Polymer Testing 106 (2022) 107462

Contents lists available at ScienceDirect

Polymer Testing

journal homepage: www.elsevier.com/locate/polytest

# Development and evaluation of novel nanofibers based on mango kernel starch obtained by electrospinning

Jaume Gomez-Caturla [a,*], Juan Ivorra-Martinez [a], Diego Lascano [a], Rafael Balart [a], Daniel García-García [a], Franco Dominici [b], Debora Puglia [b], Luigi Torre [b]

[a] Technological Institute of Materials (ITM), Universitat Politècnica de València (UPV), Plaza Ferrándiz y Carbonell 1, 03801, Alcoy, Spain
[b] Dipartimento di Ingegneria Civile ed Ambientale, University of Perugia, UdR INSTM, Strada di Pentima, 4, 05100, Terni, TR, Italy

ARTICLE INFO

*Keywords:*
Mango kernel starch
PVA
PVP
Electrospinning
Nanofibers

ABSTRACT

This work reports on the development of fibers based on natural mango kernel starch (MKS) with diameters in the nanoscalse by means of the electrospinning technique. MKS was extracted from mango kernels and two synthetic polymers, namely polyvinyl alcohol (PVA) and polyvinylpyrrolidone (PVP), were blended with MKS in order to improve its spinnability, obtaining MKS/PVA and MKS/PVP fibers with a 10 wt% of total polymer concentration. Several electrospinning conditions (voltage and flux) were tested in solutions of MKS concentrations ranging from 0 to 5 wt% for both group of fibers. The morphology of all the fibers was evaluated by field emission scanning electron microscopy (FESEM) and their topography was analysed by means of atomic force microscopy (AFM). MKS/PVA nanofibers were obtained with a diameter range from 0.146 to 0.315 μm, with a "smooth fiber concentration threshold" of 3 wt%, while MKS/PVP, fibers with diameters from 0.080 to 0.339 μm were produced, but 5 wt% MKS concentration fibers were beaded fibers, as a result of an excess in starch concentration. Finally, the roughness of the optimal fibers showed quite a similar trend to that of fiber diameter, presenting roughnessess between 80 and 343 nm.

## 1. Introduction

In the last decade, nanomaterials have attracted great attention, as they have allowed to improve and modernize all sorts of industries, including food, medicine, pharmaceutical, cosmetics, electronics, energies, etc. [1–4]. Among all the nanostructures available in the field of nanomaterials, nanofibers are especially interesting due to their excellent mechanical performance, encapsulation efficiency, high surface area and ease of processability [5]. There are several methods to produce nanofibers, namely rotary spinning, self-assembly, electrospinning or wet spinning, which have been used to obtain fibers in the micro or nano scale [6]. In this context, electrospinning is the most common and effective methodology to fabricate nanofibers due to its simplicity and its ability to control the morphology of the fibers depending on the application they are destined for.

In this sense, the popularity of the electrospinning process has increased in recent years. The electrospinning technique makes use of electrostatic forces to produce electrically charged jets from viscoelastic polymer solutions that upon drying, by the evaporation of the solvent [7,8]. It is a process that allows to produce fibers in the submicron range, this feat would be rather difficult by using standard mechanical fiber-spinning techniques [9,10]. The properties of the electrospun nanofibers such as diameter and shape can be manipulated through the variation of the parameters of the electrospinning process. These fibers have great potential to be used in technological fields such as analytical chemistry, tissue engineering and drug delivery, among others [11–13]. This technique has proved to be especially useful in the fabrication of polymer scaffolds, and it has explored several avenues regarding the use of both synthetic and natural polymers. Particularly, natural polymers are of great interest as a result of their intrinsic properties in terms of biocompatibility, biodegradability, non-toxicity and efficient bioactivity responses towards several animal models [14], which makes them very attractive for biomedicine. These polymers are generally derived from animal and plant sources [15]. Polymers such as starch, xylan or cellulose [16] are plant-derived polymers, while chitin/chitosan, collagen or hyaluronan are animal-derived polymers [17]. Lately, the use of starch

