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Chapter 9
Starch Nanomaterials as Functional Packaging Ingredients

Pedro Augusto Invernizzi Sponchiado, Samile Bezerra de Aguiar, Carla Arias Ivonne La Fuente, and Bianca Chieregato Maniglia

9.1 Introduction

In recent times, the field of nanotechnology has provided valuable information on the synthesis of materials, leading to the development of new substances with desired properties. Using nanoscience techniques, traditional functional properties have been improved, in addition to revealing new features and functionality. This breakthrough paved the way for manufacturing nanomaterials from natural and sustainable sources, thus expanding the potential applications of nanotechnology in the packaging sector.

Nanotechnology involves the production and characterization of materials with at least one dimension ranging from 1 to 100 nm in length [1]. In the realm of packaging and other sectors, nanomaterials integrated into the matrix phase must possess not only these nanoscale dimensions but also exhibit distinct physical and chemical properties that differ significantly from their macroscale counterparts [2]. This distinction has significant implications and offers various applications. Typically, organic additives offer several advantages, such as a positive environmental impact due to their low energy consumption during manufacturing, as well as

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ease of combustion and recycling [3]. In addition, their relatively simple processability and non-abrasive nature contribute to notable cost savings [3].

In this context, starch nanomaterials have emerged as promising functional packaging ingredients in recent years, offering a range of benefits for the development of sustainable and efficient packaging solutions. Derived from renewable resources, such as corn, potatoes, and cassava, starch nanomaterials (SNMts) possess unique properties at the nanoscale, making them highly desirable for enhancing packaging performance [4]. With advancements in nanotechnology, these materials have demonstrated remarkable potential in improving the barrier properties, mechanical strength, and biodegradability of packaging materials. In addition, SNMts have shown promise in addressing food safety concerns through their antimicrobial and oxygen-scavenging capabilities.

There are two main types of nanostarches: starch nanocrystals (SNCs) and starch nanoparticles (SNPs). SNCs are crystalline portions formed by the breakdown of the amorphous regions within starch granules [5]. In contrast, SNPs are derived from gelatinized starch and may contain both amorphous and crystalline regions [5]. These SNMts can be obtained using methods such as acid and enzymatic hydrolysis, antisolvent precipitation, high-pressure homogenization, ultrasound, electrospinning, self-assembly, and emulsification [6, 7].

This chapter discusses the use of SNMts as functional packaging ingredients, highlighting their role in enabling eco-friendly and innovative packaging solutions that extend the shelf life of products while reducing environmental impact. In addition, this chapter explores how starch modification from the microscale to the nanoscale helps in the mechanical and biological properties (antimicrobial and release of bioactive compounds) of packaging.

9.2 Fundamentals of Starch

Starch is a granular homopolysaccharide mainly composed of two natural polymers known as amylose and amylopectin, as represented in Fig. 9.1. These polymers are both composed of D-glucose residues linked together through $\alpha(1 \rightarrow 4)$ glycosidic bonds, differing from each other in molecular size and structural disposition: while amylose has a size distribution of approximately 10^5–10^6 Da and generally exhibits a linear chain organized in a helical structure, amylopectin is approximately 4–5 times larger than amylose (10^7–10^8 Da) and has a branched chain with a branching level of 4–5% with $\alpha(1 \rightarrow 6)$ linkages [8]. This homopolysaccharide is normally composed of 70–80% amylopectin, which comprises the semicrystalline regions of starch granules, and approximately 20–30% of amylose in amorphous zones. However, the proportion of amylose can range from less than 1% in some waxy starches to more than 70% in the high-amylose type [9], with the amylose content varying according to the botanical origin [10].

In terms of its biological functions, there are two types of this polysaccharide: transitory and storage starch [11]. Transitory starch, also known as temporary or

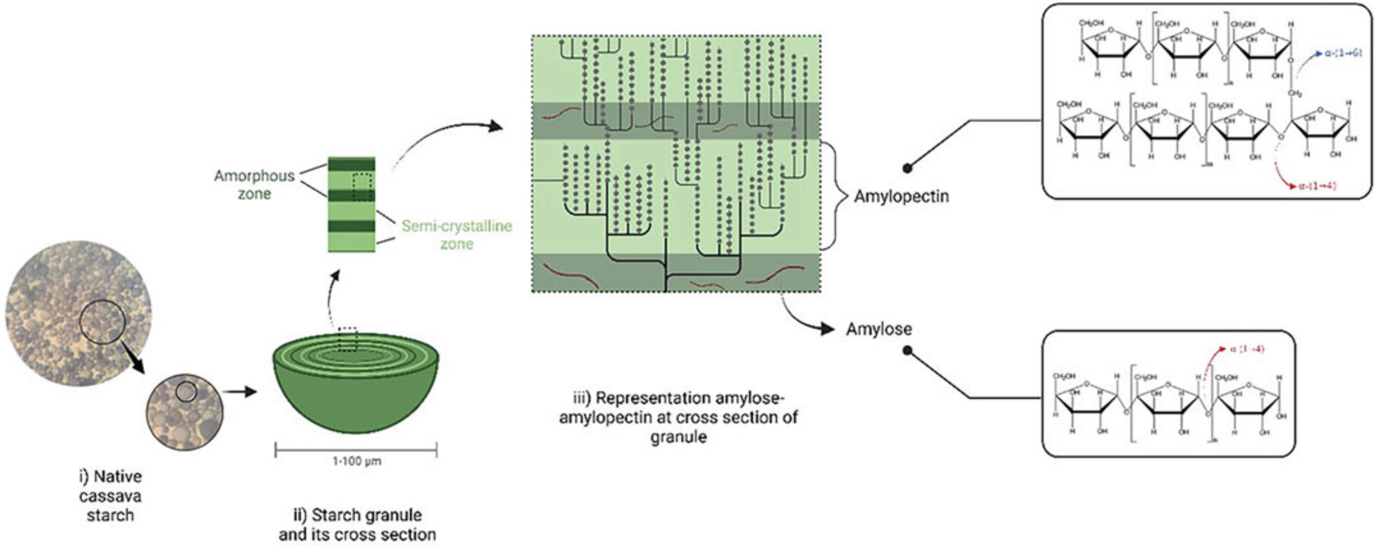

Fig. 9.1 Schematic representation of (i) Cassava starch granules; (ii) Cross section of starch granules with the alternate distribution of the crystalline and amorphous lamellae; (iii) Representation of the inner structure of the starch granules with the distribution of amylose and amylopectin

transient starch, is synthesized and accumulated during the day in photosynthetic tissues such as leaves, to be subsequently used during the night for respiration. It has a relatively simple structure with smaller granules [12, 13]. In plants, the transient starch serves two main functions: (a) as an alternative carbon source for the photosynthesis (faster than by sucrose synthesis); and (b) as a carbon supply at the night. On the other hand, storage starch is synthesized and found in non-photosynthetic tissues such as roots, tubers, and seeds, and is stored for long periods and has a complex structure with different proportions of amylose and amylopectin [14]. Owing to its characteristics, abundance, and multiple functionalities, this type of starch has a significant commercial value and finds application in various industrial sectors [14]. These applications range from being an additive in the food industry [15, 16] to serving as a raw material for biodegradable plastics production [4, 16].

