



Advances in the formation and properties of nanofiber biomaterials from polyvinyl alcohol/carboxymethylcellulose/nanosilver systems for medical applications

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ABSTRACT

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Poly (vinyl alcohol) (PVA) and sodium-carboxymethylcellulose (Na-CMC) are hydrophilic polymers with excellent biocompatibility and biodegradability and have been applied in various biomedical areas due to their favorable properties. Polyvinyl alcohol/sodium-carboxymethylcellulose composites have attracted considerable attention due to the synergistic relationship between the two polymers and the development of novel blends with improved properties. On the one hand, polyvinylalcohol is a versatile polymer with better mechanical properties than sodium-carboxymethylcellulose. On the other hand, sodium-carboxymethylcellulose has high biodegradability and biocompatibility but suffers from poor mechanical properties. Therefore, blending two polymers can benefit from the individual component properties. This review highlights recent progress in the fabrication, properties, and potential applications of polyvinyl alcohol/sodium-carboxymethylcellulose/silver nanoparticles composites based on nanofibers in the fields of drug delivery, food packaging, and biomaterials for curing burns and wounds.

1. Introduction

The electrospinning main setup consists of a high-voltage power supply connected to a spinneret and a grounded collector operating under ambient conditions at room temperature. The solution is charged when a sufficiently high voltage is applied to the solution droplet at the tip of the spinneret, leading to electrostatic repulsion, droplet stretching, and acceleration toward the collector of opposite polarity [1]. The solvent evaporates, leaving polymer nanofibers [2]. Currently, there are two main standard setups for electrospinning: vertical and horizontal, electrospinning has many disadvantages [3]. First, in the fabrication of organic nanofibers, the types of polymers used are limited, and the behavior of the nanofibers is not well understood. In addition, the use of inorganic nanofibers

has been limited owing to their friability after calcination. The high-cost production of electrospun nanofibers with a large diameter represents a significant challenge in nanofiber fabrication.

Most human organs and tissues, such as bone, collagen, dentin, and skin, are present in nanofibrous forms. They are characterized by organized hierarchical fibrous structures that realign at the nanometer scale, motivating and steering most nanofiber research toward biomedical and bioengineering applications [4]. The physical–chemical properties of fibers and fiber mats, such as surface area, diameter, and porosity, which resemble those of the extracellular matrix (ECM), are unusual. This novel morphology enhances cell behavior by segregating tissues from one another and offering promising anchorage and support for cells

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[5]. The characteristics of the ECM may be especially appropriate for determining the functional characteristics of biomaterials. Fiber mats are used to control cell proliferation, migration, and other aspects of cell tissue [6].

Nanofibrous materials are also used in other biomedical applications, such as medical implants, wound dressing, antimicrobial agents, drug delivery vehicles, biomimetic actuators, dental materials, enzyme immobilization scaffolds, and protective textiles for chemical and biological threats [7].

Biocompatible and biodegradable polymers are required to fabricate nanofibers for use in biomedical applications. The polymers may also have to be treated with bioactive cell-organization ligands to perform certain functions [4]. With the release of cytokines, functionalization was found to reduce the inflammatory activation of T cells, mast cells, and macrophages [8]. For tissue engineering approaches, nanofiber scaffolds with seeded cells are incorporated [9]. For wound healing, the porous structure helps drug particles diffuse out of the matrix more efficiently. The drug release rate can be measured by controlling the thickness of the synthesized nanofibrous mat [10]. For drug delivery systems, nanofibrous membranes are implemented with a drug component to deliver the targeted drug to the human body [11].

Electrospun nanofibers exhibit high potential for prosthetic devices used in surgical operations. The soft texture and nature of the prepared fibers make them excellent candidates for use as coatings for hard tissue prosthetic devices. This fibrous coating sheet inhibits device failure by diminishing the stiffness mismatch at the tissue/device interphase by acting as an interphase between the host tissues and the prosthetic system [12]. It has also been reported that nanofibrous materials have been selected as unique candidates for a wide range of tissue prostheses, including breast, vascular, and blood vessel prostheses [13]. In addition, Popryadukhin et al. fabricated vascular prostheses based on nanofibers from a copolymer of ϵ -caprolactam and hexamethylenediamine adipate (HMDAA) [14]. Recently, for liver tissue engineering, Semnani et al. [15] fabricated nanofibrous scaffolds from polycaprolactone (PCL) and chitosan (CS) via a novel collector to improve the pore size and orientation for cell infiltration.

Wound dressings represent a significant issue to be addressed in the biomedical field. The warm, nutritious, and moist environment offered by wound beds provides ideal conditions for microbial growth [16]. Excellent antimicrobial dressings should exhibit good broad-spectrum antimicrobial behavior, provide a moist environment, undergo gas permeation, and perform well against antibiotic-

resistant bacteria to improve healing processes (Fig. 1) [17].

Consequently, to avoid microbial infection, transepidermal water loss leads to the acceleration of wound regeneration [19], and quick care of skin wounds is needed [20]. Therefore, skin barrier restoration is very effective for treating injuries.

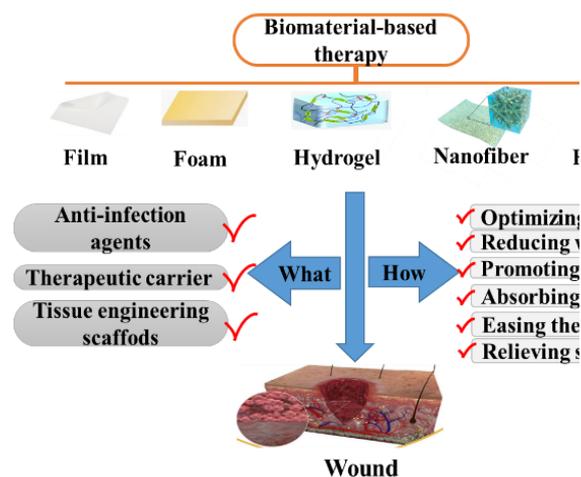


Fig. 1. Desired characteristics of wound dressing products [18].

2. Bionanocomposites were obtained from sodium-carboxymethylcellulose (Na-CMC) and polyvinyl alcohol (PVA).

Na-CMC is a semicrystalline, water-soluble, nontoxic [21], low-cost [22], and biodegradable material [23] with excellent film-forming ability; however, it suffers from low conductivity [24] and a lack of strength [25]. Extensive studies have been conducted on the application of Na-CMC in single polymer electrolyte systems; however, some problems limit its application in this field because of its small elongation at break, exceptionally stiff behavior (less than 8%), and loss of the electrochemical stability required for electrochemical devices. Additionally, a single polymer cannot offer outstanding chemical, mechanical, and physical properties for a wide range of applications, particularly in energy storage devices [26]. Na-CMC has several desirable characteristics, including emulsification, thermal filming, gelation, and inspissation [27]. Owing to its biodegradability and biocompatibility, Na-CMC can be employed for biotechnological and pharmaceutical applications [28]. For the production of Na-CMC, chloroacetic acid reacts with OH groups on the hydroglucose units (AGUs) of cellulose. In the cosmetics and food industries, Na-CMC has been utilized as a water-retention agent and stabilizer [29]. Na-CMC, a highly hydrophilic derivative of cellulose, is extensively used as a suspending and thickening agent in the pharmaceutical and food industries. Because of its good modifiability,

nontoxicity, and swellability, Na-CMC has attracted the attention of scientists in the field of hydrogels for drug delivery applications. Numerous investigations have reported the development of hydrogels based on Na-CMC as carriers for water-soluble drugs [30]. Na-CMC-based hydrogels have the potential to be used for absorption, drug delivery, wound healing, and enzyme immobilization owing to their biodegradability, biocompatibility, and solubility [31].