* Corresponding author.
*E-mail addresses:* jaugoca@epsa.upv.es (J. Gomez-Caturla), juaivmar@doctor.upv.es (J. Ivorra-Martinez), dielas@epsa.upv.es (D. Lascano), rbalart@mcm.upv.es (R. Balart), dagarga4@epsa.upv.es (D. García-García), franco.dominici@unipg.it (F. Dominici), debora.puglia@unipg.it (D. Puglia), luigi.torre@unipg.it (L. Torre).

https://doi.org/10.1016/j.polymertesting.2021.107462
Received 7 November 2021; Received in revised form 23 November 2021; Accepted 26 December 2021

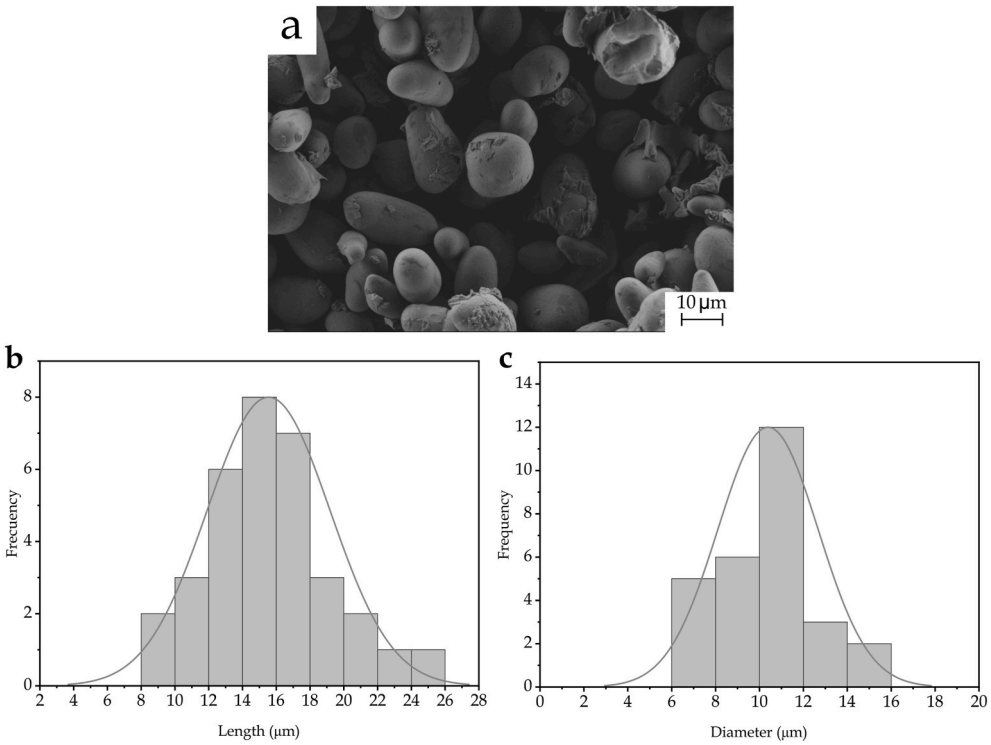


**Fig. 1.** Morphology of the mango kernel starch (MKS): a) FESEM image at 1000× magnification of the mango kernel starch particles; b) Histogram of the length of the MKS particles; c) Histogram of the diameter of the MKS particles.

in electrospinning has increased, especially in combination with synthetic polymers.

Starch is one of the most important natural polymers, being the second largest source of biomass just after cellulose [17]. It is a homo-polysaccharide which is compounded of glucose molecules linked by glycosidic bonds [18], whose main constituents are amylose and amylopectin. This polymer has been widely used in lots of industries thanks to its renewability, biodegradability, biocompatibility and its low cost [19]. It can be obtained from several vegetal sources, such as potato [20], rice [21], maize [22], pea [23] and other several fruits and vegetables like cassava [24].