As a storage carbohydrate in plants, starch is quantitatively one of the most abundant materials in nature and can be obtained from many resources such as cassava, potato, sweet potato, corn, and cereal grains [17]. The different sources of starch can directly influence their functional properties, including gelatinization temperature, paste viscosity, and retrogradation speed. This is because starches obtained from different botanical sources may undergo some structural variations, such as differences in the granule size, composition, and molecular architecture of amylose and amylopectin [18]. Once amylopectin comprises the crystalline region, a higher proportion of this polymer leads to starches with greater crystallinity [19]. Moreover, the gelatinization properties (represented by transition temperatures) depend on the molecular structure of amylopectin and the amylose-amylopectin ratio. Once that gelatinization initiates in the amorphous regions, it slowly spreads to the periphery, causing strain and tearing the crystalline zone. High transition temperatures correspond to a high degree of crystallinity and stability of the granule to gelatinization [20]. Naturally, starch granules can exhibit different shapes and sizes, ranging from spherical or polygonal to elongated, and they can have sizes ranging from submicron to approximately 100 μm, depending on their source [21]. For example, cassava starch has an amylose content (AC) of approximately 15% and flake-shaped, irregular granules with a mean size of 20 μm. Potato starch, on the other hand, has a mean AC of 21% and irregular, oval-shaped granules with a mean size of 35 μm. Corn starch contains 28% of amylose and angular granules with 16 μm [22–24].

9.3 Starch-Based Nanomaterials and Their Production

In addition to the traditional modifications applied to this polysaccharide at the microscale, such as physical [25–27] and chemical [28–30] methodologies, starch at the nanoscale has emerged as a new type of changed starch with the advantages associated with size reduction and increased surface area, along with the quantum effects relative to the nanoscale [31]. In the food industry, nanomaterials can act as a reinforcement for packaging materials, enhancing their mechanical and thermal properties. In this context, starch nanomaterials can be used to improve the thermal and mechanical resistance of food packaging, additionally with the potential to incorporate biocompatibility, biodegradability, and non-toxicity into materials [32–34].

SNMts can be categorized as follows:

(a) **nanocrystals (SNCs)**, which are crystalline sheets produced from the disruption and disorganization of the semicrystalline structure of starch granules [33, 34];
(b) **nanoparticles (SNPs)**, defined as nanosized amorphous solid particles with less than 10% of crystallinity degree [35, 36];
(c) **nanovesicles (SNVs)**, or nanoliposomes, are lipid vesicles coated with starch nanomaterials [37];
(d) **nanofibers (SNFs)**, which are cylindrical nanosized structures [38];
(e) **nanomicelles (SNMs)**, which are defined as colloidal dispersions consisting of a hydrophobic core and a hydrophilic shell based on starch nanomaterials [5].

However, some authors distingue the SNMts into only two major groups in terms of crystallinity, namely nanocrystals (SNCs), and nanoparticles (SNPs). This categorization is based on the distinct structural characteristics and degree of crystallinity exhibited by these two nanomaterials: while SNCs possess a crystalline platelet-like structure with defined and ordered crystalline regions, the SNPs are amorphous solid particles with less structural definition and unorganized regions [4, 34].

In general, starch nanomaterials can be obtained through two main approaches: “top-down” and “bottom-up”. In the first approach, the starch-based nanomaterials are produced by breaking down starch granules using methods such as acid and enzymatic hydrolysis (AH and EH, respectively), and physical treatments [5]. Although these methods are usually simple and inexpensive, they are less effective for starch granules with irregular shapes and tiny sizes [39]. In contrast, in the “bottom-up” approach, the starch nanomaterials are generated through physical and/or chemical interactions and the accumulation of starch chain precursors (monomers and oligomers), such as nanoprecipitation, electrospinning, and self-assembly [40]. These methods are capable of producing nanostructured starch with a controllable shape and size [39]. The production of starch nanomaterials using some of the methods mentioned above is presented in Table 9.1.

As a “top-down” approach, acid hydrolysis (AH) is a typical methodology for the production of SNCs and is based on the use of acids such as sulfuric acid, hydrochloric acid, and citric acid, which penetrate the amorphous regions of starch granules to break the glycosidic bonds of the polymers while leaving the crystalline zones undamaged [4]. In general, the AH occurs in three steps: (i) surface granule erosion; (ii) radial diffusion of the acid, which causes a collapse of the granule wall; and (iii) fragmentation of the regions [31]. To ensure an effective procedure, it is important to consider factors such as temperature (below the gelatinization point of starch), acid and starch concentrations, time of reaction, and rotation [46]. Velásquez-Castillo et al. [47] showed that varying the temperature of acid hydrolysis of quinoa starch (30, 35, and 40 °C) affected the physical properties of the