Na-CMC is a derivative of cellulose composed of carboxymethyl groups ($-\text{CH}_2\text{-COO}^-$) bound to a cellulose backbone [32]. Na-CMC is a highly water-soluble polysaccharide with a wide range of applications due to its biocompatible and biodegradable nature [33]. Along with many other applications, the remarkable binding and stabilizing features of Na-CMC have made it useful in the food industry as an emulsifier and thickener [34]. Furthermore, it is used in textile products as a coating agent and is well exploited in coating colors and printing inks in the pulp and paper industry [35]. Owing to its polymeric structure, Na-CMC has also been used in cosmetic and pharmaceutical formulations, such as in lotions and toothpastes, as well as in soaps and detergents. Na-CMC has been reported to exhibit a pH-sensitive swelling response in which its anionic nature allows it to shrink in acidic media and swell in neutral or anionic environments [36]. With respect to electrospinning, the production of electrospun nanofibers from pure Na-CMC has proven to be difficult because of its rigid structure, which does not allow chain entanglement [37]. Na-CMC is an anionic polyelectrolyte with several ionizable groups. Strong ionic bridges are formed after the ionization of active Na-CMC groups in water, which can couple to many ions with opposite charges. Moreover, the formation of a Na-CMC gel would create a 3D network of large molecules, which would actively hinder polymer chain movement. Therefore, blending Na-CMC with other polymers is viewed as a way out of this challenge.

Purified Na-CMC [38] is widely used in pharmaceuticals and medical practices due to its adhesiveness, biodegradability, biosolubility, and nontoxicity, as well as its ability to form gels, suspensions, microneedles, nanofibers, and films and to possess sorption and stabilizing properties, making it suitable for drug delivery and active substances in the treatment of burn wounds, cancer and hemostatic pharmaceutical devices [39]. Different concentrations of Na-CMC solutions stabilize the synthesis of metal nanoparticles and nanorods, and the size and shape of the carboxymethyl groups can be controlled during synthesis [40].

PVA is the first synthetic colloid that was first developed by Haehnel and Herrmann in 1924 [41]. It was supported by a paper in Hangzhou City, China [42]. Owing to its strong nonirritancy [43], harmlessness, and hydrophilicity [44], among other water-soluble polymers, PVA has been selected for gel preparation [45]. The PVA gel offers several advantages, including easy machinability [46], low toxicity [47, 48], good strength [49], and high water content [50]. Currently, PVA-based materials are extensively utilized in the medical, industrial, and agricultural industries [51].

The manufacturing of PVA consists of vinyl acetate monomer polymerization into polyvinyl acetate (PVAc) and subsequent acetate group hydrolysis to produce PVA [52]. On the basis of the catalyst used, three hydrolysis methods are applicable for PVA preparation. These methods include acidolysis, aminolysis, and alkaline hydrolysis [53]. On an industrial scale, PVAc-to-PVA conversion is usually carried out with alkaline alcohol. In this hydrolysis method, esters are interchanged with methanol in the presence of sodium hydroxide to hydrolyze the acetate groups [54]. The PVA production outline is illustrated in Fig. 2.

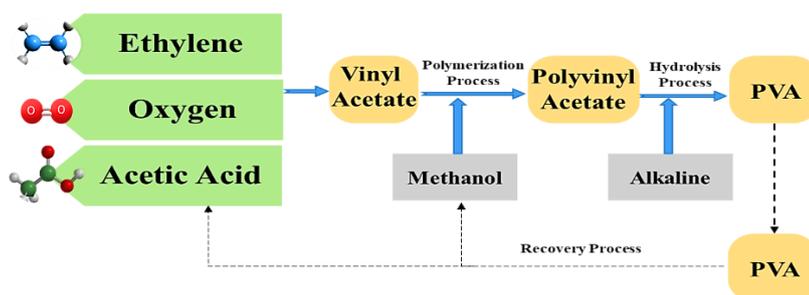


Fig. 2. Production outline of PVA [53, 54].

PVA is a hydrophilic synthetic polymer with semicrystalline, planar zigzag structures and good mechanical properties [55, 56]. PVA is chemically and thermally stable [57] and resistant to degradation under

most physiological environments [58]. Moreover, it is water soluble because of its elevated polarity, nontoxicity [59], and high biocompatibility [60], and it can be processed easily. PVA also has promising

potential for producing biodegradable films [61].

One of the important derivatives of cellulose is Na-CMC, in which carboxylic groups ($-\text{CH}_2-\text{COOH}$) are bound to some OH groups of natural cellulose [62, 63]. According to the literature, the introduction of Na-CMC into the PVA matrix results in improvements in these polymer properties [64]. Therefore, this paper reviews the properties of Na-CMC/PVA composites as well as their applications in the fields of drug delivery, food packaging, and agriculture.

PVA is a biocompatible low-cost synthetic polymer with preferable chemical and thermal stability that is approved for use in many medical procedures [65]. PVA can be an effective tool in the electrospinning of biopolymers for generating dispersive effects and acting as a fiber formation support [66].

To date, the technique has been successfully applied for blending collagen, alginate, hyaluronic acid, chitosan, and Na-CMC with nontoxic and biocompatible synthetic polymers such as polyvinylpyrrolidone (PVP) or PVA, which can improve the processability of biopolymers without affecting their biocompatibility [67].

Nanofibers with diameters of 300 to 150 nm were obtained from an oligoaniline (GelOA)/PVP solution, and it was revealed that the presence of an aniline oligomer leads to enhanced conductivity and thermal properties and a decreased degradation rate and drug release [68].

PVA/aluminosilicate ($\text{Al}_2(\text{OH})_4\text{Si}_2\text{O}_5 \times n\text{H}_2\text{O}$) composite nanofiber with a diameter of 300 ± 20 nm and well-enhanced mechanical properties were successfully prepared by electrospinning. The effects of HNT content on the mechanical properties of PVA/HNT composite nanofibers were investigated, and the results revealed a 72.4% increase in tensile strength at the optimal filling content [69].

3. Silver nanoparticles based on nanocomposites

Silver nanoparticles (Ag NPs) have been applied in antibacterial medical textiles, wound dressings, antimicrobial catheters, medical masks, tissue engineering scaffolds [70], and water purification because of their strong bacteriostatic and bactericidal effects as well as broad-spectrum antimicrobial activities [71]. Ag NPs can be formed from their silver salts via various approaches, such as chemical, physical, irradiation, and biological reduction [72].

Through the in situ thermally induced redox reaction on PVA/ AgNO_3 composite fibers combined with the carbonization of PVA and the reduction of Ag^+ , the synthesized Ag/C fibrous catalyst was prepared with nanosilver particles with an average diameter of 130 nm immobilized on loose microstructural carbon layers.

The synthesized Ag/C fibrous catalyst exhibited excellent catalytic activity and was reused for at least five cycles for the reduction of Ag NPs, which may hold great promise for effective and eco-friendly wastewater treatment [73].

Using chemical methods, sodium borohydride Ag NPs with a size of 43.33 ± 5 nm were incorporated into regenerated cellulose nanofibers with different concentrations and sizes ranging from 300–400 nm in diameter, which were used as antimicrobial agents against gram-negative *Escherichia coli* BH5 α , gram-positive *Spectromyces arenus*, and *Aspergillus flavus*, and strong inhibitory effects were determined [74].

Nanofibers of PVA/CS/hyaluronic acid containing hormones with diameters of 261–288 nm were prepared by electrospinning. The obtained nanofibers were highly hydrophobic, and for 48 hours, 61% of the hormones were released from the nanofibers [75].

Nanofibers of poly (lactic acid) containing silver sulfadiazine/[Mg-Al]-layered double hydroxide (LDH) were obtained by electrospinning. PLA nanofibers containing silver sulfadiazine/[Mg-Al]-LDH have been shown to have significant inhibitory effects on *Escherichia coli* and *Staphylococcus aureus* [76].

