

## MICROENCAPSULATION OF ESSENTIAL OILS OBTAINED FROM NATURAL HERBAL FOR USE IN THE FOOD INDUSTRY

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### Abstract

*Microencapsulation is a technological process in which solid, liquid or gaseous substances of small size are completely surrounded by individual polymeric coatings to avoid physical and chemical and for maintain the biological and physicochemical properties of core materials. Microencapsulation can be applied with success to entrap natural compounds, like vegetal extracts. Essential oils could have antibacterial and antifungal properties and have screened as potential sources of novel antimicrobial compounds. In food science and biotechnology, microencapsulation involves incorporating of natural ingredients, volatile additives, polyphenols, enzymes, bacteria into small capsules, giving them stability, protection and preservation, against nutritional losses, acting as antimicrobial agents. The aim of this literature review is to describe and functional properties and the benefits of various oils with antimicrobial activity obtained from natural herbals, application of encapsulated oils in food industry and microencapsulation techniques.*

**Key words:** biotechnology, microencapsulation, essential oils, antimicrobial activity.

### INTRODUCTION

Microencapsulation is used as a potential solution to solving punctual technological problems, most often leading the development of innovative processes or to the development of new products (Li SP et al., 1988; Finch CA, 1985; Arshady R. , 1993). Microencapsulation is more and more applicable in the field of biotechnology, especially in food and agriculture. In recent decades, encapsulation of active compounds has become a process of great interest and importance, being suitable for food, chemical, pharmaceutical and cosmetic ingredients. In the food field, microencapsulation is used to extend the shelf life of flavored, spices in dry mixtures; isolates additives used for baked goods, which are released only under the influence of heat; protecting vitamins; masking the taste, smell or color.

Microencapsulation can be successfully applied to encompass natural compounds such as essential oils or plant extracts containing polyphenols with antimicrobial properties well known for use in food packaging.

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them stability, protection and preservation, against nutritional losses, acting as antimicrobial agents.

### Essential oils from natural herbs and their benefits

Essential oils from natural herbal, the odorous, volatile products of an aromatic plant's secondary metabolism are well-known antimicrobial agents that could be used to control food spoilage and foodborne pathogenic bacteria. They have long been served as flavouring agents in food and beverages, and due to their content of antimicrobial compounds, they possess potential as natural agents for food preservation. The antimicrobial activity of essential oils is assigned to a number of small terpenoid and phenolic compounds, which also in pure form exhibit antibacterial or antifungal activity. Given the fact that consumers demand less use of chemicals in food products, more attention has been given to the search for naturally occurring substances able to act as alternative antimicrobials and antioxidants. Plant essential oils are becoming more popular as naturally derived antimicrobial agents.

#### *Oregano essential oil*

According to (Olmedo et al., 2014; Muriel-Galet et al., 2015), oregano essential oil contain ingredients, including carvacrol, thymol,  $\alpha$ -

terpinene,  $\gamma$ -terpinene, terpinen-4-ol, p-cymene,  $\alpha$ -terpineol, and sabinene. Carvacrol and thymol constituting about 78% to 82% of the total oil, are the principal phenolic compounds responsible for antioxidant and antimicrobial activities. According to the researchers, adding a higher amount of oregano oil increased the retention of  $\alpha$ -tocopherol in the meat (Botsoglou et al., 2003). According to the studies, oregano oil is one of the oils with high antimicrobial activity, its phenolic components permeate and depolarize the bacterial cytoplasmic membrane, leading to cell death. According to Rodriguez-Garcia et al. (2015) the antimicrobial efficacy of essential oil is in the order: oregano/ clove / coriander/ cinnamon > thyme > mint > rosemary > mustard > cilantro/ sage.