Table 9.1 Starch nanomaterial production methods and their applications

Starch source	Material type	Method of production	Major findings	Reference
Cassava starch (17% amylose)	Cassava starch-based nanocomposite films with cassava SNCs	Cassava SNCs prepared by AH	Enhanced mechanical and barrier properties, as well as film solubility; reduced WVP, while increased traction resistance and elastic modulus of films	[33]
Waxy maize starch (99% amylopectin)	Nanocomposite films made of normal corn starch and debranched SNPs	Debranched SNPs prepared by ultrasonication	Addition of debranched SNPs in films enhance its mechanical properties; reduce the water vapor penetrability of the films	[36]
Normal corn starch (26.5% amylose)				
HI-MAIZE® 260 starch	Starch-based films with SNFs	SNFs prepared by electrospinning methodology with posterior cross-linking	The cross-linking improved the tensile strength and the water contact angle of nanocomposite films (with SNFs obtained by electrospinning)	[38]
Cassava starch (28% amylose)	Cassava starch-based nanocomposite films filled with waxy maize SNPs or cassava SNPs	Waxy SNPs obtained by AH; waxy SNPs and cassava SNPs produced by gamma irradiation	Waxy SNPs and cassava SNPs formed by gamma irradiation exhibited the same thermal degradation behavior; the addition of cassava SNPs as cassava matrix reinforcement did not significantly improve its mechanical properties, while the use of waxy SNPs let to an enhancement of 102% in the storage modulus of nanocomposite films	[41]
Waxy maize starch (99% amylopectin)				
Potato starch (31% amylose)	Nanocomposite films made of pea starch and potato starch nanoparticles	SNPs fabricated through recrystallization (self-assembly of short amylose)	Enhanced physicochemical properties of pea starch-based nanocomposite film with the addition of SNPs; increased tensile	[42]

Pea starch (40% amylose)			strength and thermal properties; the addition of SNPs decreases the WVP of films	
Cassava starch (30% amylose)	Starch-based nanocomposite films with SNPs and cellulose nanoparticles	SNPs obtained by ultrasound (SNPs-U) and AH (SNPs-AH)	The addition of SNPs and cellulose nanoparticles influenced the morphology, WVP, and mechanical properties of the nanocomposite films; SNPs-AH had higher crystallinity than SNPs-U	[43]
Quinoa starch (22.8% amylose)	Nanocrystals made of quinoa starch and waxy maize starch	SNCs obtained by AH	Influence of AH temperature over morphological characteristics, crystallinity, and yield of SNCs; better properties were obtained for SNCs at 35 and 40 °C	[44]
Waxy maize starch (99% amylopectin)				
Maize corn starch (29% amylose)	Polyurethane-based nanocomposite films, filled with maize corn SNPs	SNPs synthesized by ultrasound-assisted AH method	Nanocomposite films showed 99% resistance to bacterial transformation; a reduction of 60% WVP in films with addition of 20% SNPs; mechanical and thermal properties of the polyurethane-based films were improved with addition of SNPs	[45]

AH acid hydrolysis, *SNCs* starch nanocrystals, *SNFs* starch nanofibers, *SNPs* starch nanoparticles, *WVP* water vapor permeability nanocrystals produced. The SNCs developed at 30 °C exhibited micrometric dimensions and an irregular shape, unlike those developed at 35 and 40 °C, which presented better morphologies and properties suitable for use as reinforcement materials. In addition, acid hydrolysis also has been combined with other methods. Amini and Razavi [48] used simultaneous ultrasound and sulfuric acid hydrolysis to prepare corn SNCs. By adjusting the temperature, acid/starch concentration, and duration of the process, the researchers obtained the best nanocrystals within 45 min of reaction (using both methods, ultrasound, and hydrolysis), yielding up to a 22% and resulting in particle sizes smaller than 100 nm.

In addition to chemical hydrolysis, the use of enzymes such as α-amylase, β-amylase, and pullulanase to break and de-branch starch chains is also an interesting pathway. Similar to acid hydrolysis, the enzymatic method (EH) proceeds in two stages: initially attacking the starch chains in the amorphous regions, rapidly, followed by a slower action on the crystalline lamella [39]. Owing to this similarity in mechanisms, EH, and chemical hydrolysis, also tends to result in the development of starch nanomaterials with high crystallinity. However, according to Marta et al. [39], the use of enzymes to produce SNMts has the advantage of being carried out in a shorter production time compared with AH, along with generally yielding a higher amount of SNMts. Dukare et al. [49] developed nanostarches from three different sources (maize, potato, and cassava) using the EH technique with α-amylase, an enzyme that catalyzes the cleavage of α-1,4-glycosid bonds in starch. The researchers achieved an improved yield compared with the nanomaterials obtained by AH (18, 29, and 41% compared to 16, 25, 35%, respectively). However, the size of the nanostarches produced by EH was significantly larger than that obtained by AH, and this difference may be attributed to the larger molecular size of α-amylase compared to the acid molecules, which could difficult its penetration and the attack on starch granule regions. Moreover, a combination of acid and enzymatic technique can also be employed to improve the properties of starch nanomaterials developed. Hao et al. [50] prepared SNCs from waxy potato starch by glucoamylase hydrolysis followed by acid hydrolysis. The results demonstrated enhanced stability of SNCs with the combination of these two methods, along with improved crystallinity and reduced time required for acid hydrolysis. This improvement was attributed to the prior enzymatic attack, which possibly created pores facilitating acid penetration into the granules.

In addition to the mentioned “top-down” approaches, various physical methods can also be used to produce SNPs, including gamma irradiation (GI), high-pressure homogenization (HPH), and ultrasonication. These physical methods offer several advantages over other “top-down” techniques, as they are less expensive, time-efficient, and require a reduced use of chemical reagents [39]. In this context, gamma irradiation is widely employed since is a fast and convenient method that is independent of catalysts. Gamma radiation is a form of electromagnetic radiation with high energy and short wavelength, and it is used in GI methodology to generate free radicals that can cleave the glycosidic linkages of starch polymers, mostly in the amorphous region, leading to the formation of smaller fragments [41, 51]. Despite being a radical-based mechanism, it is important to emphasize that the free radicals involved in this process are easily recombined in water, resulting in final solutions with free radicals [52]. Lamanna et al. [41] employed gamma radiation to prepare SNPs from cassava and waxy maize starches, using a dose of 20 kGy. The authors obtained nanoparticles with an average size of approximately 20 and 30 nm. Additionally, they observed that incorporating waxy maize starches-gamma radiated as a reinforcement in the cassava matrix led to the formation of starch nanocomposites with improved mechanical properties (an increase of 102% in storage modulus).

As mentioned previously, two other advantageous physical methods used to produce SNPs are high-pressure homogenization (HPH) and ultrasonication. HPH is considered a "green" technique that enables the production of smaller particles with better homogeneity. It involves the flow of a fluid such as a starch suspension, through a small orifice (throttle valve) under high pressure [53]. The reduction in particle size to the nanoscale can be explained as a result of the disruption of linkages inside the granules, especially the hydrogen bonds, induced by the mechanical shear forces generated during the flow [51]. Ahmad et al. [54] developed SNPs from sago starch using the environmentally friendly HPH procedure, at a pressure of 250 MPa. Ultrasonication was used at the end to ensure the proper dispersion of the produced nanoparticles. The obtained SNPs exhibited a highly narrow size distribution, and when incorporated as reinforcement material in starch films, they effectively reduced the water vapor permeability and improved the transparency of the films.