Pant et al. [77] reported the synthesis of nylon-6 nanofibers incorporated with Ag NPs. Methoxy poly (ethylene glycol) and formic acid were used as electrospinning solvents to convert AgNO_3 to Ag NPs. The study revealed that the fabricated Ag/nylon-6 composite nanofibers had an isotropic morphology, with Ag NPs dispersed uniformly throughout the polymer galleries. Furthermore, the composites have excellent antibacterial properties for use in biofilms, wound dressings, and filtration. Shamshi Hassan et al. [78] described the synthesis of electrospun nanofibers from bimetallic ZnO/Ag-embedded polyurethane. The incorporation of Ag and ZnO particles into the prepared materials resulted in an enhanced bactericidal effect without any harmful effects on normal mouse fibroblasts. This behavior confirmed the benefit of using these mats for filtration, clinical, and textile applications.

4. Synthesis of Na-CMC/PVA composites

Grafting, crosslinking, and degradation are ways to improve or modify polymer materials. Owing to its compatibility, PVA can be modified by Na-CMC, and this characteristic is the result of the hydrogen bonding between the OH groups in PVA and the carboxymethyl groups on Na-CMC. The chemical structures of PVA and Na-CMC and their interactions are illustrated in Fig. 3. Fig. 4 also shows the crosslinking of PVA and Na-CMC by cellulose acetate (CA).

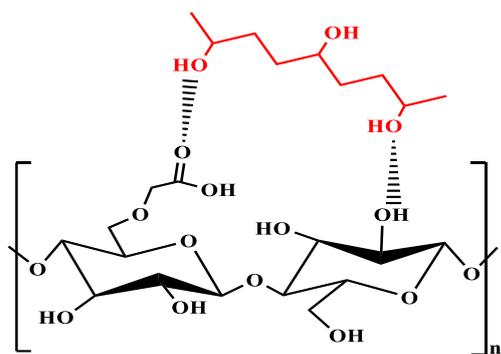


Fig. 3. Molecular interaction of PVA and CMC.

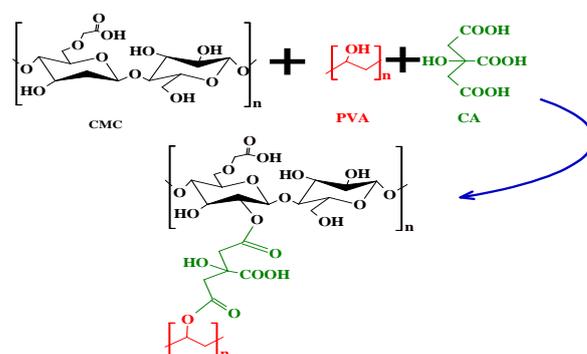


Fig. 4. Crosslinking of PVA and CMC by CA.

Zhang et al. [79] prepared a potential coating material based on a Na-CMC/PLA blend film. Biodegradable Na-CMC–PVA blend films were successfully prepared via intra- and intermolecular crosslinking reactions. Owing to its favorable biodegradability, the prepared blended film has environmentally friendly characteristics and can be used as a potential material for controlling the release of fertilizer coatings.

Using a solution-casting method, Sayed et al. [80] incorporated copper oxide and PVA nanoparticles into Na-CMC films and investigated their electrical, optical, and structural properties. At low temperatures, Schottky emission was the conduction mechanism in the Na-CMC/PVA blend. After the addition of PVA, the transparency of the Na-CMC film increased from 87% to 89%. The results showed that the optical constant, refractive index, and insulating properties of Na-CMC were controlled by blending Na-CMC with PVA. Additionally, nonohmic behavior was observed in the current–voltage characteristics of the blend.

Saadieh et al. [81] developed biopolymer blend electrolytes based on Na-CMC/PVA via the casting solution method. The highest ionic conductivity (9.12×10^{-6} S/cm) was reported for the blend electrolytes with a Na-CMC to PVA ratio of 80:20 at room temperature. According to the results, a decrease in the dielectric loss and dielectric constant was observed with increasing frequency. This was reported to be the result of ion accumulation contributed by the electrode, ionic polarization, and molecular polarization.

The Na-CMC/PVA blend was synthesized via the solution casting method and subsequent γ -ray irradiation. They proposed that γ -ray irradiation could control the physical properties of this copolymer. Therefore, they suggested the use of a Na-CMC/PVA blend for several electronic and industrial devices because of its improved charge storage capacity and dielectric strength [82].

5. Properties of the CMC/PVA composites

The physical and chemical properties of polymers are influenced by their chemical structure. The flow and morphology of polymers depend on their chemical structure, which leads to different physical properties [83]. Using a casting method in the presence of a glycerol-containing plasticizer, Taghizade et al. [84] developed Na-CMC/PVA/starch (S) composite films. According to the results, the Na-CMC/PVA/S blends presented greater thermal stability than did the PVA/S blends. This was reported to be due to the addition of Na-CMC, which improved the thermal stability of the PVA/S blend.

Miao et al. [85] prepared a negatively charged nanofiltration membrane based on a Na-CMC/PVA composite produced via interfacial polymerization. The resulting composite membrane exhibited high long-term stability. According to this study, the cross-linked PVA-based composite could provide a good balance between salt rejections and permeate flux under lower operating pressures. Tajeddin et al. [86] reported water absorption of 22.03% for a CMC/PVA blend film prepared by the casting method after 24 hours. Zhu et al. [87] reported that a pure PVA film had low water sorption, which was enhanced by adding Na-CMC to the composite film. This was reported to be due to the blending of two different molecule types, resulting in structural deformation and the formation of more hydrophilic networks. Furthermore, the solubility of the composite was approximately 70% after 24 h.

Polymer blend films are widely used for metal ion adsorption [88]. Wang et al. [89] prepared Na-CMC/PVA hydrogels via the freeze–thaw process for heavy metal ion adsorption. The formation of crystallites occurred due to the separation of phases in the polymer solutions during the freezing stages, leading to 71% insoluble hydrogels. The swelling ratio for the pure PVA hydrogels was 416%, whereas a higher swelling ratio of 1437% was obtained for the Na-CMC/PVA hydrogel containing one-third of the Na-CMC and two-thirds of the PVA. This hydrogel had Ag^+ adsorption capacity of 8.4 mg/g. The prepared hydrogels have potential applications for wastewater treatment and the removal of heavy metal ions.

Abutalib et al. [90] studied the dielectric and electrical characteristics of Na-CMC/PVA/ZnO nanorods prepared via the casting method. The results showed that the dc conductivity exhibited Arrhenius behavior that increased with temperature, whereas the ac conductivity followed Jonscher's law. These nanocomposites have been proposed as potential candidates for applications such as electrochemical devices because of their significant improvement in ac and dc conductivities.

In a study conducted by Goswami et al. [91], bionanocomposite films of Na-CMC/PVA/V₂O₅ were prepared by solution casting of the Na-CMC/PVA thin film and subsequent impregnation of V₂O₅ into the film. The differential scanning calorimetry (DSC) results revealed a lower transition temperature for the Na-CMC/PVA/V₂O₅ nanocomposite than for the Na-CMC/PVA film, making it suitable for smart windows. The total conductivity results demonstrated the good electrical behavior of the prepared bionanocomposite. Therefore, the electrical and optical properties of Na-CMC films have been reported to be significantly enhanced in the form of Na-CMC/PVA/V₂O₅ nanocomposites, enabling them to be used in applications such as storage devices and smart windows.

El-Newehy et al. [92] used the electrospinning technique to prepare Na-CMC/PVA nanofibers for the controlled release of diclofenac sodium (DCS). The study revealed that the Na-CMC/PVA nanofiber blend exhibited good mechanical properties. Additionally, the in vitro release study revealed that the presence of Na-CMC led to the sustained control of DCS release from the nanofiber mats. The Na-CMC/PVA nanofiber system could be a promising material for drug delivery applications because of the low cost and biocompatibility of the blend. The general applications of the CMC/PVA composites are shown in Fig. 5.

