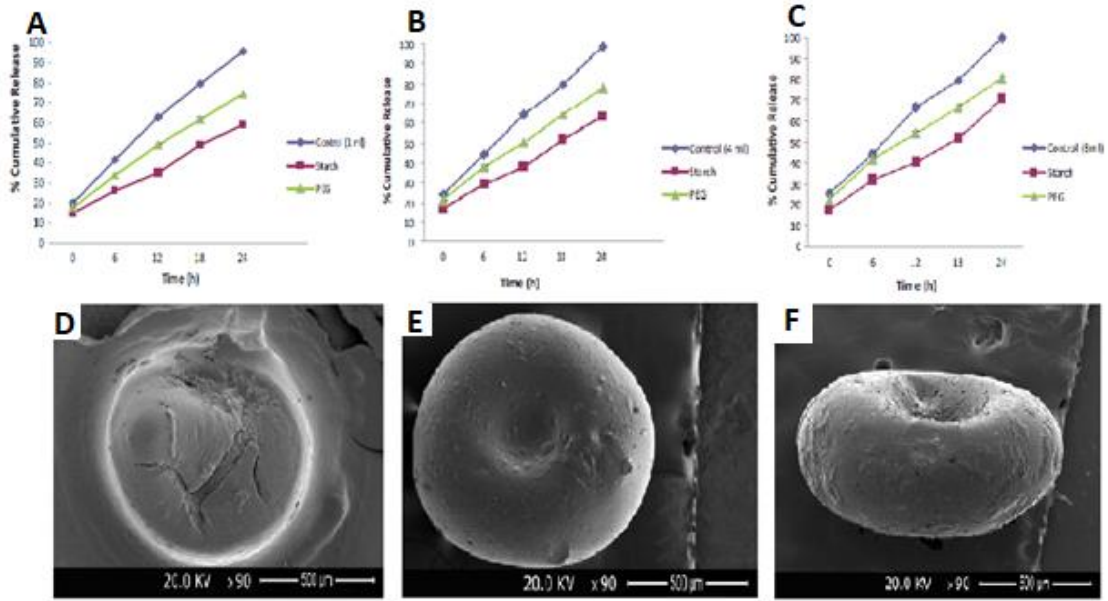


Figure 12. Effect of varying loading on release of neem Aza-A (A) 1 ml nanoemulsion along with starch and PEG coated, (B) 4 ml nanoemulsion along with starch and PEG coated and (C) 8 ml nanoemulsion along with starch and PEG coated. And Scanning electron microscopic image (D) control, (E) starch and (F) PEG for agriculture applications (Redrawn from Jayakumar et al., 2012)