Asensio et al. (2015), found that completion of the oil in organic cottage cheese decreased, during storage, chemical deterioration. Also oregano essential oils hold antifungal and insecticidal properties and can be used for the prevention of neurodegenerative excitement (Almeida et al., 2013). Oregano oil due to its preservative effects and pleasant flavor is used as a food ingredient. *Basil oil* according to researchers has a powerful medicinal value (Baliga et al., 2013). Its main components of phenolic and terpenoid derivatives include methyl eugenol (42.58%) followed by caryophyllene (26.88%) and eugenol (10.66%). Basil oil has antibacterial, antioxidant (Chanwitheesuk et al., 2005) and antifungal (Kumar et al., 2010) properties. Khanna et al. (2010) and Baliga et al. (2013) found that basil oils can also inhibit cholesterol synthesis and improve digestive performance. According to Sutaphanit and Chitprasert (2014) microencapsulation of basil oil with gelatin insured protection against chemical and physical loss under fast storage conditions at 60°C for 49 days. *Rosemary oil* (*Rosmarinus officinalis* L.) Rosemary is the most used medicinal and aromatic plant in the world, because of its phenolic compounds and essential oil (Rozman and Jersek, 2009). The research related to rosemary essential oil has mainly focused on its , antifungal (Soylu et al., 2010), antibacterial (Jiang et al., 2011), anticancer (Degner et al., 2009), insecticidal (Zoubiri and Baaliouamer, 2011), gastric, antiseptic, anti-inflammatory,

antioxidant and antiviral properties (Barni et al., 2012). Also, rosemary oil helps in perception improving (Moss et al., 2003). Microencapsulation of rosemary oil accomplish functional activity with high retention volatiles. *Cinnamon leaf oil* is appreciated for its flavor in addition to its antimicrobial properties (Singh et al., 2007; Ayala-Zavala et al., 2008). Antifungal and antioxidant properties of cinnamon leaf oil are due to volatile components such as eugenol and cinnamaldehyde (Combrinck et al., 2011). Cinnamon leaf oil it has been found to have antimicrobial (Matan et al., 2006), anti-inflammatory (Gunawardena et al., 2014) and antidiabetic properties (Ping et al., 2010). The U.S. Food and Drug Administration consider cinnamon oil as a Generally Recognized as Safe compound (Tzortzakis, 2009). Microencapsulation of cinnamon leaf and garlic oil with  $\beta$ -cyclodextrin reveal good antifungal activity against *Alternaria alternata*. Due to improved stability, solubility, and bioavailability, cinnamon leaf and garlic oil microcapsules could have important applications in the food industry (Ayala-Zavala et al., 2008).

*Thyme oil*. Thyme is a known source of essential oil and also a phytonogenic food additive. Essential oil of thyme is widely used in food and the flavor industry, as well as in the manufacture of cosmetics and perfumes. Its antimicrobial and antioxidant activities are mainly attributed to the presence of carvacrol, cinnamaldehyde, thymol, geraniol and eugenol (Sipailiene et al., 2006; Navarrete et al., 2010). Jouki et al. (2014) have found that the incorporation of essential thyme oil into edible films has increased safety and the shelf-life of ready to eat foods.

Essential oils have the potential to be used in the food industry as a preservative to increase the shelf life and prevent damage of products because they are a source of natural antimicrobial substances. The essential oils could also reduce side effects by their replacement of chemical preservatives (Abhishek K.D. et al., 2016). A variety of molecules derived from essential oils have bioactive properties with antibacterial activity that can be used directly in food products. In Table 1 few examples of latest studied essential oils and their composition and properties are given.

Table 1. Essential oils from natural herbal, components and activity

Oil type	Species	Components	Properties of essential oils	References
Oregano oil	<i>Origanum vulgare</i>	Carvacrol, thymol, $\alpha$ -terpinene, $\gamma$ -terpinene, terpinen-4-ol, p-cymene, $\alpha$ -terpineol, and sabinene.	antioxidant, antifungal	Olmedo et al., 2014; Muriel-Galet et al., 2015
Basil oil	<i>Ocimum basilicum</i>	Methyl eugenol, caryophyllene, eugenol	antioxidant, antibacterial, antifungal	Baliga et al., 2013; Chanwitheesuk et al., 2005; Kumar et al., 2010
Rosemary oil	<i>Rosmarinus officinalis L.</i>	Camphene, ( $\alpha$ & $\beta$ -Pinene), limonene, & camphor	antifungal, antibacterial	Rozman and Jersek, 2009; de Barros Fernandes et al., 2014; Fernandes et al., 2013; Soylu et al., 2010; Jiang et al., 2011
Cinnamon leaf oil	<i>Cinnamomum zeylanicum</i>	Eugenol, cinnamaldehyde	antifungal, antioxidant	Ayala-Zavala et al., 2008; Singh et al., 2007; Combrinck et al., 2011
Thyme oil	<i>Thymus vulgaris</i>	Carvacrol, cinnamaldehyde, thymol, geraniol and eugenol	antioxidant, antimicrobial	Sipailiene et al., 2006, Navarrete et al., 2010

