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A Review of Loading Methodologies of Active Aromatic Substance into Biopolymers

Dibyankar Barik[1], Rakesh Kumar[2,]*

Abstract

Biopolymer-based films are increasingly being studied as sustainable alternatives to conventional packaging materials due to their biodegradability, renewability, and potential for functionalization. There are several natural polymers in the category of biopolymers such as soy protein, zein, cellulose, chitosan, and several other biodegradable synthetic polymers such as polylactic acid and polycaprolactone. However, these often show limitations in barrier properties, mechanical strength, and resistance to microbial contamination. To overcome these drawbacks, a wide range of bioactive additives, including nanoparticles, nanomaterials, essential oils, and organic acids, have been incorporated into biopolymeric matrices to impart antioxidant and antibacterial activity. In addition to active aromatic substances, inorganic nanofillers, such as zinc and silver, are widely incorporated in the biopolymers to fabricate packaging films and membranes with significant antimicrobial properties. Even cellulose nanocrystals, an organic filler, are widely used in the packaging sector. The effectiveness of these active films strongly depends not only on the type of bioactive compound but also on the loading methodology, which governs dispersion, stability, retention, and release behavior of the active agents within the polymer network. This review highlights the loading methodologies adopted to incorporate the additives into the biopolymers and how they can significantly enhance the performance of biopolymer films and support their application in active and sustainable packaging systems.

Keywords: Biopolymers, food packaging, nanomaterials, antimicrobial properties, loading methodologies

INTRODUCTION

Till date, synthetic materials are predominantly used to prepare food packaging films due to their excellent barrier and resistance properties, but there is a shift towards the usage of biopolymers in the packaging sector. The reason for using biopolymers in the packaging sector is that they are biodegradable in nature, breaking down through fragmentation and conversion into carbon dioxide, water, and biomass under aerobic conditions and hydrocarbons, methane, and biomass under anaerobic conditions [1]. Several biopolymers, such as starch, cellulose, chitosan, and proteins, like whey and soy protein, are being widely explored as biopolymers in the packaging sector [2].

Inorganic substances, including nanosized Ag [3], ZnO [4], etc., essential oils, phenolic acids, and phenolic extracts, have been reported to be used in the fabrication of active packaging materials. It has been reported that the use of nanomaterials has an edge over bulk materials. The incorporation of nanomaterials into food systems has several benefits [5].

The need for certain additives, such as salt and preservatives, can be reduced.

***Author for Correspondence**
Rakesh Kumar
E-mail: rakeshkr@cusb.ac.in

[1]Research Scholar, Department of Biotechnology, Central University of South Bihar, NH-120, Gaya-Panchanpur Road, Post- Fatehpur, P.S.- Tekari, Gaya, Bihar, India
[2]Professor and Head, Department of Biotechnology, Central University of South Bihar, NH-120, Gaya-Panchanpur Road, Fatehpur, Tekari, Gaya, Bihar, India

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- On the property side, there will be reduced permeability to vapor and oxygen.
- The mechanical properties of the nanomaterial-loaded biopolymers are significantly enhanced.
- Additionally, nanomaterials can also impart antibacterial properties.

It has been stated that bio-nanocomposites are suitable as active and/or intelligent packaging materials due to their enhanced mechanical, thermal, barrier, antimicrobial, and antioxidant properties. These materials, when used in the packaging sector, focus on extending shelf-life and reducing microbial growth in food products [6]. To prepare bio-nanocomposites, it is essential to consider the methods for loading these nanomaterials into biopolymers. This review paper discusses the different types of loading methodologies.

LOADING METHODOLOGIES

There are several techniques for incorporating active substances into biofilms. However, the selection of biopolymers and bioactive substances is crucial.