In addition to HPH, ultrasonication is a rapid and environmentally friendly physical method for producing SNPs through the application of high-frequency waves. The process involves the formation of gas bubbles in the medium, at a process called as cavitation, which subsequently collapse, generating a high local pressure and shockwaves that will break down the starch granules into smaller nanoparticles [39]. To maintain the integrity of the modification during the process, it is important to control certain parameters such as starch dispersion, frequency of ultrasound, temperature, and total time [51]. SNPs from waxy maize and standard maize starches were produced using ultrasonication at low temperatures (8–10 °C). After 75 min of ultrasonication, the SNPs were obtained with an average size between 100 and 200 nm and exhibited a decrease in crystallinity [48]. Alternatively, this method can also be used combined with another "top-down" method, such as Bajer [55] who developed SNPs from corn and waxy corn starches using both AH and ultrasonication. Applying only one of these methods it was obtained nanoparticles with an average size of around 3–4 nm. However, by employing both methods, even smaller particles were formed, with average sizes of up to 3 nm, without the formation of aggregates.

A second approach to producing SNPs is known as the "bottom-up" approach and is based on the principle of building the nanoparticles from smaller components and gradually assembling them into the desired nanomaterials. Therefore, they are methods that have the advantage of developing particles with precise control over their size and composition [7]. Among the various "bottom-up" approaches, in recent years, the most used methods explored as alternatives for producing SNPs are nanoprecipitation, electrospinning, and self-assembly [5]. In this context, nanoprecipitation is a simple and scalable technique that involves the rapid addition of a non-solvent, such as ethanol, propanol, or acetone, into a starch solution under a vigorous agitation. Rapid addition leads to the formation of a supersaturated starch solution, resulting in nucleation and growth of particles, culminating in the precipitation of nanoscale particles [4, 56]. Factors such as polymer concentration, mixing speed, and temperature can affect the size and morphology of the SNPs. Furthermore, Wu et al. [57] reported that the proportion of solvent/non-solvent used in the nanoprecipitation process can affect the size of the nanoparticles, as an increase in the amount of non-solvent usually leads to a decrease in particle size. In addition, the source of starch also can influence the formation of SNPs, as shown by Qin et al. [58]. In their work, SNPs were prepared from seven native starches, including waxy corn, normal corn, high-amylose corn, potato, tapioca, sweet potato, and pea, by precipitating with ethanol. This process leads to the formation of nanoparticles with a mean particle size ranging from 30 to 75 nm, and a positive correlation was observed between the size of the native starch granule and the resulting SNP. In other words, the smaller the starch granules in the source material, the smaller the resulting nanoparticles were produced.

Recently, the electrospinning technique, also known as electrospraying, has been widely used to produce starch nanomaterials, particularly starch nanofibers with diameters of up to 100 nm, depending on the parameters used [5]. With the advantage of developing SNFs with precise control over fiber diameter and morphology, the nanofibers produced by this method show several advantageous properties, such as high surface-area-to-volume ratio, high porosity with small pore size, and superior mechanical properties depending on the source material [59]. In this “bottom-up” method, a polymer solution is extruded through a small opening called a spinneret, while a high voltage (usually between 100 and 500 kV/m) is applied between this solution and a collector. As the voltage increases, the droplet of the solution undergoes deformation, changing from a pointed shape to a conical shape, and is then ejected from the syringe pump toward the collector, where it is deposited as randomly oriented nanofibers [60, 61]. Among the various applications of starch nanofibers, including biomedical delivery systems and materials for tissue engineering [61, 62], SNFs have also been developed for the production of films in the food packaging industry. For example, Cai et al. [62] synthesized SNF-based films for food packaging, that were coated with a hydrophobic surface made of stearic acid. SNFs were obtained using 25% of starch with a mean diameter of 365 ± 97 nm and uniform distribution, exhibiting a smooth surface and random orientation. Moreover, the films prepared from these SNFs exhibited excellent flexibility, making them suitable for use in food packaging.

In addition to the mentioned “bottom-up” approaches, self-assembly is a versatile method that allows obtaining different types of SNMts, such as nanoparticles and nanovesicles [5]. In general, self-assembly is a process in which molecules, such as starch, organize themselves into stable nanostructures spontaneously, driven by intermolecular interactions such as hydrophobic and Van der Waals interactions, hydrogen bonding, electrostatic forces, and π- π aromatic stacking [63]. In order to enhance the properties of starch chains and facilitate self-assembly, in some cases, a pretreatment of starch is applied. This can involve the use of combined methods, such as chemical or enzymatic hydrolysis. From this perspective, a starch-based film of pea starch composed of SNPs derived from potato starch was prepared by Jiang et al. [42]. The process involved the self-assembly of short amylose combined with prior enzymatic treatment of the potato starch. The SNPs obtained through the self-assembly displayed a globular shape with particle sizes ranging from 15 to 30 nm under optimized conditions. In addition, the incorporation of SNPs into the pea-starch films decreased water vapor permeability and an improvement in the mechanical properties of the materials.

9.4 Applications in Food Packaging and Coating

9.4.1 Thermal Stability Agents

To develop improved food packaging, several film parameters can influence the final quality of the packaging, such as optical, mechanical, barrier, and thermal properties [64]. In this case, the thermal characteristics of materials are crucial for their use as commercial products, considering diverse aspects ranging from the transportation process to their storage and final usage of these manufactured items.

In general, during film characterization, the thermal stability can be determined by a simple method known as thermogravimetric analysis (TGA), which is based on the measurement of the mass variation of a sample upon heating over time. In other words, TGA allows for obtaining information about the thermal degradation behavior of materials [65]. In addition to TGA, differential scanning calorimetry (DSC) can also be used to investigate how the materials respond to heating, providing information about the melting point (T_m) of the films and their enthalpy change (ΔH) [45].