Among all tropical fruits, mango is one of the most popular [25]. The processing of this fruit generates 25–40% of waste [26], considering peels and kernels, which stands for a great opportunity of reutilizing them under a circular economy concept. Mango kernels from different cultivars have been reported to possess a 74–75% content in polysaccharides, being 60% starch, 9–13% fat content, 6–9% proteins and 2–3% ash [27]. This makes mango kernel the optimal source from which starch can be extracted. Mango kernel starch (MKS) has been reported to present amylose contents between 15 and 30 wt% [28].

In spite of their great advantages, some of the main drawbacks of natural polymers are their poor solubility, poor tensile strength and high surface tension [29,30], which makes starch a poor electrospun fiber forming agent, being its fibers brittle and water-sensitive [17,31]. In order to overcome this problem, there are two main available paths. On the one hand, modifying chemically and physically native starch by reactions like crosslinking or substitutions with reporter molecules and biomolecules, which result in pre-gelatinization [32]. On the other hand, blending starch with linear-chain synthetic polymers is another approach that can improve the spinnability of the solutions [17]. Some of the polymers that have been explored in combination with starch are poly(caprolactone) [33], poly(ethylene oxide) [34], poly(lactic acid) [35], poly(lactide-co-glycolide) [36] or poly(vinyl alcohol) [37].

Polyvinyl alcohol (PVA) is a hydrophilic semi-crystalline synthetic polymer which has great application in the fabrication of fibers for food industry [23,38] and biomedical issues such as tissue engineering or drug delivery due to its chemical and thermal stability, biocompatibility and hydrophilicity [39]. Furthermore, it presents great processability, nontoxicity and water-solubility, providing it with excellent spinnability, which is perfect for enhancing the poor spinnability of natural polymers like starch. Milasius et al. [40] investigated the production of composite fibers made of potato starch and PVA by electrospinning using water and ethanol as solvents. Woranuch et al. [41] studied the synthesis of rice starch/PVA nanofibers by electrospinning in alkaline solution conditions, obtaining very thin fibers of diameters between 36 and 151 nm.

Polyvinylpyrrolidone (PVP) is another synthetic polymer that has been gaining importance over the years in the production of nanofibers. PVP possesses amphiphilicity, thanks to the polarity of its lactam group, making it hydrophilic, and the chemical nature of its non-polar methylene moiety, providing it with lipophilicity [42]. This polymer has found utility in several “biocompatible” applications, such as several drug delivery systems, as a result of its solubility in water and different organic solvents, wide availability, chemical stability, nontoxicity and biocompatibility [43]. PVP has also received great attention from the food, cosmetic and textile industries [44]. Dai et al. [45] blended PVP with emodin, an extract of polygonum cuspidate, a medicinal plant, to make drug carrier nanofibers by electrospinning and use them as a novel drug delivery system.

The aim of this study is to produce totally biocompatible nanofibers with great natural content, based on starch extracted from mango kernel coming from wastes of the mango industry. These fibers will be produces by electrospinning. Interestingly, there has not been any research study regarding the use of mango kernel starch (MKS) for electrospinning of nanofibers. Considering the low spinnability of MKS, two synthetic polymers will be used to produce blend solutions, namely PVA and PVP. On the one hand, different solutions of MKS/PVA,with distilled water as a solvent in order to make them as natural as possible, varying the concentration of starch from 0 to 5 wt%, will be electrospun under several electrospinning conditions. On the other hand, another set of

**Table 1**
Summary of compositions, experiment conditions and evaluation of the electrospun nanofibers preliminary studies according to the composition of the fibers, voltage (V), distance between the injector and the collector (D) and flux (F).