Loading Methods

In the first technique, films can be prepared with biopolymeric material that possesses intrinsic bioactive compounds. In the second technique, bioactive compounds can be mixed with a biopolymer to fabricate the films. In the third method, one can encapsulate the bioactive compounds within the biopolymer to produce films. Incorporating bioactive substances into electrospun nanofibers, compared to conventional biopolymeric films, offers advantages for food packaging coatings. The electrospun fiber exhibits a high surface-to-volume ratio; hence, packaging material obtained from the incorporated electrospun fiber shows higher crack resistance and more flexibility in terms of mechanical properties. Encapsulation includes physical and chemical methods such as spray drying/lyophilization, and physicochemical methods such as coacervation.

Direct Blending

Films with inherent bioactive compounds can be produced from saffron flour extracted from the residue of saffron dye, babassu mesocarp, a byproduct of the babassu oil extraction industry [7, 8]. Both materials contained inherent phenolic compounds that showed antioxidant activity and were obtained from agricultural products and byproducts [9]. It is necessary to explore the potential of biopolymer-based materials obtained mainly from agricultural products and byproducts, derived from the processing of fruits and vegetables, which are rich sources of low-cost bioactive biomaterials.

Films incorporated with bioactive compounds directly into agro-based polymers are the most common type of incorporation of bioactive compounds in films. Prior to biopolymer film formation, bioactive compounds can be incorporated into the biopolymer during solubilization [10, 11]. Information regarding the solubilization characteristics and the maximum allowable concentration of bioactive material to be added to the biopolymer is required. Knowledge of optimum pH and temperature is also needed, alongside other important factors include solubility compatibility, maximum loading capacity, and molecular interactions between the bioactive compound and the polymer matrix. The advantage of this technique is that it allows a homogeneous distribution of the bioactive compound in the biopolymeric matrix with slow release to the surrounding environment [12]. Tea Polyphenol [13], Chinese chive root extract [14], essential oils [15], green coffee oil and *Acca sellowiana* waste byproduct [16], feijoa peel flour has been incorporated as bioactive compounds in the carboxymethyl cellulose [10] and pectin [17], whey protein isolate films as a biopolymer. The phenolic constituents of the extract increase the number of intermolecular crosslinks in the film-forming solution, thereby stabilizing their structure, reducing the effect of the plasticizer, and enhancing the film's thickness and strength [18].

Some bacteriocins, like nisin, natamycin, ε-polylysine, and enterocins, have been incorporated in antimicrobial biopolymeric films to produce active protein-based packages and effectively reduce *Listeria monocytogenes* growth [19, 20]. In addition to bacteriocin, chelating compounds, such as

EDTA, as well as acidulant agents, such as sodium lactate, potassium sorbate, citric, acetic, malic, lactic, tartaric, sorbic, and *p*-aminobenzoic acids, have been employed to improve antimicrobial properties of the protein films [21]. Acidulants are small molecules with several hydroxyl groups that facilitate protein–protein interactions by developing protein–plasticizer hydrogen bonds. Antimicrobial enzymes, such as the LactoPerOxidase System (LPOS) and lysozyme, have also incorporated into protein films with antimicrobial activity.

Metallic NP, such as silver nanoparticles (AgNP) and layered silicates, such as the montmorillonite (MMT) nano-clay mineral, have been incorporated in the protein matrix to impart antimicrobial effects and enhance barrier and mechanical properties so that the shelf life of the packed food can be improved significantly [22]. Soy, casein, zein, ovalbumen, and whey protein-based nanoparticles have been used as delivery systems for food bioactive ingredients because they are entirely biocompatible with food formulations. Protein nanoparticles of size 100–1000 nm can increase the residence time of bioactive compounds. It has been reported by Tang et al. that soy protein-based nanoparticles perform better as nanocarriers for bioactives over other food proteins mentioned earlier [23].

Protein-Based Films as Carriers of Antioxidant Agents

Antioxidants are also incorporated in packaging materials because they delay lipid oxidation. The oxidation of lipids reduces food shelf life and is responsible for off-flavor, off-odor, product discoloration, and loss of vitamins in the food [24]. Natural antioxidants that have been used are α-tocopherol [25], ascorbic acid, the combination of α-tocopherol and ferulic acid [26], and coumarin. Catechin–Kradon leaf and Murta-leaf extract has also been added to gelatin films to increase antioxidative capacity [27].