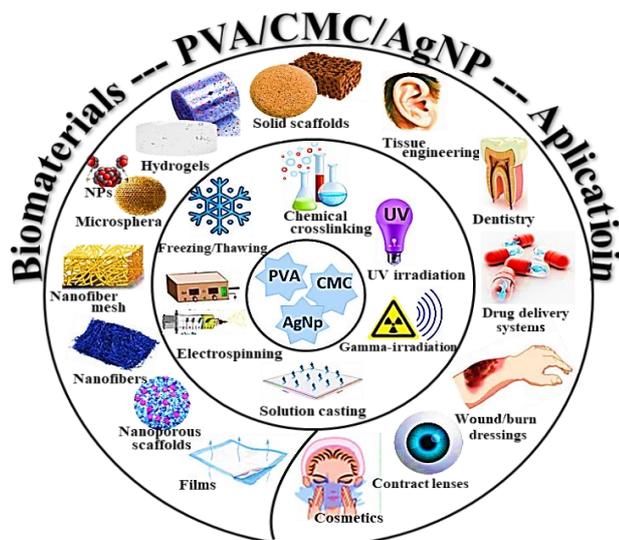


Fig. 5. Applications of Na-CMC/PVA/Ag NPs composites [93].

6. Applications of the CMC/PVA composites

7. Application in food packaging

Innovative food packaging is active packaging in which advances in material sciences, packaging, food safety, and food technology are combined to satisfy consumer demand for safe and fresh-like products. Because of environmental considerations over the past few years, the use of synthetic polymers has been restricted. Biopolymers, including proteins, lipids, and polysaccharides, have received increasing amounts of attention. Active agents can be incorporated into the polymer structure in active packaging, leading to a low diffusion rate of active compounds and the maintenance of high concentrations of active agents on the surface of packaging materials during shelf-life [94].

In the cosmetics and food industries, Na-CMC has been utilized as a water retention agent and stabilizer. PVA has also been incorporated to improve mechanical properties [95].

Todjiyev et al. [96] fabricated Na-CMC/PVA films with clove oil via the casting method as active packaging for ground chicken meat. The results demonstrated an increase in the tensile strength and puncture force of the Na-CMC film and a decrease in the water vapor transmission (WVP) rate with increasing PVA concentration. A negligible oxygen transmission rate was observed in all the samples. During refrigerated storage, the control samples spoiled after 4 days, whereas the packing of the meat samples in the prepared films resulted in lower total viable counts and a shelf life of 12 days. The efficacy of Na-CMC/PVA/clove oil films against *Bacillus cereus* and *Staphylococcus aureus* in ground chicken meat was also studied. It was proposed that the prepared films have great potential for the active packaging of meat products. Fasihi et al. [97] produced biodegradable active films based on Na-CMC/PVA/oleic acid (OL) containing rosemary essential oil (REO) via Pickering emulsions. The results demonstrated that the films containing REO had considerable antimicrobial and antioxidant properties. In the films with 3% REO, the fungal inhibition against *Penicillium digitatum* was 100%. In bread slices that were packed with the active films containing 3% REO, no fungal growth was observed at 25°C after 60 days of storage. This might be due to the regular, slow release of REO resulting from the Pickering emulsions.

Villarruel et al. [98] developed blend films based on Na-CMC and PVA and modified them with UV radiation in the presence of sodium benzoate (SB). According to the results, the newly developed materials presented different chemical and thermal stabilities than the single components did. Both blend films and UV-induced films exhibit very low oxygen barrier

properties, making them suitable materials for packaging applications with selective oxygen permeability. Moreover, UV treatment of the films containing SB inhibited the growth of a wide spectrum of microorganisms and increased their insolubility in water, making them potential materials for use as food packaging emulsions.

6.2. Applications in biomedicine

Considerable effort has been made to improve the efficacy of drug delivery by investigating new materials [99, 100]. In this context, natural polymers play a substantial role in biomedical applications [101]. Recently, drug delivery systems based on natural polymer hydrogels have attracted the attention of researchers [102]. Na-CMC is a cellulose derivative with high hydrophilicity and is widely applied as a suspending and thickening agent in the pharmaceutical industry [103]. The desirable properties of Na-CMC hydrogels, such as their modifiability, nontoxicity, and good swellability, have encouraged scientists to use this material for drug delivery [104].

One of the pressing public health issues is burn treatment. Despite efforts made for its prevention, such as public campaigns, its worldwide incidence is high. Dressings are usually required for the treatment of second-degree burns. Cell hydration through maintaining a moisturized environment and promoting necrotic tissue debridement are important properties of an ideal dressing. It should also be able to successfully remodel tissue and maintain transparency to monitor healing. Other characteristics include infection inhibition, pain reduction, nontoxicity, and vapor and oxygen transmittance. Most of these properties are offered by hydrogels; therefore, they have been utilized as biomedical devices for wound treatment. They can be permeable to oxygen while acting as barriers to microorganisms [105]. Hence, hydrogels are considered patient-friendly systems for drug delivery [106]. Many wound dressing materials are hydrogel dressings because their advantages outweigh their disadvantages. Fig. 6 schematically shows the role of the hydrogel membranes in the wound healing phase.

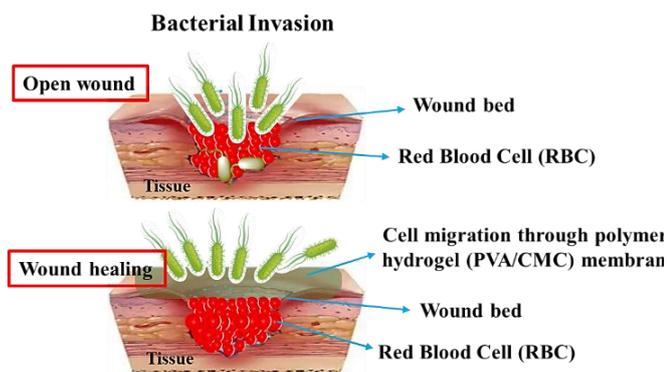


Fig. 6. Role of hydrogel membranes in accelerating and enhancing the wound healing phases[107].

Hydrogels are hydrophilic polymers that are cross-linked and swell in aqueous media while their structural integrity is maintained. However, hydrophobic drugs can be loaded in low amounts because of the hydrophilic network of hydrogels. To overcome this restriction, polymer chemistry investigations have focused on the development of a variety of network structures. Owing to its biodegradability and biocompatibility, Na-CMC can be utilized in biotechnological and pharmaceutical applications [108].

Another option for the production of biocompatible hydrogels is PVA, which has a semicrystalline structure. PVA hydrogels are able to deliver moisture and absorb exudate from the wound site. PVA gels can be physically or chemically crosslinked. Fig. 7 shows the synthesis of the CMC/PVA hydrogel.

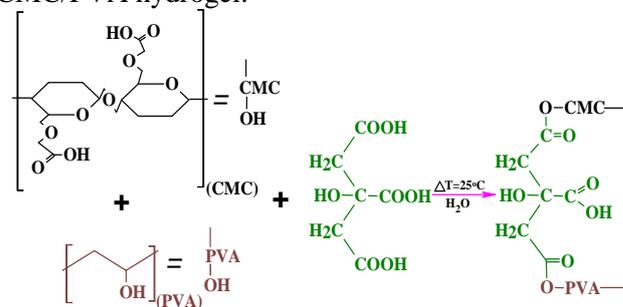


Fig. 7. Synthesis and the mechanism of the crosslink reaction of the of PVA-CMC hydrogels.

Polymeric blend films were developed for the extended release of water-soluble drugs on the basis of citric acid cross-linked Na-CMC/PVA (Fig. 7). The model drug gentamicin sulfate (GTM) was used. The incorporation of PVA increased the mechanical strength of the Na-CMC/PVA hydrogel films. The swellability of the hydrogels was improved by increasing the PVA content in the hydrogel films. Thus, the developed hydrogel films are considered promising biomaterials for the delivery of basic drugs that are soluble in water.