| Sample | Starch (wt.%) | PVA (wt.%) | PVP (wt.%) | Solvent | V (kV) | D (cm) | F (mL/h) | Fiber evaluation |
|---|---|---|---|---|---|---|---|---|
| P1 | 2 | 8 | 0 | Water | 25 | 15 | 0.2 | Smooth fibers |
| P2 | 2 | 8 | 0 | Water | 27 | 15 | 0.15 | Smooth fibers |
| P3 | 2 | 8 | 0 | Water | 30 | 15 | 0.15 | Smooth fibers |
| P4 | 2 | 8 | 0 | Water | 30 | 18 | 0.15 | Poor fibers |
| P5 | 8 | 2 | 0 | Water/ethanol[a] | 20 | 15 | 0.45 | – |
| P6 | 8 | 2 | 0 | Water/ethanol[a] | 20 | 18 | 0.75 | Particles |
| P7 | 8 | 2 | 0 | Water/ethanol[a] | 25 | 15 | 0.35 | Particles |
| P8 | 10 | 0 | 0 | Water/ethanol[a] | 18 | 15 | 0.55 | Particles |
| P9 | 10 | 0 | 0 | Water/ethanol[a] | 20 | 18 | 0.75 | Particles |
| P10 | 10 | 0 | 0 | Water/ethanol[a] | 23 | 13 | 0.45 | – |
| P11 | 0 | 0 | 10 | Water | 20 | 15 | 0.15 | Beaded fibers |
| P12 | 0 | 0 | 10 | Water | 24 | 15 | 0.15 | Beaded fibers |
| P13 | 0 | 0 | 10 | Water | 29 | 15 | 0.15 | Beaded fibers |
| P14 | 2 | 0 | 8 | Water | 30 | 15 | 0.05 | Beaded fibers |

[a] (50%/50%).

**Table 2**
Summary of compositions and experiment conditions of the electrospun nanofibers according to the weight content (wt.%) of mango kernel starch (MKS) and PVA (distilled water as solvent), voltage (V), distance between the injector and the collector (D) and flux (F).

| Sample | Starch (wt.%) | PVA (wt.%) | V (kV) | F (mL/h) |
|---|---|---|---|---|
| 1 | 0 | 10 | 25 | 0.15 |
| 2 | 0 | 10 | 27 | 0.15 |
| 3 | 0 | 10 | 29 | 0.15 |
| 4 | 2 | 8 | 25 | 0.15 |
| 5 | 2 | 8 | 27 | 0.15 |
| 6 | 2 | 8 | 30 | 0.15 |
| 7 | 3 | 7 | 25 | 0.15 |
| 8 | 3 | 7 | 27 | 0.15 |
| 9 | 3 | 7 | 29 | 0.15 |
| 10 | 4 | 6 | 32 | 0.06 |
| 11 | 4 | 6 | 34 | 0.06 |
| 12 | 4 | 6 | 36 | 0.06 |
| 13 | 5 | 5 | 30 | 0.1 |
| 14 | 5 | 5 | 32 | 0.07 |

**Table 3**
Summary of compositions and experiment conditions of the electrospun nanofibers according to the weight content (wt.%) of mango kernel starch (MKS) and PVP (using metoxyethanol as solvent), voltage (V), distance between the injector and the collector (D) and flux (F).

| Sample | Starch (wt.%) | PVP (wt.%) | V (kV) | F (mL/h) |
|---|---|---|---|---|
| 15 | 0 | 10 | 18 | 0.2 |
| 16 | 0 | 10 | 20 | 0.2 |
| 17 | 0 | 10 | 22 | 0.2 |
| 18 | 2 | 8 | 18 | 0.2 |
| 19 | 2 | 8 | 20 | 0.2 |
| 20 | 2 | 8 | 22 | 0.2 |
| 21 | 3 | 7 | 18 | 0.2 |
| 22 | 3 | 7 | 20 | 0.2 |
| 23 | 3 | 7 | 22 | 0.2 |
| 24 | 4 | 6 | 16 | 0.2 |
| 25 | 4 | 6 | 18 | 0.2 |
| 26 | 4 | 6 | 20 | 0.2 |
| 27 | 4 | 6 | 22 | 0.2 |
| 28 | 5 | 5 | 16 | 0.2 |
| 29 | 5 | 5 | 18 | 0.2 |
| 30 | 5 | 5 | 20 | 0.2 |
| 31 | 5 | 5 | 22 | 0.2 |

solutions of MKS/PVP, dissolved in methoxyethanol due to the ineffectivity of distilled water tested in preliminary studies, with concentrations of starch ranging from 0 to 5 wt% will also be electrospun under varying electrospinning conditions. The morphology and diameter of the fibers has been characterized by field emission scanning electron microscopy (FESEM) and atomic force microscopy (AFM). This completely biocompatible nanofibers could prove to be especially useful for drug delivery applications and food packaging [46], with the possibility of reconducting mango wastes into the same industry that produced them (circular economy).

