https://doi.org/10.17221/200/2023-CJFS

Applications of polycaprolactone in the food industry: A review

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Citation: Oney-Montalvo J.E., Dzib-Cauich D.A., Ramírez-Rivera E.J., Cabal-Prieto A., Can-Herrera L.A. (2024): Applications of polycaprolactone in the food industry: A review. Czech J. Food Sci., 42: 77–84.

Abstract: The food industry is always looking for ways to innovate in elaborating and packaging food products to ensure the consumer receives the highest quality. The new proposals include the use of polycaprolactone (PCL), a commonly used biopolymer that is soluble in many organic solvents. The PCL features can be modified by forming blends with other polymers and bioactive molecules to expand their applications in the food industry. For instance, there have been developments in packages and active substances that incorporate microcapsules based on PCL. This review explores the applications of PCL in the food industry, encompassing its role as a biodegradable active package and as an encapsulating agent. The review underscores the potential of this polymer in the context of the food industry.

Keywords: active package; agroindustry; biopolymer; microcapsule

The food industry requires novel alternatives to produce and package food and beverages to increase shelf life (Firouz et al. 2021). Nowadays, more consumers demand high-quality foods to benefit their health. Moreover, it is urgent to stop using non-compostable package materials due to the global pollution crisis (Hassoun et al. 2022; Rolnick et al. 2022).

One strategy for facing these challenges is to use biodegradable polymers, which can be synthesised for chemical methods or obtained from natural sources (Baranwal et al. 2022). Naturally derived polymers like polysaccharides and proteins have attractive antioxidant and antimicrobial properties (Tajeddin et al. 2020). These materials are readily biodegradable in a short period (a few months or weeks) compared to synthetic polymers (Polman et al. 2021). Some natural polymers commonly used in the food industry are chitin, chitosan, cellulose, alginate, starch, and cyclo-

https://doi.org/10.17221/200/2023-CJFS

dextrin (Baranwal et al. 2022), which possess biodegradability properties.

While only a few synthetic polymers met the critical feature of biodegradability, polycaprolactone (PCL) has gained attention in recent years thanks to its biocompatibility and biodegradability. PCL can be synthesised from monomers obtained from renewable sources and mixed with other polymers because of its miscibility (Labet et al. 2009). These features allow the creation of biomedical devices that eventually become commercially available (Mohamed et al. 2015).

The PCL has found applications in the food industry, such as designing active packaging that prevents food spoilage (Alix et al. 2013; Thakur et al. 2021). In another approach, encapsulating systems based on PCL can be designed, where capsules filled with bioactive components are incorporated into food products to increase their nutraceutical properties and added value (Ezhilarasi et al. 2012).

Recent research has focused on improving the PCL properties to make it suitable for fabricating active food packages and as an additive to formulate functional foods (Thakur et al. 2021). Although most review papers explore the use of PCL as a device for drug release and for the fabrication of tissue engineering matrices (Salehi et al. 2021; Siddiqui et al. 2021), this review highlights the potential of PCL in the food industry as an active package material and encapsulating agent.

PHYSICOCHEMICAL PROPERTIES OF POLYCAPROLACTONE

PCL is an aliphatic polyester obtained from the ring-opening polymerisation of ε-caprolactone (Figure 1). This polymeric material possesses properties similar to commodity plastics, and it has the advantage of being biodegradable. This feature offers the possibility of replacing the non-biodegradable synthetic plastics used in daily activities (e.g. bags, packages, bottles, etc.) (Thakur et al. 2021).

PCL is a semicrystalline polymer with good mechanical strength and flexibility. Its molecular weight ranges from 37 000 to 80 000 g·mol^{-1}; density is around 1.07 to 1.2 g·cm^{-3}; transmission rate to water vapour is 177 g·m^{-2} per day at 25 °C; and relative humidity is 50%. The permeability coe of O_2 and CO_2 are around 332.5 and 2 730 cm^3 STP·cm^{-2}·h^{-1}·bar^{-1} (STP – Standard Temperature and Pressure), respectively, at 20 °C and relative humidity of 50%. The fusion temperature is about 60 °C, while the degradation time is less than 24 months at a temperature of 24 °C (Thakur et al. 2021). These properties have been utilised to create biodegradable materials from PCL, which can be utilised in different industries, such as pharmaceuticals. For example, Potrč et al. (2015) designed a nanocarrier-based PCL nanofiber to deliver non-water-soluble drugs. Moreover, PCL properties can be modulated by forming blends with other polymers or bioactive molecules, expanding its potential applications (Baptista et al. 2020). The creation of biodegradable films added antioxidant compounds that can be used as packaging that prolong the shelf life of the food. For example, packages with antibacterial properties were obtained by blending PCL with chitosan, having inhibition rates of polycaprolactone/chitosan of 39.2–99.9% against *Escherichia coli*, while the inhibition rate against *Staphylococcus aureus* was 40.9–99.9%. The antimicrobial effect is associated with chitosan being able to chelate the essential trace metals needed for microbial metabolism, decreasing their availability to microbes, which will result in the inhibition of their metabolic machinery and, eventually, their death (Zeng et al. 2021). In addition to its antimicrobial effect, chitosan also stands out due to its antioxidant properties and its non-toxicity, biodegradability, and biocompatibility, making it an ideal option for packaging food products (Ke et al. 2021).