As an alternative to improving the thermal properties of films to be used in food packaging, different types of nanomaterials can be used in their matrices. According to Basavegowda and Baek [64], the presence of nanomaterials in polymeric films, for example, can act as an obstacle to heat and mass transfer, thus reducing the molecular mobility of polymers in the matrix and preventing heat-induced polymer degradation. In addition to the applications of starch nanomaterials in the food packaging systems, such as reinforcement materials and antimicrobial agents, these nanoscale starches can also be used to optimize the thermal properties of nanocomposite films. Hakke et al. [45] produced SNPs from maize corn starch and used them as nanofillers to improve several of properties of polyurethane (PU) films. TGA and DSC analysis of the SNP-PU nanocomposite films revealed an increase in ΔH values (ranging from 3.34 to 17.33 J/g for the pure PU film and the 30% SNP-PU material, respectively), and a decrease in thermal degradation as the SNP concentration increased (for the first state of weight loss of 10%: in a temperature below of 100 °C for PU film, compared to 200–300 °C for the SNP-PU films). Moreover, the difference between the melting (T_m) and glass transition (T_g)

temperatures of the nanocomposite film with the incorporation of 30 wt% of SNPs was higher than that of the PU film ($T_m = 64.7$ °C and 93.3 °C, $T_g = 33.6$ °C and 51.2 °C, respectively for pure PU and PU with 30 wt% of SNPs). All these observations were attributed to the presence of SNPs in the PU films. According to the authors, the overall increase in the operating range of temperature with the addition of SNPs will help to improve the thermal resistance of the nanocomposite films in coating applications.

In the research of Jiang et al. [42], the gradual addition of potato SNPs synthesized by enzymatic hydrolysis to the pea starch-based films was also correlated with an improvement in the thermal properties of the nanocomposite film. When compared to the control material, the melting temperature of all films with SNPs was increased (ranging from T_m of 226.91 to 235.81 °C for materials with 3 to 12 wt% content of SNPs; and T_m of 225.81 °C for the control). According to the authors, this observed improvement in the thermostability of starch films can be attributed to the presence of strong interactions between nanoparticles and the film matrix.

Nevertheless, it is important to consider that not only the presence of starch nanomaterials that can improve the thermal properties of the materials, but also the amount of starch nanomaterials used as nanofillers can influence them. As reported in the study of Li et al. [66], a gradual improvement in the thermal properties of a film with an increased amount of starch nanomaterials is not always observed. In this study, pea starch-based films (PS) with glycerol were produced, and SNCs developed by acid hydrolysis of waxy maize starch were used as nanofillers. Results obtained from TGA and DSC measurements revealed that the nanocomposite films with 1, 3, and 5 wt% of SNCs showed higher values of ΔH (ranging from 24.34 to 26.76 J/g for 1 and 5 wt% SNCs in the films, respectively; and 23.51 J/g for the native PS material) and higher thermal decomposition temperatures compared to the pure PS film. However, with the addition of 7 and 9 wt% of SNCs, the SNC-PS films showed poorer thermal performance and lower ΔH values (around 22 J/g for both materials), which was attributed to the catalyzed depolymerization of the materials due to the presence of a large number of sulfate groups on the surface of the SNCs. In this case, it was evident that the addition of SNCs could improve the thermal stability of the pea starch-based films when suitable amounts of SNCs were used, i.e., less than 5 wt%.

The main advantages of using SNPs as thermal stability agents are their biocompatibility, biodegradability, and low toxicity compared with synthetic alternatives. However, it is essential to note that the effectiveness of SNPs as thermal stability agents depends on the specific application and the processing techniques used to incorporate them into the desired materials or products. Research in this field is ongoing, and further advancements may lead to even more diverse and efficient applications of SNPs in thermal stability enhancement, such as heat-resistant coatings, thermal stabilizers in food applications, and flame-retardant materials.

9.4.2 Reinforcing Material and Mechanical Properties

SNMts have been described as an excellent option as one reinforcing material; when well dispersed on the matrix, they increase the interfacial area between matrix/fillers, and thus influences the material properties [67]. Mainly, SNPs and SNCs have attracted the attention of food packaging researchers [5].

The selection of the matrix depends on some parameters, such as the compatibility between components, the operation process, the cost, and the final application [68]. Common functional groups of nanomaterials could form links with the surface of the matrix material, holding the percolating network together [69]. In fact, nanoparticles are likely to bond with hydroxyl groups, strengthening the molecular forces between nanoparticles and starch [70].

From an industrial perspective, certain industries have already begun manufacturing nanoparticles. For instance, *Novamont* (Novara—Italy), collaborated with Goodyear Tire and Rubber to develop materials using nanoparticles derived from cornstarch. These nanoparticles have been used to partially substitute conventional carbon black and silica particles in their products [71]. This development showcases the growing trend of incorporating sustainable and renewable resources in industrial applications, leading to more eco-friendly and innovative materials.

The literature has reported that these materials improve the mechanical, thermal, and WVP properties, as a result of the formation of a filler-matrix interface [5]. The hierarchical organization of starch with its semicrystalline structure has been a key factor in the preparation of nanocrystals through different processes, i.e., controlled acid hydrolysis, as in the case of potato and waxy maize starch granules [71].

Several studies conducted have compared SNCs and SNPs. In one particular study [72], researchers examined the morphological, structural, and thermal characteristics of SNCs obtained via acid hydrolysis and compared them with SNPs derived from the same waxy maize starch, using ultrasound treatment. The findings revealed distinct differences between the two materials. The SNPs were observed to be completely amorphous, slightly smaller, and lacked any surface charge. On the other hand, the SNCs exhibited the expected platelet-like morphology and possessed a negative surface charge. Despite these variations in morphology and structure, both materials demonstrated excellent potential as reinforcing agents.

Table 9.2 summarizes some of the recent research on SNPs and SNCs from different sources and their effect on the mechanical properties of films produced from different matrices.

Although most of the studies reported increased mechanical performance due to the addition of these nanomaterials, the effect of SNPs on the mechanical properties has a strong concentration dependance. Indeed, Santana et al. [43] pointed out that low concentrations of SNPs (1 wt%) are probably unable to significantly increase the tensile strength. Moreover, Limin Dai et al. [81] reported that the aggregation of SNCs at higher concentrations, could weaken the interface adhesion between starch and nanocrystals. Therefore, and optimum concentrations must be determined to achieve the desired results.