## 2. Materials and methods

### 2.1. Materials

Mangoes from the osteen variety were purchased from the local market in Alcoy, Spain. The fruits were chosen for absence of mechanical damage or any kind of infection.

Polyvinyl alcohol was provided by Sekisui Specialty Chemicals Europe, S.L. (Product Code: Selvol E325 L-0000003310), Polyvinylpyrrolidone (PVP) was also provided by Sigma-Aldrich with an average $M_w$ of 1300000 (Product Code: 1003139835). Finally, 2-Methoxyethanol ReagentPlus was also purchased from Sigma Aldrich (Product Code: 185469).

### 2.2. Methods

#### 2.2.1. Mango kernel starch isolation

Mango kernel starch was extracted following the modified method of Nawab et al. [47]. Mango kernels were first decorticated and cut into small pieces (of about 1 $cm^3$). The kernels were then immersed into a sodium metabisulfite 0.16 wt% solution (kernel: solution weight ratio, 1:2) for 48 h to avoid browning of the kernels. Then the kernels were washed with distilled water using a 70 μm mesh screen until the filtrate reached neutral pH (7). After that, the kernels were ground in distilled water with an homogenizer (Daihan Scientific, model HG-15A with direct controller, Corea) for 5 min at 5000 rpm. The resultant slurry was then screened through a 100 μm mesh sieve followed by a 70 μm mesh sieve and was decanted for 12 h in a refrigerated environment (4 °C) to avoid enzymatic or fermentative processes. The supernatant was discarded and the starch was dried at 45 °C for 2 h, then the brownish layer was eliminated and the white layer was dried at 45 °C for 22 h. Thereafter, the white layer was resuspended in distilled water and centrifuged at 4500 rpm for 10 min, this process was repeated until the precipitate had the typical color of starch. Finally, the extracted starch was dried at 45 °C for 24 h and then pulverized. The average starch extraction yield in relation to the weight of dried mango kernels was 8.12 ± 2.86%.

The morphology of the extracted mango kernel starch was analysed by means of scanning electron microscopy (SEM). Fig. 1 gathers the morphology of the MKS particles at 1000× magnification and a histogram of the length of the particles. MKS presents the typical morphology of starch, in form of smooth, spherical and ellipsoidal granules with