Electrohydrodynamic Processing

Electrohydrodynamic processing is a technology that is based on the principle of encapsulation of functional ingredients and has been explored for the development of active coatings for food packaging [28]. The food-grade dry nano- and microencapsulation structures in the form of fiber mats by the electrospinning technique or electrospraying of particulate powders can be achieved. The advantage of electrohydrodynamic processing is its simplicity because it can be carried out in a one-step process under mild temperature conditions. Electrospinning has been effectively used as an antimicrobial coating in the food packaging sector. The properties of the materials generated by the electrospinning technique are:

- High barrier structures.
- Adhesive interlayers.
- Packaging materials with temperature buffering capacity.
- Functionalized antioxidant coatings based on α-tocopherol.

The morphology of the materials obtained by the electrospinning technique depends on the properties of the solution with which electrospinning is done and the process parameters, such as temperature, can be varied, resulting in the production of continuous layers in the form of fiber mats [29].

Microencapsulation Through Complex Coacervation

Natural oxidants tend to be easily oxidized and respond sensitively to heat and light, and microencapsulation is a strategy to increase bioavailability and retention time in the food matrix. The process also masks their aroma and delays the onset of oxidation and hydrolysis but maintains their functionality. In macroencapsulation, the wall material is encapsulating material, and the core is the central material. The merger of two complementary charged biopolymers is called coacervation. It is primarily used for the encapsulation of antioxidants, β-carotene, turmeric, and lycopene. Four classes of interactions, hydrogen bonding, polarization, electrostatic interactions, and hydrophobic bridges, are involved in coacervation. The level of interactions can be computed by considering the type of polymer, its pH, and ionic strength. Additionally, the ratio of the biopolymers mixed and their concentration are important. A typical coacervation type encompasses only a single polymer in the presence of water, whereas complex type coacervation involves two or more than two polymers, resulting in the fabrication of microcapsules with dimensions ranging from approximately 1–1000 μm [30]. The first step in a complex type of coacervation is emulsification, which involves encapsulating vitamins, probiotics, enzymes, and minerals. Virgin oil of olives in the presence of maltodextrin, carboxymethyl cellulose, and lecithin, as well as Garcinia fruit in whey protein isolates and maltodextrin, have been microencapsulated by the process of lyophilization [31]. Microencapsulation efficiency can also be determined by using the formula given below [31]. Here, absorbance was determined at 510 nm.

Encapsulation efficiency (%) = 1 – Absorbance / 1 × 100

CONCLUSIONS

Different techniques to incorporate active aromatic substances or nanomaterials into biopolymers have emerged as a transformative technology over the past decade, offering promising advancements in the food packaging sector. Soy, casein, zein, ovalbumin, and whey protein biopolymers have been discussed with the possibility of incorporating several bioactive compounds and nanoparticles. The incorporation of active compounds offers superior barrier properties and enhances the film's thickness and strength. The selection of a suitable loading methodology plays a decisive role in determining the final performance of active films, as it influences the distribution of the additive, the stability of volatile or sensitive compounds, and the rate at which bioactive agents migrate or release from the polymer matrix. Direct blending is a simple and widely used method, but it often leads to poor dispersion and aggregation of nanomaterials, as well as the rapid loss of volatile compounds. In contrast, carrier-assisted methods like protein-based films, electrohydrodynamic processing, and microencapsulation improve protection and control overactive compounds, enhancing functionality and longevity. Future development should focus on optimizing the compatibility of biopolymer matrices with active agents, enhancing long-term stability in real packaging conditions, and scaling up effective techniques without sacrificing safety and performance. Overall, advanced loading technologies offer a practical way to produce multifunctional biopolymer films with improved bioactivity, meeting the demand for eco-friendly active packaging solutions.

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