Table 9.2 Representative studies of starch nanoparticles and starch nanocrystals reinforcing polymeric matrices and its effect on mechanical properties

Polymer matrix	Nanomaterial	Nanocomposite production	Major findings	Reference
Cassava starch	Quinoa SNCs obtained by acid hydrolysis at concentrations of (0, 2.5, 5.0, 7.5 and 10%w/w)	Casting	5 wt% of quinoa SNCs was the best concentration regarding the tensile strength and Young's modulus. These properties were lower for the quinoa SNCs concentrations above or below 5 wt%	[44]
Sago powder	Sago SNPs from sago (*Metroxylon Sagu*) starch at concentration of 0–8% (w/w)	Casting	Sago SNPs enhanced the tensile strength of films up to only 4% (w/w) loading of nanoparticles	[70]
Corn starch	Taro SNPs content of 0.5–15 wt%	Casting	Taro SNPs enhanced the tensile strength of films from 1.11 to 2.87 MPa	[73]
Maize starch	Corn SNPs content of 10–15 wt%	Casting	With 10% of corn SNPs, the tensile strength of films increased about 45% and the Young's modulus about 72%.	[74]
Green banana starch	Acetylated banana SNPs at concentrations of 0.01%, 0.05%, and 0.1% (w/w)	Casting	Nanoparticle concentration increased the tensile strength and reached the highest values at higher concentration of nanoparticles.	[75]
Native water chestnut starch	Water chestnut SNPs prepared by acid hydrolysis in concentrations of 0.5, 1, 2, 5 and 10 wt%	Casting	In nano starch composite films, more force was required to burst the film samples	[76]
PPC	Core–shell SNPs concentrations between 10 and 40 wt%	Melt-blending	With 20 wt% of SNPs, the tensile strength of the composite reached 21.5 MPa, which was 3.2 times that of neat PPC	[77]
PLA	Corn SNPs synthesized via gelatinization of the esterified starch in concentration 10–30 wt%	Melt-blending	PLA nanocomposites reinforced with corn SNPs resulted in a favorable balance between high tensile strength and elongation at break	[78]

(continued)

Table 9.2 (continued)

Polymer matrix	Nanomaterial	Nanocomposite production	Major findings	Reference
Mango seeds starch	SNCs from 0 to 10 wt%	Casting	The optimized conditions (8.5 wt% of SNCs) resulted in a film with enhanced strength, modulus, although the elongation has been impaired	[79]
Commercial cross-linked cassava starch	SNCs concentrations of 2–8 wt%	Casting	Film with 6% SNCs showed the best mechanical performance	[80]
Waxy maize starch	SNCs prepared with heat-moisture treatment and acid hydrolysis at concentrations of 0–12 wt%	Casting	When the SNCs content reached 10%, the nanocomposite film had the best overall performance	[81]
Corn starch: chitosan biopolymers mixes	Corn SNCs in concentrations of 2, 4 and 6 wt%	Casting	Tensile strength was directly related to the corn SNCs ratio	[82]

PLA poly (lactic acid), *PPC* poly(propylene carbonate), *SNCs* starch nanocrystals, *SNPs* starch nanoparticles

Nanomaterials have also been employed to improve the properties of coating materials [83], resulting in an interesting tool for addressing changes in packaging materials and improving the quality, shelf life, safety, and security of food products [84]. Indeed, Bizymis and Tzia [85] reported that starch nanomaterials can increase the barrier and the mechanical properties of edible coatings**,** in which natural ingredients with antimicrobial and antioxidant activity can be incorporated to increase their beneficial effects on the fresh produce quality [86]. Wang et al. [87] cited that SNPs added to polydimethylsiloxane were used to fabricate coatings; their results indicate the promising application of SNPs in super wettability systems and show the potential of the coating. In another study, cross-linked starch–BTCAD–NHS (starch–butane tetracarboxylic acid dianhydride–N-hydroxysuccinimide) was used to modify gelatin film to be used as a coating; the nanoparticles increased the tensile strength and the elongation at break (50–300%, respectively) [88].

Limin Dai, Zhang, and Cheng [80] produced a starch-based nanocomposite film/ coating made from cross-linked cassava starch reinforced by SNCs from 0 to 8 wt% of starch. The coatings based on 6 wt% showed the best mechanical and best coating to preserve pears, and maintain the color, texture, cell membrane permeability, total phenolic, soluble solids, and titratable acid contents of the fruit.

In general, SNPs, when used as reinforcing materials, can significantly affect the mechanical properties of the materials in which they are incorporated. It is also essential to optimize the concentration and dispersion of SNPs within the material to achieve the desired mechanical enhancements effectively. In addition, factors such as particle size, shape, and surface modification can also influence the overall mechanical performance of the composite material. SNPs show promising potential as sustainable and biodegradable reinforcements for various applications, including bioplastics, coatings, and composites, where improved mechanical properties are sought.

9.4.3 Biodegradability

It is relevant to mention new approaches that have been explored, such as the addition of nanosized materials as a tool to improve the biodegradation rate, in which the structure and composition of the biomaterials significantly influence this rate in different stages. The common claim is that these nanomaterials are biodegradable and make materials more biodegradable, which makes them even more attractive. However, data to confirm this assumption are missing [84]. Synthetic nanoparticles have already been discovered in the aquatic environment. Therefore, knowledge of their biodegradability is of maximum importance for risk estimation [84].

Few studies in the literature have reported the effect on biodegradation behavior of different fillers in different matrices. For example, in the PLA matrix was observed that fillers enhanced the hydrophilicity of the composite material, promoting the polymer hydration capacity and the degradative action of microorganisms [89]. Likewise, these additives might inhibit the crystallization of the polymer, promoting the degradation rate of the PHBV polymer [89]. According to Mehmood et al. [90], low-density polyethylene (LDPE) was reinforced with titanium nanoparticles in concentrations of containing 5% (w/w), and the addition of titanium nanoparticles increased the degree of biodegradation. However, although the major studies related increased in the biodegradability due to the action of these nanomaterials, the contrary effect is also valid. Babaee et al. [91] related that a dense structure was obtained in films prepared with plasticized starch (PS)/Chitosan nanoparticles (CNPs) loaded at 1, 2, 3, and 4 wt.% and that their complete biodegradation occurred in more days than neat PS.

Regarding nanomaterials produced from starch, the literature reports that nanoparticles and nanocrystals are found to degrade faster than their macroscopic counterparts due to their higher surface area [92]; however, few studies have reported the effect of these fillers on different polymeric matrices, some of which are shown in Table 9.3.