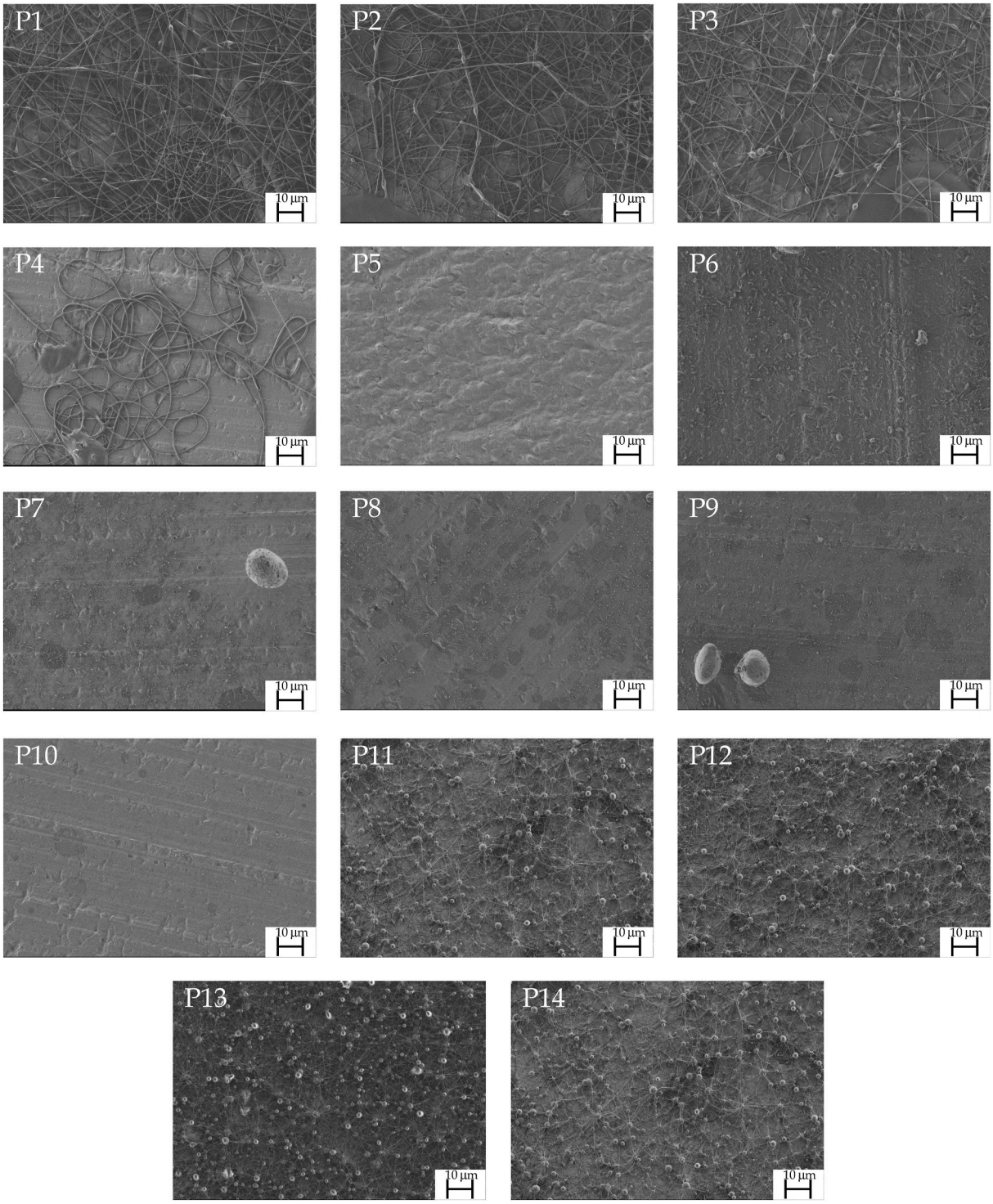


**Fig. 2.** FESEM images at 1000× magnification of all the fibers developed during preliminary studies specified in Table 3.

lengths between 8 and 26 μm (average length: 15 μm) and diameters between 6 and 16 μm (average diameter: 11 μm). The morphology herein observed is very similar to most of the starches observed by Jane et al. [48], who studied the morphology of 54 different starches from several plant materials, namely potato, lotus root, arrow root, loly bulb, ginger, wheat and yucca, among others.

### 2.2.2. Solution preparation method

*2.2.2.1. Starch/PVA fibers.* In the case of starch/PVA fibers, PVA was dissolved in hot distilled water at 95 °C and stirred for about 15 min. Then it was cooled at 65 °C and the starch was added to the solution, stirring it for about 30 more min. Once starch and PVA were correctly dissolved, the solution was cooled to room temperature and ready to use in the electrospinning process.

*2.2.2.2. Starch/PVP fibers.* For the starch/PVP fibers, methoxyethanol was used as the solvent [49]. PVP and starch were dissolved in metoxyethanol under constant stirring for about 40 min according to the established compositions. Then the solution was prepared for the