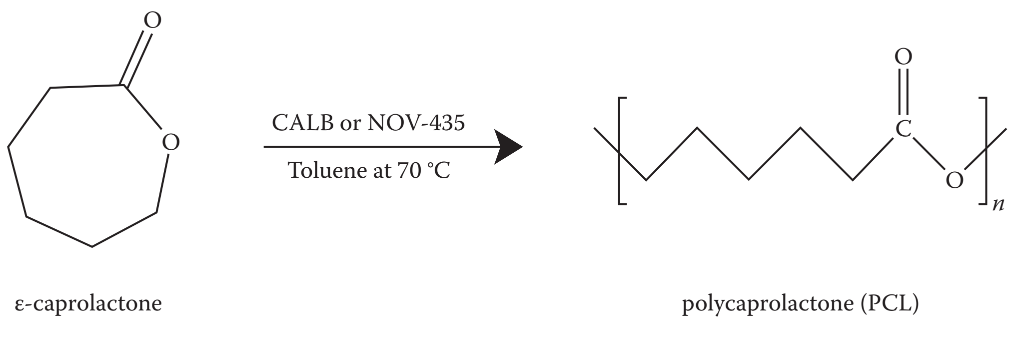

Figure 1. Reaction to obtain polycaprolactone from ε-caprolactone

CALB – *Candida antarctica* lipase B; NOV-435 – Novozym®-435

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POLYCAPROLACTONE NANOPARTICLE MANUFACTURING PROCESSES

The manufacture of polymer nanofibers from PCL has been carried out using different techniques, including chemical, physical, thermal, and electrostatic fabrications. Being the electrospinning technique one of the most used and studied; this is an interesting technology to produce ultrafine fibres with a diameter ranging from micrometres to sub-nanometers; this is achieved by applying an electrostatic field to a polymer solution (Mochane et al. 2019).

Preparation of PCL nanocomposites by electrospinning method to provide a smooth surface of nanocomposite materials with a high surface-volume ratio. Additionally, nanoparticles can be incorporated into the PCL matrix, altering the native properties of the PCL. Electrospinning is widely used in biomedical technology where most biopolymers of different structural compositions are electrospuned to comply with the requirements for biomedical applications. This opens the door to future applications of this technology in other sectors, such as the food industry, where, compared to the biomedical sector, it is just beginning to be considered (Mochane et al. 2019).

POLYCAPROLACTONE IN THE FOOD INDUSTRY

There are two main applications for PCL in the food industry: bioactive packaging and encapsulating agents (Figure 2). The literature reports that active packages based on PCL aim to preserve meat and dairy products (Plackett et al. 2006; Vargas-Romero et al. 2021). Alternatively, PCL microcapsules have encapsulated bioactive compounds (polyphenols, terpenoids, and tocopherols). The capsules could be added to food formulations to obtain enriched food products. Also, they can be loaded with additives to improve the growth of plants, which are supplied through the soil (Cesari et al. 2020; El-Messery et al. 2021).