As shown in Table 9.3, all the studies related to biodegradation in compost or soil media. To our knowledge, no biodegradation studies in aquatic environments have been reported in the literature, which highlights the importance of these type of studies.

Table 9.3 Representative studies of starch nanoparticles influencing biodegradability

Polymer matrix	Nanomaterial	Media	Biodegradability measurement	Major findings	Reference
PLA	Grafted SNCs at 5 wt%	Soil	Weight loss measurement, XPS, FTIR, and DSC	Degradation of PLA/g-SNCs nanocomposites is considerably higher than neat PLA	[93]
HDPE	SNPs at 5 wt%	Air	Thermal degradation, and photodegradation	SNPs decreased the thermal degradation temperature of PE. The photodegradation tests under UV radiation for 28 days showed polymer degradation	[94].
PBAT + starch thermoplastic	SNPs at 0.6 wt%	Compost	Photo Evaluation	Starch nanoparticles promoted faster deterioration in comparison to neat matrix	[95]
Potato starch	Potato SNPs at 0.5, 1, 2, 5, and 10 wt%	Soil	Weight losses	The results showed that the addition of nanoparticles in starch films increases the biodegradability of starch films	[96]
PVA	Corn SNCs synthesized using acid hydrolysis at 0.2–2 wt%	Soil	Weight losses	SNCs improved the biodegradability of the with PVA/S films and was higher than PVA/S film	[97]
PLA	Waxy maize SNCs at 1, 3 and 5 wt%	Dilute alkali-catalyzed hydrolysis	Weight losses	The abiotic degradation studies indicated that WSNC can accelerate the degradation process of PLA	[98]

DSC differential scanning calorimetry, *FTIR* Fourier-transform infrared spectroscopy, *HDPE* high-density polyethylene, *PBAT* poly(butylene adipate-co-terephthalate), *PLA* poly (lactic acid), *PVA* polyvinyl alcohol, *SNPs* starch nanoparticles, *SNCs* starch nanocrystals, *XPS* X-ray photo electron spectroscopy

9.4.4 Encapsulating Agents

The SNPs can be applied in encapsulation systems, which are an attractive alternative for bioactive compounds [31, 99]. The use of nanoamide as a superior encapsulation material is due to its biocompatibility, low viscosity at high concentrations, large surface area, non-toxicity, low cost, and optimal entrapment of bioactive materials [5, 100]. Thus, we have examples of various food ingredients and pharmaceutical application materials that have been encapsulated using SNPs by various encapsulation methods with different efficiencies in the encapsulation process.

According to Ahmad and Gani [101], encapsulation efficiency (EE) determines the amount of core material trapped in the carrier material, and the percentage depends on the number of compounds initially loaded during the encapsulation process. Some studies showed that the highest percentage of EE in starch without modification treatment was shown in vitamin E, with soluble SNPs reaching 91.63%; this finding also indicated that most vitamin E can be retained in SNPs [102]. The highest proportion of EE, above 97%, was produced in conjugated linoleic acid encapsulation using waxy corn starch nanoparticle encapsulation agents; conjugated linoleic acid was effectively trapped in nanostructured particles and can be absorbed with the initial modification treatment, which will effectively increase the absorption efficiency [103]. Similarly, the acetylation of banana starch nanoparticles shows better curcumin encapsulation ability than the nanoparticles without acetylation [104].

Ultrasonication assisted with acid hydrolysis of starch has currently been used to encapsulate antioxidant compounds such as L-ascorbic acid and oxalic acid with high encapsulation efficiency [105]. Horse chestnut, water chestnut and lotus, and lotus starch nanoparticles treated by hydrolysis with sodium hydroxide increased the stability and antioxidant activity of catechin, with the encapsulation efficiency reaching up to 59% and the degradation of catechin encapsulated in SNPs being lower than of unencapsulated catechin, both under digestion conditions [106]. These same starches can also efficiently release resveratrol to specific sites and exhibit higher anti-obesity and anti-diabetic activity than free resveratrol after digestion.

Quinoa SNPs loaded with piroxicam can improve anti-inflammatory activity in vitro, showing around 78% drug release in 0.1 M HCl after 2 h [107]. Unramified starch successfully incorporated epigallocatechin gallate (EGCG) into the nanoparticles with the highest encapsulation efficiency of 84.4%. Furthermore, the resulting nanoparticles showed a controlled release of EGCG in simulated gastric and intestinal fluids. The authors speculated that the developed SNPs could have good prospects as oral release nanocarriers for active compounds or drugs [108]. In recent studies, Escobar-Puentes et al. [109], used succinylated SNPs for the encapsulation of anthocyanins and observed that these systems could be used for the controlled release of bioactive compounds targeting applications such as biodegradable food packaging or as antioxidant additives in the food industry.

In general, results from the literature suggest that SNPs hold significant promise in enhancing the stability, solubility, and availability of bioactive compounds.

Moreover, they exhibit the potential to protect these valuable compounds during food processing and storage, thereby aiding in the development of functional foods.

9.4.5 Antimicrobial Agents

Starch has the remarkable ability to produce a diverse array of SNMts that can effectively stabilize bioactive compounds with antimicrobial properties against foodborne pathogens [110]. These starch-based nanomaterials, which act as stabilizers for antimicrobial compounds, exhibit superior characteristics compared with other natural polymers used as wall materials. They demonstrate faster diffusion rates, higher water solubility, enhanced absorption capacity, and superior penetration rates through biological barriers [110]. Incorporating these nanoparticles as antimicrobial agents in food products can significantly enhance food safety by inhibiting the growth of pathogenic microorganisms. Because of their large surface area, nanoparticles can efficiently attract and immobilize a higher number of microorganisms, leading to increased antimicrobial effectiveness [111].

Furthermore, the development of SNPs allows for the compartmentalization of active substances, particularly stabilizing antimicrobial compounds known for their efficacy [110, 112]. This opens new possibilities for the creation of safer and more stable functional foods with enhanced antimicrobial properties.