### Materials used as encapsulation matrices

**Carbohydrates.** For microencapsulation the most commonly used shell materials are carbohydrates such as starches and maltodextrins. Carbohydrate based materials because they have poor interfacial properties have to be chemically modified to improve surface activity.

Hydrolysed *starches* are depolymerised ingredients produced by hydrolysing starch with acid and/or enzymes. These wall materials offer the advantage of being inexpensive; low viscosity at high solids; and excellent protection to encapsulated core materials. The degree of protection is directly related to the dextrose equivalent of the hydrolysed starch, higher-dextrose systems are less permeable to oxygen and result in powders with higher encapsulation efficiencies (Dalglish D.G., 2006).

*Cyclodextrins* have also been used in encapsulation of food oils and flavours. They are cyclic molecules containing six ( $\alpha$ -), seven ( $\beta$ -) or eight ( $\gamma$ -) glucose monomers that are produced from starch. These monomers are connected to each other, giving a ring structure that is relatively rigid and has a hollow cavity with the ability to encapsulate other molecules. Many reports have demonstrated that inclusion complexes are virtually completely stable to oxidation compared to other wall materials. Reineccius et al. found that  $\gamma$ -cyclodextrin generally functioned better than ( $\alpha$ -) and  $\beta$ -cyclodextrins in terms of initial flavor retention.

Encapsulation in  $\beta$ -cyclodextrin is a method for controlling the odor and reactivity of active compounds during the release of natural antimicrobial compounds.  $\beta$ -cyclodextrin is a cyclic molecule made up of 7 D-glucose monomers linked via a cone (1,4) bond. A hydrophobe is a cavity, while the outer face is hydrophilic. These properties have made  $\beta$ -cyclodextrin an option for encapsulation from several organic and inorganic compounds. Encapsulation in  $\beta$ -cyclodextrin is considered as a molecular complex, in which the hydrophobic active constituents of the essential oil can interact in the hydrophobic cavity of  $\beta$ -cyclodextrin, indicating that when forming the capsule, the outer molecule is hydrophilic.

**Proteins.** Functional properties of the proteins, including the ability to interact with water solubility, film forming and emulsifying and stabilizing emulsion droplets, have many of the desirable characteristics for a wall material. One of the commonly used proteins is gelatine. According to research, in recent years for the potential of new wall materials, for the encapsulation of flavors and oils, were studied other proteins, especially soy and milk proteins, such as whey protein concentrate, skimmed milk powder and caseinates. These proteins change their structure during emulsification through unfolding and adsorption at the oil water interface and by forming resistant multilayer around oil droplets and also with the help of repulsive forces, make significantly stable emulsions which are critical for encapsulation purposes. Investigations have proven proteins to function well for oils.

**Gums.** Acacia gum, usually known as gum arabic, due to its excellent emulsifying properties is mostly used gum. Due their emulsion stabilization and film forming properties of gums make them a suitable microencapsulation agent. The constituent of gum Arabic are L-rhamnose, D-glucuronic acid, L-arabinose and D-galactose with approximately 2% protein, which is responsible for emulsifying properties of gum arabic (Dickinson E., 2003). According to Krishnan et al. (2005) gum arabic compared maltodextrins and modified starch, have been found to be a better wall material for encapsulation of cardamom oleoresin, the resulting microcapsules exhibit a free-flowing character. Gum arabic have shown good properties as wall material for encapsulation of cumin oleoresin by spray-drying (Amr M. et al., 2015). Usually, gum arabic is preferred because it produces stable emulsions with most oils over a wide pH range and forms a visible film at the oil interface. Gum arabic because of this emulsifying efficiency, has been usually used to encapsulate lipids. Gum Arabic is ideally for the microencapsulation of lipids because of both its surface activity and its film forming properties.