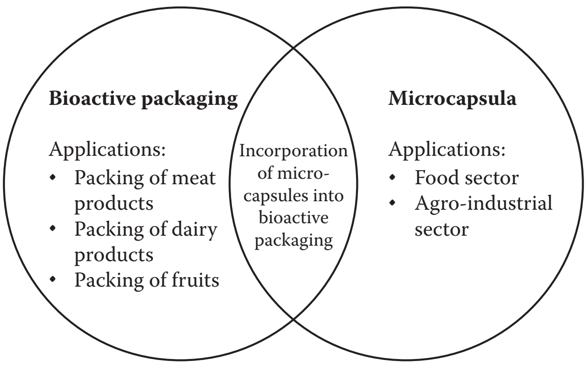

Figure 2. Applications of polycaprolactone in the food industry

POLYCAPROLACTONE AS ACTIVE BIODEGRADABLE PACKING

Packaging plays a key role not only in the preservation of food products but also in storage and transportation (Wyrwa et al. 2017). Unfortunately, most plastic packages are designed for single use only. As petroleum-derived materials, the biodegradation of plastic packages takes hundreds of years because these are unrecognised by the organisms that normally break organic matter down, leading to severe contamination issues (Mangaraj et al. 2018). A better alternative is using biodegradable active packaging (Motelica et al. 2020), which are made of biopolymers, being popular biodegradable polymers used to fabricate bio packages: the poly(lactic acid) (PLA), poly(acrylic acid) (PAA), chitosan, and PCL (Muller et al. 2017).

These include biocompatibility, adjustable degradation kinetics, and the possibility of introducing functional groups (by chemical methods) to modify the hydrophobic nature of PCL (Mohamed et al. 2015; Thakur et al. 2021). On the other hand, the disadvantage is that PCL has a low melting point (60 °C) compared with other polymers and its complex and expensive production, in addition to an acceleration of its decomposition due to the presence of enzymes and hydrolytic conditions that degrade the PCL.

PCL can be further enhanced by adding natural elements such as essential oils, bacterial sources, or natural extracts to give antioxidant, antifungal, and antibacterial properties. (Motelica et al. 2020). For example, Figueroa-Lopez et al. (2018) fabricated ultrathin PCL fibres with black pepper oleoresins; then the fibres were deposited onto a gelatine film to obtain food packaging with antibacterial properties. In another work, Zou et al. (2020) incorporated halloysite nanotubes with chlorogenic acid in electrospun chitosan/PCL fibres. This system was designed to ensure a long-term sustained release of chlorogenic acid, thereby increasing the shelf life of perishable goods. PCL has been widely used in food packaging design for different types of food products, as described in (Table 1). In these examples, active substances like propolis, citric acid, and naringin are incorporated into the packaging to give antimicrobial activity and prolong the shelf life of the food.

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Table 1. Applications of polycaprolactone (PCL) as biodegradable active packaging in the food industry

Food product	Polymer-based packaging	Active substance	Reference
Cheese	PCL/PLA	allyl isothiocyanate	Plackett et al. (2006)
Chicken	PCL/chitosan	citric acid	Sogut et al. (2019)
Cheese	PCL	grapefruit seed extract	Lyu et al. (2019)
Not defined in the section of 'Food product'	ECL/PCL/GEL	*Zataria multiflora* essential oil and zinc oxide nanoparticle	Beikzadeh et al. (2019)
Pork	PCL/chitosan	propolis	Vargas-Romero et al. (2021)
Beef	PCL	Ag-doped halloysite Ag-doped montmorillonite	İlaslan et al. (2022)
Salmon	PCL	linalool	Li et al. (2022)
Blackberry	PLA/PCL electrospun nanofibers	oregano essential oil-loaded β-cyclodextrin	Shi et al. (2022)
White cheese	PCL/GEL	cumin essential oil	Shanbehzadeh et al. (2022)
Not defined in the section of 'Food product'	PCL/casein electrospun nanofibers	green tea essential oils	Yavari-Maroufi et al. (2023)
Foodstuff	PCL	peptide LfcinB $(21–25)_{Pal}$	Rodríguez-Sánchez et al. (2023)
Strawberries	zein/PCL electrospun nanofibers	β-caryophyllene	Ullah et al. (2023)
	PCL/ECL nanofibrous film	natamycin and trans-cinnamic acid	Wu et al. (2024)

ECL – ethylcellulose; GEL – gelatin; PLA – poly(lactic acid)