6.3. Application in agriculture.

Blended synthetic polymer films have the potential to be used as coating materials for controlling fertilizer release. However, owing to their nonbiodegradability and high poison content, some blended films might cause severe soil pollution. Therefore, the development of natural polymer-based films with biodegradability, nocity, and low price is critical for the production of coated fertilizers [109].

Na-CMC/PVA blend films were produced via a cross-linking reaction between Na-CMC and PVA for application as coating materials. The results indicated that the prepared Na-CMC/PVA films had smooth

surfaces. A decrease in the water permeability and absorbency of the blended films was observed as the PVA content increased [110].

The fabrics were precoated with PVA, and cross-linking with Na-CMC was carried out with citric acid. According to the results, the PVA precoating and subsequent cross-linking of Na-CMC led to a synergistic effect for a considerable increase in fertilizer release management and moisture. Obtaining a controlled release of fertilizer with improved water absorption/retention behavior is an outstanding step forward in the field of efficient sustainable agriculture.

8. Synthesis of Ag NPs in a Na-CMC/PVA matrix on a nanofiber mat

Stable Ag NPs in solutions containing Na-CMC and PVA were synthesized, and their structure, morphology and physicochemical properties were studied [111]. The morphology and diameter of the Na-CMC/PVA nanofibers containing silver nanoparticles were studied via atomic force microscopy and scanning electron microscopy. Investigations have shown that nanofibers with diameters of 50 ± 15 nm can be obtained from Na-CMC/PVA/Ag NP solutions [112].

The size and form of the Ag NPs that formed in the nanofiber structure were determined via XRD, UV-VIS spectroscopy and dynamic light scattering, and nanoparticles with diameters ranging from 5–26 nm were observed. The nanofiber mats containing Ag NPs exhibited significant antibacterial activity against both *Staphylococcus epidermidis* and *Candida albicans*. Nanofiber mats containing stable Ag NPs could be used as bactericidal facemasks for air filtration and for the treatment of burn wounds [113].

Various factors, such as the Na-CMC, PVA and AgNO_3 solution concentrations, irradiation wavelength and time, affect the reduction efficiency and stability as well as the shape and size of the Ag NPs [114] and the structure of the nanofibers.

The influence of various factors on the formation of nanofibers is crucial for controlling their properties and achieving the desired morphologies [115]. The properties of the polymer solution, such as viscosity, surface tension, and conductivity, play a significant role in nanofiber formation. These properties affect jet stretching during electrospinning and ultimately determine the fiber diameter and morphology [116]. The concentration of the polymer solution affects the viscosity and solution properties, thereby influencing the fiber diameter and morphology. Higher concentrations typically result in thicker fibers due to increased solution viscosity and stretching behavior [117]. The choice of solvent or solvent mixture for dissolving the polymer affects its solubility and solution properties. Different solvents can alter the drying

kinetics and electrospinning process, leading to variations in fiber morphology [118].

For visual determination of the possibility of nanofiber formation, various concentrations of Na-CMC and PVA at different ratios were subjected to electrospinning.

The identical electrospinning conditions for a 2% volume ratio of 80/20 Na-CMC led to facile polymer and nanofiber production. A lower Na-CMC/PVA ratio of 2% Na-CMC resulted in suitable nanofiber formation only at a 30/70 ratio. This could be attributed to the high viscosity at lower ratios. With the exception of the 0.5% Na-CMC/PVA solution, electrospun nanofibers were successfully formed with a 50/50 volume ratio of Na-CMC/PVA solution at a 1–3% concentration. These results confirmed that a higher Na-CMC viscosity leads to more uniform nanofiber formation, where poor formation is effectively prevented by increasing the viscoelasticity. Next, the conditions for the replacement of sodium ions in Na-CMC with silver ions (Ag^+) were studied.

The separated solutions of Na-CMC with a degree of substitution of 0.97 and degrees of polymerization of 850 and PVA were mixed at volume ratios of 100:0 and 50:50, and when 0.50 mol.% Ag^+ was added, a PVA/ CMC^-/Ag^+ polymer–metal complex hydrogel was formed as a result of Ag^+ and Na^+ ion exchange. The addition of Ag^+ to a PVA/Na-CMC solution increases the viscosity of the solution owing to the decreased solubility of the PVA/ CMC^-/Ag^+ complexes generated because of the formation of coordination bonds between the carboxylate groups ($-\text{COO}^-$) of the Na-CMC macromolecules and Ag^+ . Adding 0.5 mol.% to Na-CMC/PVA at a ratio of 0:100 led to an insignificant decrease in the solubility and an increase in the viscosity without gel formation.

When the Na^+ ions in Na-CMC with a DS of 0.97 are replaced by 0.20, 0.30, 0.40 and 0.50 mol.% Ag^+ ions, Ag^+CMC^- hydrogel complexes with poor water solubility are formed. Most likely, the carboxyl groups of Na-CMC are able to form complexes [119] with silver ions. The critical concentration of Ag^+ that causes the formation of the Na-CMC hydrogel was 0.5 mol.%.

Aqueous solutions of Na-CMC exhibit polyelectrolyte characteristics [120]. Unlike other alkali metals, Ag^+ reacts with carboxymethyl groups ($-\text{CH}_2\text{COO}^-$) in Na-CMC solutions and forms a polymer-metal complex, Ag^+CMC^- with low solubility in water, which is explained by the formation of ion–coordination bonds between them.

Next, the formation of stable Ag NPs in the structure of Na-CMC/PVA/ Ag^+ solutions via the photochemical reduction method [121] was studied.

The formation of Ag NPs in Na-CMC/PVA structures under the influence of photolysis can be considered an electron-stimulated atomic process, and

its mechanism is explained by the Gurney–Mott theory. The reaction sequence was subsequently determined according to the Mott–Gurney mechanism [122].

For the formation of stable Ag NPs from the Na-CMC/PVA/Ag⁺ solution, first, the carboxymethyl

groups (-COO⁻) in the Na-CMC macromolecules act as “traps” for silver cations (Ag⁺) [123] and gather them around, and the reduction of Ag⁺ proceeds via the following mechanism. (Fig. 8.)

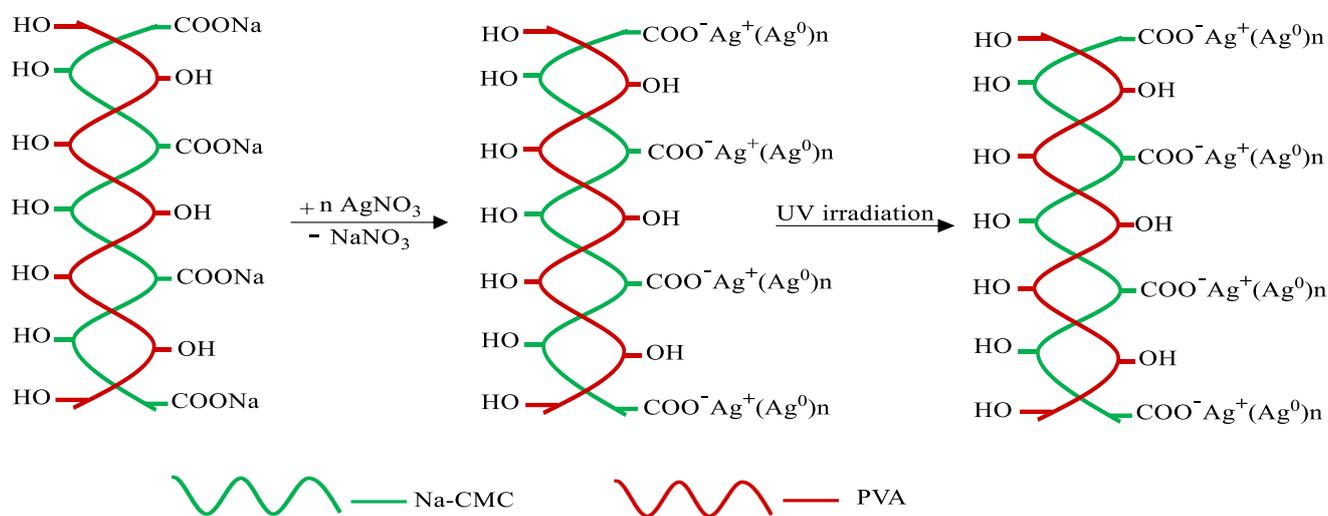


Fig. 8. The photochemical reduction process of silver cations in a PVA/Na-CMC⁻/Ag⁺ solution.