Potassium sorbate is considered a GRAS additive (generally recognized as safe) and is known for its antimicrobial properties against yeasts, molds, and various bacteria. However, it is vulnerable to oxidation when exposed to environmental conditions. On the other hand, carvacrol serves as another food preservative and is the primary component found in oregano or thyme oils. It exhibits strong antimicrobial activity against both pathogenic microorganisms and foodborne diseases [110]. Alzate et al. [110], have found that starch-based micro- and nanoparticles, created through antisolvent precipitation (AP), offer a viable method for incorporating potassium sorbate. In another study by the same authors [113], potassium sorbate was successfully stabilized using esterification-modified SNPs. The results showed that the retention capacity of the stabilized potassium sorbate ranged from 41.5 to 90 mg/g, indicating that it is a highly promising antimicrobial agent for food systems. Notably, this approach effectively inhibited potassium sorbate oxidation even under ambient conditions. Fonseca et al. [114] used SNFs to stabilize carvacrol through electrowinning. They prepared solutions containing different concentrations of carvacrol (0%, 20%, 30%, and 40% v/v) with SNFs. The results showed that starch nanofibers loaded with 30% carvacrol led to significant reductions in various pathogens: 89% for *Listeria monocytogenes,* 68.0% for *Salmonella Typhimurium*, 62% for *Escherichia coli*, and 49.0% for *Staphylococcus aureus.* Based on their findings, the authors concluded that starch nanofibers (SNFs) hold immense promise as a vehicle for the controlled release of carvacrol in antimicrobial and antioxidant food packaging applications. Nieto-Suaza et al. [75] employed acetylated SNPs loaded with curcumin, which were then incorporated into films based on banana starch and *aloe vera*. The resulting films demonstrated a controlled release of curcumin when applied to various food products. The researchers suggested that these films have the potential to effectively manage microbial growth in food items, making them promising candidates for food preservation and safety applications.

9.5 Future Trends

Biomass is one of the most promising renewable energy sources, demonstrating its potential as a valuable resource in producing competitive nanomaterials [115]. These nanomaterials are derived from diverse sources such as agricultural waste, forest residues, industrial byproducts, and sewage sludge [115]. Indeed, rice and wheat straws, cotton and corn stalks, sugarcane bagasse, bamboo biomass, shaddock, banana, and rice husk [116], stems, pulp, stem, shell, straw, seeds, husk, peel, stubble, roots, livestock slurry, waste generated during the maintenance of woodlands, and waste from vegetables and fruits hold potential as valuable raw materials to be used in various applications, making them a potential solution for economic, social, and environmental problems [117, 118].

One example pertains to the significant environmental burden posed by potato peel waste, with accumulations reaching several tons per year, resulting in substantial negative impacts [119], minor quantities are sold for supplementary animal feed at very low cost and the rest is discarded as waste [50]. SNPs produced from potato peels have unique properties that are suitable for large-scale applications [119]. Hence, the development of cost-effective techniques for transforming waste potato peel into valuable products is crucial [120]. Another example is cassava starch nanoparticles that could be produced from peels and stems and used in the pharmaceutical industry as a novel drug vehicle [121], which resulted in major advantages such as biocompatibility, biodegradability, easy modification, efficient carrier, and potential targeting [115].

The primary emphasis of incorporating innovative nanomaterials in food packaging has been to enhance the overall quality and safety of products, promote healthier lifestyles, and improve well-being [122]. The advantages of these nanomaterials have been widely studied, but their health implications with it are still in the process of feasibility and verification [122], mainly because of the toxicological effect [123] of nanomaterials due to the risk of migration from packaging to food matrix [122, 123]. Thus, the employment of these materials in the field of food packaging has yet to be allowed due to security concerns.

Consequently, to assess the potential risks, researchers should thoroughly investigate the interactions and stability of nanomaterials in food and feed, as well as their behavior within gastrointestinal systems and biological tissues. In addition, comprehensive studies on the toxicity evaluation of nanomaterials, encompassing chronic exposure and carcinogenicity, are imperative [124].

Another point is related to the use of unconventional starches in the production of nanoparticles, which is a promising and innovative strategy that aims to avoid competition with the food sector while taking advantage of the unique properties of these starches to create materials with diverse applications. To overcome this competition, researchers have explored non-conventional starches, which are obtained from alternative sources, such as agro-industrial residues, non-food plants, and even microorganisms [125].

These unconventional starches can be an abundant and affordable source for the production of nanoparticles with various industrial and medical applications [126]. Different unconventional starches have been explored for nanoparticle production such, as pine seed [127], lotus seed [128], quinoa [47], and pine [129]. By using unconventional starches to produce nanoparticles, several advantages can be achieved: sustainability, low cost, abundance, versatility, and non-food applications.

The production of nanoparticles from unconventional starches is still in the research and development phase. Challenges include optimizing starch extraction and modification processes to obtain the desired characteristics in nanoparticles and ensuring the safety and stability of the final products. In short, the use of unconventional starches in the production of nanoparticles is a promising and sustainable approach that allows exploration of new technological opportunities without compromising the food supply. This approach can contribute to the development of advanced materials with innovative applications in different sectors, thus boosting the economy and scientific research.

9.6 Conclusions

In conclusion, starch nanomaterials (SNMts) offer a promising and innovative solution for functional packaging ingredients in various industries. Starch is one of the most abundant biopolymers on earth, and its nanoscale presents exciting opportunities to enhance packaging material's performance and sustainability.

The unique properties of SNMts, including their biodegradability, renewable nature, and biocompatibility, make them an attractive alternative to conventional packaging additives derived from fossil fuels. These nanomaterials can improve packaging properties, such as barrier performance, mechanical strength, and moisture resistance, while also enabling the controlled release of active compounds for food preservation and other applications.

Moreover, the facility of modification and functionalization of SNMts allows for tailoring their characteristics to meet specific packaging requirements, thereby addressing the diverse needs of different products and industries. By incorporating SNMts into packaging formulations, companies can move toward greener and more sustainable practices, thereby reducing their environmental impact and promoting circular economy principles.

However, challenges related to large-scale production, cost-effectiveness, and compatibility with existing packaging processes must be addressed to fully realize the potential of SNMts in functional packaging applications. Continued research and development efforts are crucial to optimize processing techniques and ensure the safety and regulatory compliance of these novel ingredients.

In conclusion, SNMts represent a promising frontier in the field of functional packaging ingredients. Their eco-friendly nature, versatile properties, and potential for widespread applications make them an essential component in driving the future of sustainable packaging solutions, benefiting both industries and the environment alike. As advancements continue and adoption increases, starch nanomaterials are set to revolutionize the packaging landscape, ushering in a new era of greener, more efficient, and safer packaging materials.

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Conflict of interest The authors declare no conflicts of interest.

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