Meat products. The quality of meat products is affected by microorganisms, and the oxidation of lipids and proteins can compromise their safety (Fang et al. 2017). Active packages based on PCL have been proposed as an alternative to maintaining the safeness and quality of meat products (Fang et al. 2017; Vargas-Romero et al. 2021; Li et al. 2022). Vargas-Romero et al. (2021) designed active packaging with antioxidant and antimicrobial properties to maintain the freshness of pork loin chops for more time. For this purpose, Colombian propolis was added to chitosan/PCL films. This study, which was conducted over 20 days at 4 °C, showed that pork's colour deterioration and bacterial proliferation were slowed down by four days due to propolis's antimicrobial and antioxidant properties. By contrast, Sogut and Seydim (2019) used grape seed extract (15% *w*/*w*) as the active substance in nanocellulose-containing PCL-chitosan bilayer films. The effectiveness of this material was tested on chicken breast fillets, which were stored in refrigeration for 15 days. After this period, the physicochemical and microbiological properties were evaluated. The samples packaged with bilayer films showed a lower level of lipidic peroxidation and minimum proliferation of aerobic mesophilic bacteria and coliform compared to the control. The study concluded that the bilayer PCL/chitosan films successfully released the functional groups of grape seeds, making them an attractive option for active packaging. İlaslan et al. (2022) developed PCL films containing silver-doped nanoclays to prevent microbiological contamination while storing ground beef meat. Results exhibited strong antibacterial activity against pathogens and a bacteriostatic effect against bacterial populations that occur naturally in meat. Li et al. (2022) also used PCL films to package fresh salmon. Their research modified the films with linalool, which was released continuously during storage. This packaging reduced the decomposition rate of fresh salmon.

Dairy products. Dairy products are rich in nutrients, with many advantages for frequent consumers. However, these products are susceptible to contamination by microorganisms, sometimes pathogens, which can negatively affect their quality and safety. To address this issue, active packaging has demonstrated its capability to prevent contamination without compromising the quality of dairy products (Alizadeh et al. 2020). For example, Plackett et al. (2006) incor-

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porated 10% *w*/*w* of α-cyclodextrin-allyl isothiocyanate (α-CD-AITC), an antifungal agent, in PCL/PLA films for cheese packaging. This material effectively reduced fungus contamination thanks to the controlled release of the α-CD-AITC. Shanbehzadeh et al. (2022) developed PCL/gelatin nanofiber-based films to package Iranian white cheese. The films were modified with cumin essential oil (CEO) and zinc oxide (ZnO) nanoparticles to add antibacterial properties. The strongest antibacterial capability against *S. aureus* was observed when 3% of CEO and ZnO were used. Despite this, the sensory attributes of cheese were not affected by the incorporation of CEO and ZnO. Similarly, Lyu et al. (2019) added grapefruit seed extract (GSE) as an antimicrobial agent in PCL films for packaging cheese. The antimicrobial activity of PCL films increased as the GSE extract content increased.

Fruits. Applications that have been reported within the fruit sector have focused on 2 products, strawberries and blackberries, obtained in all cases positive data that demonstrated the potential of PCL to be used as bioactive packaging material with the ability to contribute to the conservation of fruits after harvest (Shi et al. 2022; Ullah et al. 2023; Wu et al. 2024). The aforementioned was achieved by adding different antioxidant compounds (oregano essential oil, β-caryophyllene, natamycin, and trans-cinnamic acid) that contributed to the increased shelf life of strawberries and blackberries. As a result of good inhibitory activity against *E. coli*, *S. aureus*, and *Botrytis cinerea*, without affecting the organoleptic quality of the fruits (Shi et al. 2022; Ullah et al. 2023; Wu et al. 2024).

In the examples mentioned above, both applications in the meat, dairy, and fruit sectors, PCL's main function in developing active packaging materials is the ability to improve the functional and technological properties of many active materials. For example, when PCL is included in a mixture with PLA, there is an increase in thermal stability and toughness measured in the form of impact strength of the biopolymer that is produced (Ostafinska et al. 2015).

POLYCAPROLACTONE AS ENCAPSULANT AGENT

Microcapsules have a core-shell configuration where active substances are usually polyphenols, organic acids, and essential oils (Figure 3) (Peanparkdee et al. 2016). This system offers the advantage of controlling the release of active components and developing functional foods with improved sensory properties. Microcapsules can be applied to improve dairy, meat, and cereal products, to name a few (Calderón-Oliver et al. 2022). The pharmaceutical industry was the first to use PCL microcapsules to protect those drugs sensitive to chemical degradation and as a controlled releasing system (Pohlmann et al. 2013). After that, PCL microcapsules were applied to the food industry by designing functional food packages. Earlier, Park and Kim (2005) encapsulated α-tocopherol in the PCL matrix; this antioxidant is the main component of vitamin E and is widely used to enrich food products.

Table 2 describes some reports about using PCL as an encapsulating agent in the food and agriculture industries. The protocol is defined based on the compound's physicochemical properties that need to be encapsulated. A double emulsion and evaporation methodology was followed for polyphenols, while a microemulsion polymerisation reaction encapsulated geraniol (a monoterpenoid).