Thus, the photostimulated formation of Ag NPs in the Ag⁺CMC⁻ hydrogel can be considered an electron-stimulated nuclear process on the basis of the Mott–Gurney theory, as in the case of photography.

The FT-IR spectra provided additional information about the functional groups of CMC, CMC/PVA, CMC/PVA/Ag⁺ and CMC/PVA/Ag⁰. The prominent FT-IR peaks of CMC and PVA indicated the presence of O–H stretching vibrations and intermolecular H bonding at 3419.3 and 3445.2 cm⁻¹, respectively. The sharp band at 1734.7 cm⁻¹ corresponds to the C=O stretching of the acetate group of PVA, and the backbone aliphatic C–H stretching vibrations give rise to sharp bands at 2938.7 and 2854.5 cm⁻¹.

The strong absorption peak at 1098.0 cm⁻¹ was assigned to the C–O stretching of PVA, and the band at 1377.8 cm⁻¹ was attributed to the combination of CH–OH.

The IR spectrum of Na-CMC shows absorption bands due to C–H stretching at 2912.7 cm⁻¹ and due to C–O stretching of the ether group of the carboxymethylation of cellulose or the ether linkage [1,4-d-glucoside] of cellulose at 1118.0 and 1059.6 cm⁻¹.

The absorption spectra in the 1423.2–913.5 cm⁻¹ wavenumber range characterize the plane deformation fluctuations of the hydroxyl groups in the Na-CMC sample, and the peak attributed to carboxyl groups (COO⁻) formed in the 1605.1 cm⁻¹ absorption field.

The CMC/PVA nanofibers presented characteristic peaks at 1598.6 and 1379.4 cm⁻¹, which are attributed to the stretching vibrations of C=O and

CO, respectively, and the peaks at 1419.9 and 1326.3 cm⁻¹ are attributed to the bending vibrations of CH.

A peak at 1589.3 cm⁻¹ was observed in the FT-IR spectrum of the Na-CMC/PVA/Ag NP nanofiber mat. Na-CMC characterizes the binding of carboxyl groups and silver ions (-COO⁻Ag⁺) to ion coordination bonds in macromolecules.

Next, the sizes and shapes of the Ag NPs formed in the PVA/Na-CMC solutions were characterized through UV–Vis spectroscopy and DLS.

When the PVA/Na-CMC/Ag⁺ solution was irradiated with UV light in air at room temperature, the solution first became more yellow–brown than dark-brown, indicating the formation of silver clusters. After prolonged irradiation, the irradiated surface of the Ag NPs was worn for approximately 30 min, after which the Ag NPs became agglomerates of Ag NPs after 60 minutes of UV irradiation.

Before irradiation, a wide absorption band was observed in the spectrum, and in the 280 nm region, an absorption band appeared in the form of a shoulder due to the transfer of electrons from RCOO⁻ to Ag⁺ ions. When the samples were irradiated with light at a wavelength of 254 nm, absorption bands appeared in the wavelength range from 250 nm to 450 nm, the intensity and maxima of which varied depending on the irradiation time.

These bands are assigned to photolytic silver [124].

After 5 minutes of UV irradiation of Na-CMC/PVA solutions containing Ag⁺, a stable colloidal system of nanosilver with a pale–yellow color is formed at the absorbance maximum at $\lambda_{\text{max}} = 290$ nm, at which

point silver clusters begin to form [125]. No changes were observed for the initial CMC/PVA solutions or the unreduced Ag^+ in the CMC/PVA/ Ag^+ in the spectral region of 200–800 nm.

With an increase in irradiation time to 10 min, a bathochromic shift is observed, the absorption band appears at 310 nm, and a hyperchromic effect is also observed. This is due to the formation of stabilized Ag NPs with a diameter of 5–26 nm, the sizes of which were determined by DLS.

After photolysis of the CMC/PVA/ Ag^+ solution for 20 min, an absorption band corresponding to particles with a multimodal distribution appears, the maximum intensity of which is observed at $\lambda_{\text{max}} = 365$

nm, which corresponds to Ag NPs with a size of 30–65 nm. After 30 min of UV photolysis of the CMC/PVA/ Ag^+ solution, the absorption band at $\lambda_{\text{max}}=400$ nm was attributed to larger Ag NPs with sizes of 45–86 nm. Further irradiation of the CMC/PVA/ Ag^+ system for 60 min led to the appearance of absorption bands with maxima at $\lambda_{\text{max}} = 420$ nm and $\lambda_{\text{max}} = 340$ nm. This is due to the increase in the number of larger Ag NPs with diameters ranging from 5–35 nm with changing nanoparticle shape [123].

Next, the diameter and morphology of the Na-CMC/PVA and Na-CMC/PVA/ Ag NP nanofibers were studied via AFM (Fig. 9).

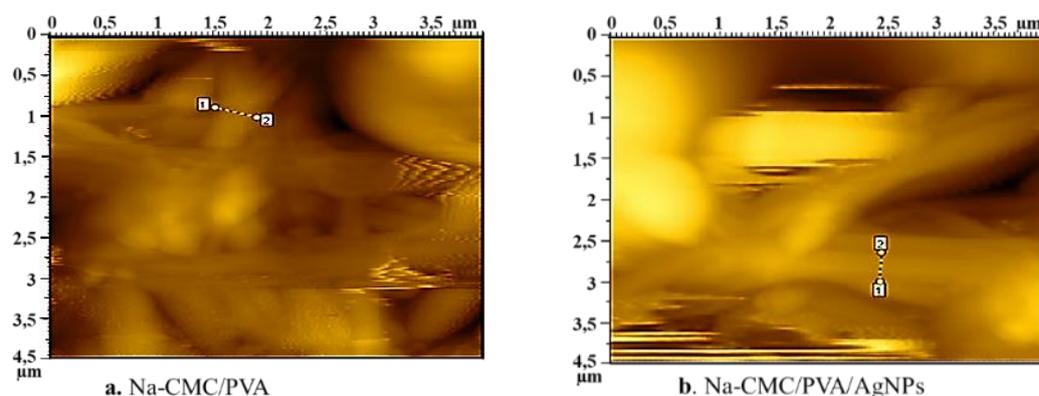


Fig. 9. Results of AFM studies of diameter and morphology of nanofiber obtained from Na-CMC/PVA and Na-CMC/PVA/ Ag NPs.

The surface of the Na-CMC/PVA nanofibers was smooth, with an average diameter of $90\text{--}183\pm 30$ nm (Fig. 9b). After treatment with AgNO_3 solution as well as UV radiation, many AgNPs were deposited on the surface and in the network of the nanofibers, indicating successful reduction and restoration of Ag^+ ions to metallic Ag NPs (Fig. 9b). However, the average diameter of the Na-CMC/PVA/ Ag NP nanofiber, with sizes of approximately $60\text{--}156\pm 23$ nm, was smaller than that of the Na-CMC/PVA nanofibers. The reason for this difference could be the high viscosity of the Na-CMC/PVA/ Ag NP solution, in which the balanced force among surface tension and viscoelastic and electrostatic forces could still achieve stable jet flow during the electrospinning process.

Next, the diameter and morphology of the nanofibers obtained from the Na-CMC/PVA, CMC/PVA/ Ag^+ and Na-CMC/PVA/ Ag NPs solutions were studied via SEM (Fig. 10).

The charge density, surface tension, and viscoelastic properties of a polymer solution are very

important parameters for the successful electrospinning of polymers [127].