### **Microencapsulation techniques for essential oils**

There are several methods of producing the microcapsule using different types of coating materials as well as generating particles of different sizes, thickness and core permeability, thus adjusting the release. Generally, these techniques are divided into two categories: physical methods and chemical methods. Chemical methods can also be subdivided into physico-chemical and physico-mechanical techniques.

*Emulsification* is a method used in a extensive variety of pharmaceutical and food products. Emulsion technology is an important step in the microencapsulation of oils. In general, emulsification is applied to encapsulate bioacids in aqueous solutions, which can be directly used in liquid form. Emulsions are prepared by homogenizing the oil, water and emulsifier, using a homogenizer (Augustin et al., 2006).

Microencapsulation of essential oils by using *supercritical fluid technology* is of great relevance to the pharmaceutical, cosmetic and food industry. This method has several inherent advantages: nontoxicity, mild solvent removal, product degradation, and the process utilizes a wide variety of materials that produce controlled particle sizes and morphologies.

*Spray drying* is a physico-chemical method and is the most frequently used technique to encapsulate flavors. Spray drying can be described as a simple process, capable of producing a wide range of good yield microcapsules, including microcapsules loaded with aromatic oils or aroma Tonon et al. (2011) found that spray-drying is a technique that involves the atomization of emulsions into a drying chamber at a relatively high temperature, which leads to water evaporation and, therefore, crust formed at fast rate and quasi-instantaneous entrapment of oil. The process involves four steps: preparing a dispersion or emulsion; homogeneity of dispersion; atomizing the feed emulsion; and dehydration of the atomized particles. Spray drying is the microencapsulation technique most commonly used in the food industry and is used to encapsulate a wide range of ingredients (Beristain et al., 2001, 2002).

The *coacervation* technique can be divided into two main groups: aqueous and organic. Aqueous phase coacervation can only be used to encapsulate water-soluble materials. Organic coacervation allows the encapsulation of a water-soluble material but requires the use of organic solvents.

*Freeze-drying* is a simple process and is used for the dehydration of almost all heat-sensitive materials and aromas like oils. Before drying, the oil is dissolved in water and frozen between -90°C and -40°C (Heinzelmann et al., 2000; Amr M. et al., 2015) and then the surrounding pressure is reduced and enough heat is added to allow the frozen water in the material to sublimate directly from the solid phase to the gas phase (Oetjen and Haseley, 2004).

*In situ polymerization* according to the researchers has become the most used method for the preparation of microcapsules and functional fibers. In situ polymerization is a microencapsulation method which, by adding a reactant inside or outside the core material,

leads to the formation of a wall (Amr M. et al., 2015). *In situ* polymerization differs from other encapsulation polymerization processes because no reactant is included in the base material. According to the researchers, Amr M. et al. (2015) microcapsule formation is performed with an oil emulsion in a melamine-formaldehyde resin solution and sonication process to emulsify the oil in the aqueous phase, then the resin is added with stirring and then the pH of the emulsion to the acid finally forming the microcapsule shells. By using this method, the melamine reaction with formaldehyde is promoted at the oil droplet interface, producing a crosslinked melamine-formaldehyde polymer film as a microcapsule shell.

## CONCLUSIONS

Microencapsulation is an important tool for the preparation of high quality oil products and health benefits in the food industry to improve their chemical, oxidative and thermal stability. Essential oils are natural products that consist of complex mixtures of many volatile molecules. Essential oils due to their source of natural antimicrobial substances are used in the food industry as a preservative to prevent damage and to increase the shelf life of products. Despite their many applications, essential oils are very sensitive to environmental factors when used as such, and encapsulation is a relevant alternative that enhances essential oils stability. Various microencapsulation techniques have been successfully used to achieve this purpose.

According to studies, the most commonly used matrices for microencapsulation are carbohydrates such as maltodextrins and starches because it offers the advantage of being cheap; have low viscosity and excellent protection for encapsulated base materials.

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