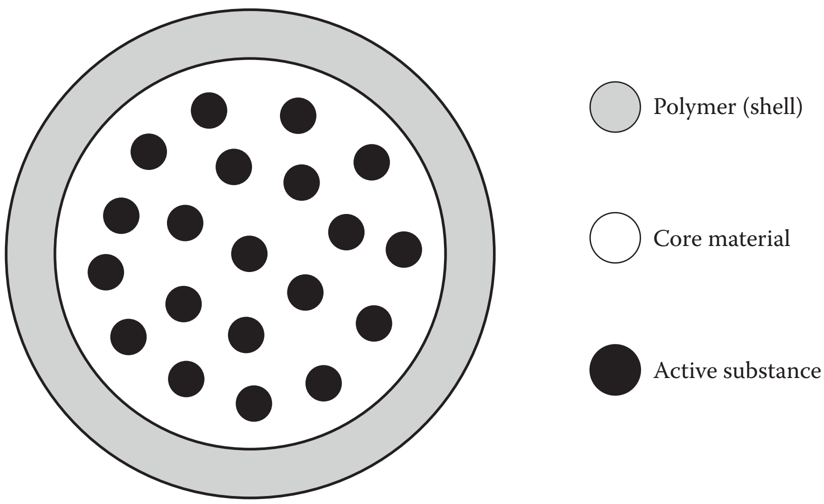

Figure 3. Core-shell configuration for microcapsules carrying active compounds

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Table 2. Applications of polycaprolactone as an encapsulating agent in food and agricultural industries

Industry	Encapsulated compound	Encapsulation method	Reference
Food	polyphenols from olive leaves	double emulsion/evaporation	El-Messery et al. (2021)
	geraniol	microemulsion polymerisation	Carniel et al. (2021)
Agroindustry	citric acid and naringenin	double emulsion/evaporation	Cesari et al. (2020)

Polycaprolactone as microencapsulating agent in the food industry. Most bioactive substances are sensitive to pH, temperature, and humidity conditions. In this sense, the protection of bioactive substances is crucial to preserve the bioactive compounds until their consumption. EI-Messery et al. (2021) extracted polyphenols from olive leaves loaded in PCL microcapsules and added them to a yoghurt formulation. The *in vitro* digestion test revealed a higher content of polyphenols in yoghurt with PCL microcapsules compared to the yoghurt with non-encapsulated polyphenols, which were lost before the intestinal phase was reached. This study demonstrated the suitability of PCL to protect polyphenolic compounds and their use in functional yoghurt formulations. This microencapsulation method was applied by Carniel et al. (2021) to preserve geraniol, a monoterpene attractive for its antibacterial and antifungal properties. By mini emulsion polymerisation, capsules of 148 nm on average were obtained with an encapsulation yield of 95%. The microcapsule formation was verified by transmission electron microscopy, and protection against volatilisation of geraniol was proved by thermogravimetry by measuring the release of encapsulated geraniol occurring at 100 °C higher temperatures above the temperature at which the natural compound volatilises.

Microencapsulation in agroindustry. The PCL is also attractive to agroindustry, which aims to develop renewable materials for sustainable agriculture using biodegradable materials (Birania et al. 2021). In the agroindustry, active materials that release compounds that contribute to optimal crop growth have been investigated recently to improve the quality of fruits and vegetables. It is well known that citric acid and naringin are involved in the interaction between plants and microorganisms. Citric acid has been reported as a chemoattractant agent for rhizobacteria and inorganic phosphorus solubilisers, while naringin as an inducer of several nod genes in the *Rhizobium* spp., necessary to carry out the initial event of the symbiosis and to fixate the atmospheric nitrogen effectively. (Cesari et al. 2020). The PCL encapsulated these compounds, ensuring the signal molecules were present during the plant development. This is through a gradual release of citric acid and naringin, leading to a more sustained delivery of signal molecules to the soil and facilitating the continuous supply (slow release) of nutrients to plants.

CONCLUSION

Despite the PCL applications being mainly focused on tissue engineering and regenerative medicine, other applications in the food industry and agroindustry have recently started to be explored. In the food industry, PCL is manufactured in porous or dense films to support bioactive compounds in the design of packages and prolong the shelf life of food products. Regarding encapsulating applications, the PCL protects bioactive compounds, allowing them to be added to food products and increasing their nutritional value. In agroindustry, the PCL microcapsules protect the substances of interest from degradation, guaranteeing their stability until they reach the plants or soils. Despite the progress, the PCL potential in the agricultural and food industries still must be explored.

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Received: November 27, 2023
Accepted: March 18, 2024
Published online: April 9, 2024