The nanofibers formed from the Na-CMC/PVA solution at a volume ratio of 50:50 had an average diameter of $80\text{--}160\pm 20$ nm and a smooth surface (Fig. 10-a). During the electrospinning of a CMC/PVA solution containing silver ions (Ag^+), “beaded” nanofibers [128] are formed, and the average diameter of the nanofibers decreases to $60\text{--}110\pm 20$ nm (Fig. 10-b). The reason for this might be that the surface tension mainly affects the surface area of the beads. In the case of no surface tension, the jet breaks down into drops. A lower surface tension tends to result in the formation of more beads in the electrospun products. Figure 10-c also shows that the average diameter of the CMC/PVA/ Ag NP nanofibers ranged from $50\text{--}130\pm 20$ nm.

The reversion of silver ions to silver metal under the action of an electric field in the electrospinning process [129] can also cause defect formation in nanofibers. It is advisable to conduct more in-depth scientific research in this direction.

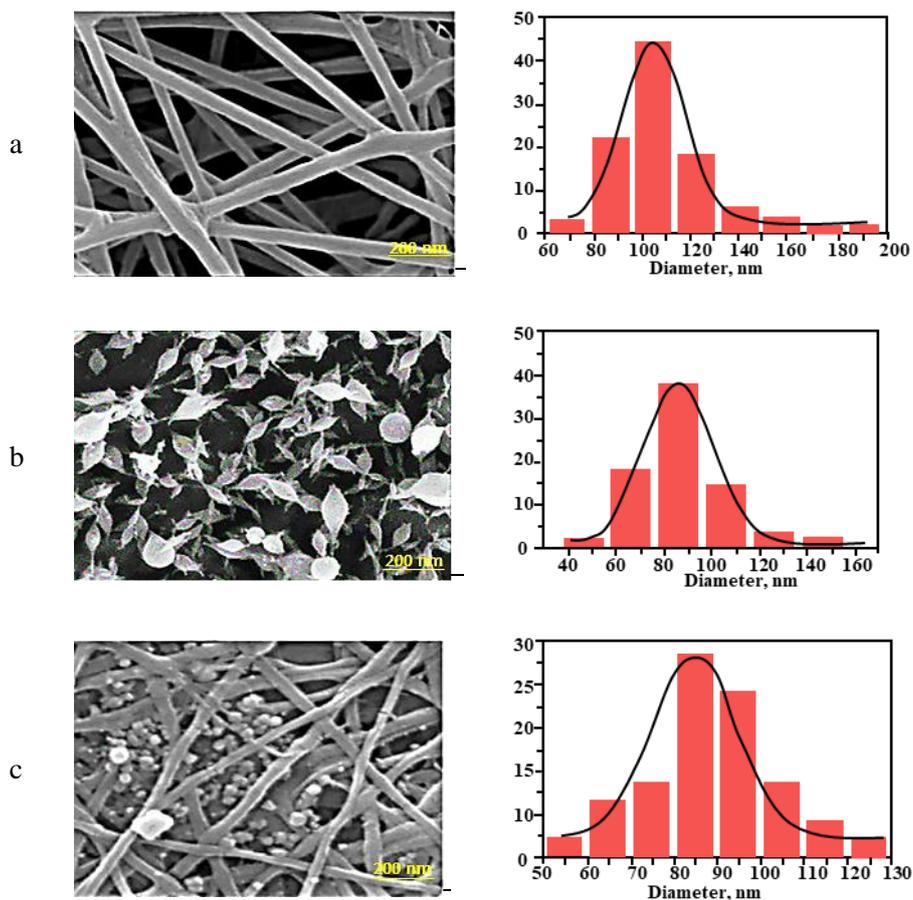


Fig. 10 SEM images of Na-CMC/PVA (a), CMC/PVA/Ag⁺ (b) CMC/PVA/Ag⁰ (c) nanofibers

SEM cannot be used to observe the optimal Ag NP size distribution in nanofiber structures because of the low voltage used to monitor the electron beam reflected by the samples. Because of this low voltage, the obtained electron beam has limited energy, so the

depth of electron penetration into the Na-CMC/PVA polymer is small.

To determine the structure of the Ag NPs formed from the Na-CMC/PVA/Ag NP nanofibers, X-ray studies were carried out (Fig. 11).

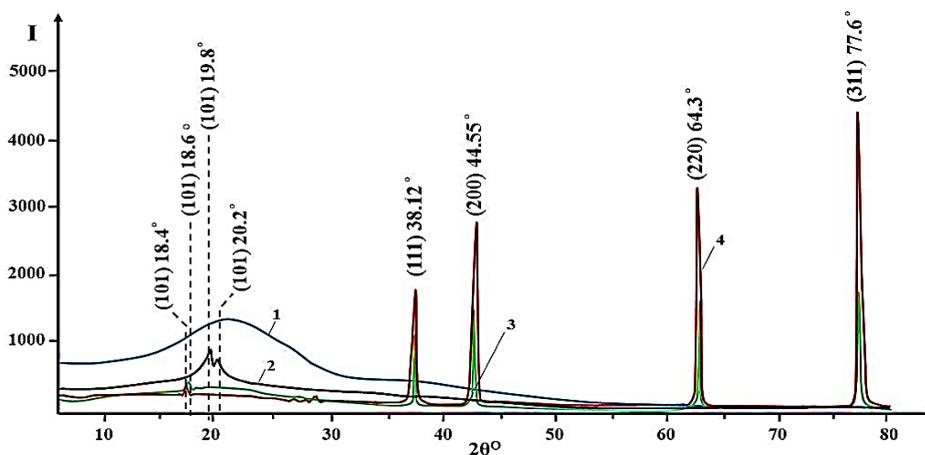


Fig. 11. Diffractogram of a Na-CMC film (1) and nanofibers formed from a Na-CMC/PVA solution (2), a Na-CMC/PVA/Ag⁺ solution (3), and a Na-CMC/PVA/Ag NP solution (4).

X-ray diffraction analysis (Fig. 11, line 1) revealed that the Na-CMC film had an amorphous structure with an amorphous halo at $2\theta=21.6^\circ$. In the nanofibers obtained from the Na-CMC/PVA solution, an amorphous structure appears at $2\theta=19.8$ and 21.6° for the (101) plane (Fig. 11, line-2).

In the diffractograms of the nanofiber mats obtained from the Na-CMC⁻/PVA/Ag⁺ and Na-CMC/PVA/Ag NPs solutions, 2 phases were identified: the first phase was an amorphous phase, and the second phase was composed of Ag NPs (Fig. 11, lines 3 and 4).

The results of the X-ray structural analysis revealed that the Ag NPs had a cubic syngony, crystal lattice parameters of $a=b=c=4.086 \text{ \AA}$ and $a=b=c=90^\circ$, and diffraction angles of $2\theta=38.12^\circ$, 44.55° , 64.3° and 77.6° , exhibiting crystal reflections characteristic of the (111), (200), (220) and (311) reflection planes, respectively (Fig. 11, lines 3 and 4).

Peak broadening was used to estimate the size of the Ag NPs crystallites. The size of the crystallites affects the width of the X-ray diffraction lines [130]. These regions with larger sizes have narrower reflections in the diffractogram. Scherrer's equation revealed that the average size of the Ag NPs crystallites in the nanofibers was 22 nm.

The antimicrobial activities of the nanofiber samples containing silver cations and Ag NPs were examined against pathogen test cultures of

Staphylococcus epidermidis and *Candida albicans*. The abilities of the nanofibers to inhibit the growth of the test bacteria are presented in Table 1.

For identification of antimicrobial effects, the samples were placed into test tubes containing thioglycolic media in the case of *Staphylococcus epidermidis* or Saburo media in the case of *Candida albicans*. The test tubes were numbered as follows: (1) control strain and CMC/PVA nanofibers; (2) CMC/PVA/Ag⁺ nanofibers containing 0.263 wt.% AgNO₃; (3) CMC/PVA/Ag⁰ nanofibers containing 0.009 wt.% AgNO₃; (4) CMC/PVA/Ag⁰ nanofibers containing 0.263 wt.% AgNO₃; and (5) CMC/PVA/Ag⁰ nanofibers containing 2.63 wt.% AgNO₃.

The control samples were prepared by adding the CMC/PVA nanofibers to 10 wt.% physiological solutions in the same media. Six-hour test cultures with a final concentration of 1000 cells/mL were added to each test tube. The samples were incubated at 34°C for 48 h for *Staphylococcus epidermidis* and for 72 h for *Candida albicans*. Microbiological tests revealed that all the samples exhibited antimicrobial activity against these opportunistic pathogens in humans.

The concentration-dependent antimicrobial efficacy of Ag NPs at different concentrations on the abovementioned organisms is shown in Table 1.

Table 1. Antimicrobial activity of Na-CMC/PVA nanofibers containing silver cations and AgNPs against strains at a concentration of 10^3 CFU mL^{-1} .

№	Nanofiber samples	Ag NPs content, wt%	Diameter size of nanofiber, nm	Strains	
				<i>Staphylococcus epidermidis</i>	<i>Candida albicans</i>
1	Control MC/PVA	-	80–160±25	$5 \times 10^{12} \text{ CFU mL}^{-1}$	$1 \times 10^8 \text{ CFU mL}^{-1}$
2	CMC/PVA/Ag ⁺	0.25	60–110±18	$2 \times 10^{10} \text{ CFU mL}^{-1}$	$1 \times 10^6 \text{ CFU mL}^{-1}$
3	CMC/PVA/Ag ⁰	0.025	40–156 ± 23	$2 \times 10^7 \text{ CFU mL}^{-1}$	$1 \times 10^2 \text{ CFU mL}^{-1}$
4	CMC/PVA/Ag ⁰	0.25	50–130±22	Absent	Absent
5	CMC/PVA/Ag ⁰	2.5	70–180±26	$1 \times 10^6 \text{ CFU mL}^{-1}$	Absent

Note: CFU mL^{-1} is the number of colony-forming units per mL

Table 1 shows that in sample №4, CMC/PVA/Ag⁰ nanofibers 50–130±22 nm in diameter containing 0.25 wt.% Ag NPs completely inhibited the growth of *Staphylococcus epidermidis* and *Candida albicans*; thus, this sample was the most active nanofiber mat. The CMC/PVA nanofiber sample used as a control did not possess antimicrobial activity, and the growth of the strains was observed (Table 1, sample №1). The nanofibers of CMC/PVA/Ag⁺ had a diameter of 60–110±18 nm (Table 1, sample №2), containing 0.25 wt.% Ag NPs showed less antimicrobial activity than did sample №4 against *Staphylococcus epidermidis* and *Candida albicans*. This result may be explained by the fact that the total content of Ag⁺ ions in such nanofibers was completely associated with the

carboxylate anions (COO⁻) of Na-CMC and the limited mobility of Ag⁺ and was inactivated due to connections with functional groups on the surface of the cells [131].

CMC/PVA/Ag⁰ nanofibers with diameters of 40–156±23 nm containing 0.025 wt.% Ag NPs (Table 1, sample № 3) inhibited the growth of *Staphylococcus epidermidis* and *Candida albicans* but were less active than samples № 4 and № 5 because of the low content of Ag NPs [132].

The sample of CMC/PVA/Ag⁰ nanofibers with a diameter of 70–180±26 and containing 2.5 wt.% Ag NPs completely inhibited the *Candida albicans* strain and was less active against *Staphylococcus epidermidis* than sample №4.

This difference may be explained by the formation of agglomerates of Ag NPs on the surface of the nanofibers with increasing concentrations of Ag NPs and by their large surface areas, which limited their ability to penetrate into the cell walls [133].

The relatively high antimicrobial activity of the Ag NPs compared with that of Ag⁺ may be caused by the following: The inability of Ag NPs to form chemical bonds with functional groups on the surface of *Staphylococcus epidermidis* and *Candida albicans* cells and, probably, their ability to penetrate into the nucleus of cells and inhibit cell growth and activity;

- A decrease in the size of the Ag NPs led to an increase in the total surface area and acceleration of their contact with cells [134] and penetration into the cell nuclei of both microbial strains;

- As the nanoparticle size increased and their shape changed from spherical to rod shaped, the total surface area readily decreased, thereby leading to a limited ability of the resulting nanoparticles to enter the cell walls of the *Staphylococcus epidermidis* and *Candida albicans* strains.

9. Future insights and conclusions

In this study, recent findings on the properties and applications of Na-CMC/PVA composites and blends, including drug delivery, food packaging, and medical biomaterials, are reviewed. These novel composites have great potential for the active packaging of food products. Moreover, they show enhanced water solubility, leading to improved bioavailability and dissolution for wound healing. These properties make them promising materials for hydrogel production to deliver drugs and moisture to wound sites.

Na-CMC/PVA composites are gaining great attention for use in biomedical applications because they offer various advantages in different fields. The properties of these materials can be tailored by controlling their structure to enhance their properties for various applications. CMC/PVA hydrogels, as wound dressings, act as barriers to microorganisms while retaining their permeability to oxygen. Thus, these attractive composites can be promising materials for drug delivery and wound dressing applications, and the incorporation of other reinforcements or components might be investigated to improve their properties as well as new production methods. They can also be useful for producing masks to protect against coronavirus. Therefore, further well-designed studies are needed for the development and enhancement of these effective biomaterials.

Using Na-CMC and PVA as raw materials, deionized water as a solvent and silver nitrate as an antibacterial agent based on antibacterial nanofiber mats were prepared by electrospinning. The spinability

of Na-CMC was increased by the addition of PVA and silver nitrate. Solutions of Na-CMC⁻/PVA/Ag⁺ and Na-CMC/PVA/Ag NPs were obtained, and their physicochemical properties were studied. The possibility of nanofiber formation from Na-CMC⁻/Ag⁺/PVA and Na-CMC/PVA/AgNPs solutions by electrospinning has been established.

New dipole–dipole bonds formed between the asymmetric and symmetric carboxymethyl and hydroxyl groups of Na-CMC during interactions with Ag⁺ in the nanofibers of Na-CMC⁻/Ag⁺/PVA and were detected in the 1601–1589.34 cm⁻¹ range of the FTIR spectra.

It was established that the carboxylate groups (COO⁻) within the Na-CMC⁻/PVA/Ag⁺ macromolecules could play the role of “nanoreactors”, where according to the theory of Mott–Gurney, the carboxylic groups of Na-CMC “trap” silver cations and promote the photo stimulated formation of Ag NPs. During the electrospinning of the Na-CMC/PVA solution containing silver cations, the formation of Ag NPs on the surface of the beaded nanofibers was observed due to the restoration of silver cations by electron irradiation.

The formation of Ag NPs in the Na-CMC/PVA nanofibers depended on the duration of photolysis and the Ag⁺ concentration. The ratio of Na-CMC to PVA and the viscosity of the solution determine the size and morphology of the nanofibers.

The morphology and diameter of the nanofibers were studied via AFM and SEM analysis. The results confirmed that from the Na-CMC/PVA/Ag NP solution, nanofibers with diameters ranging from 50–130 nm could be obtained.

UV spectroscopy, DLS and XRD analyses revealed that Ag NPs with sizes ranging from 5–26 nm formed on the nanofiber structure.

A correlation between the sizes of the nanofibers and the content of Ag NPs immobilized within the nanofibers and their biological activity was established. Decreasing the diameter of the nanofibers and the content of Ag NPs enhances their antimicrobial activity.

Nanofiber mats containing stable Ag NPs could be used as bactericidal facemasks for air filtration and for the treatment of burn wounds.

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Conflict of interest: The authors declare that there are no conflicts of interest regarding the publication of this article.

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