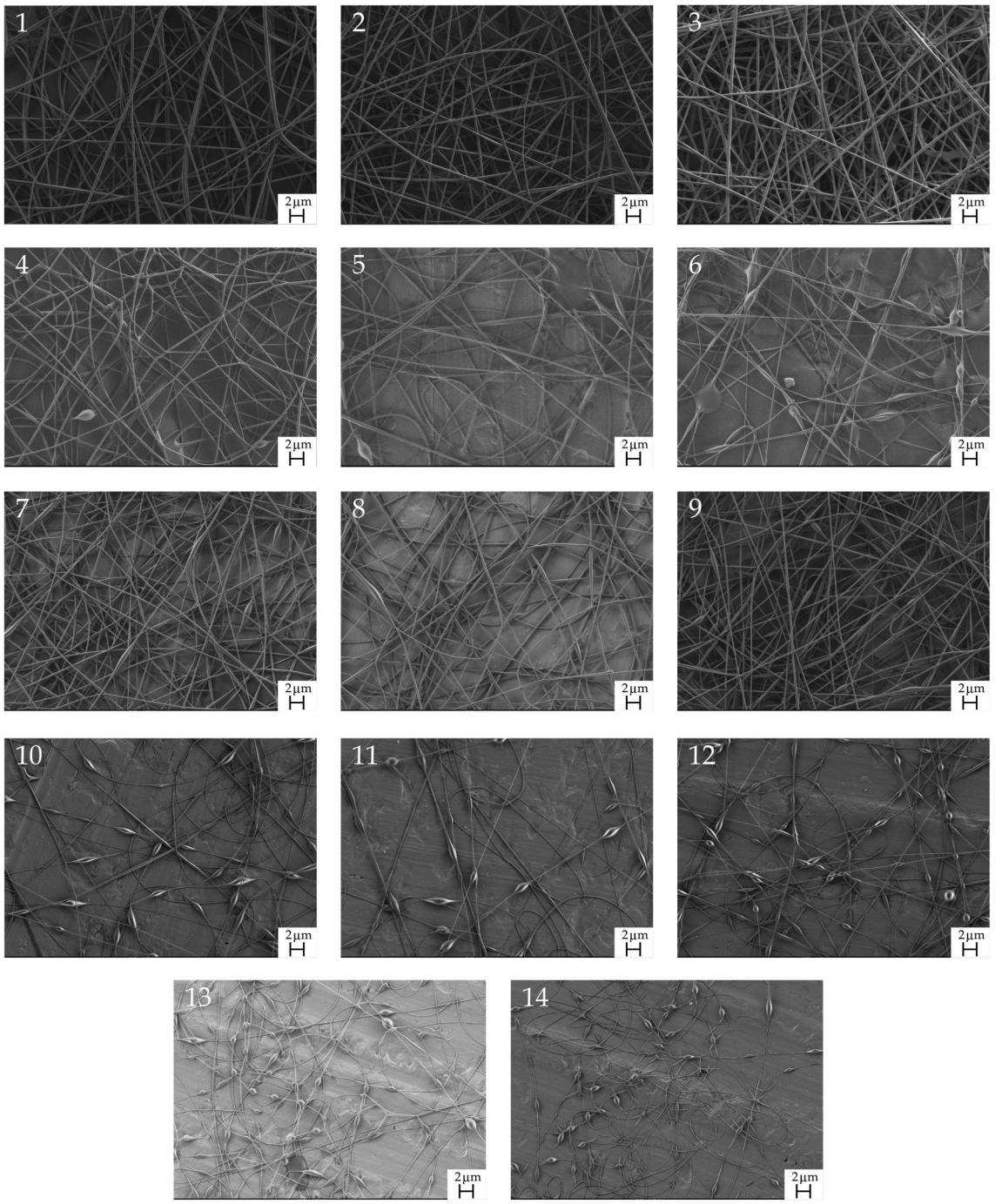


**Fig. 3.** FESEM images at 2500× magnification of all the MKS/PVA fibers specified in Table 3.

electrospinning process.

### 2.2.3. Electrospinning process

The electrospinning process was carried out using a Fluidnatek LE-50 benchtop line with a variable high-voltage 0–35 kV power supply. The system was equipped with a motorized injector able to scan towards a metallic collector (20 × 20 $cm^2$) that allows to obtain an homogeneous electrospun deposition. The corresponding solution (starch/PVA or starch/PVP) was first placed into a 3 mL syringe, connected by polytetrafluoroethylene (PTFE) tubes to a stainless-steel needle of 0.7 mm of diameter. The needle tip was connected to the positive terminal of the power supply, while the metal collector was connected to the negative one. A piece of aluminum foil was placed on the collector and the solution was electrospun for about 5–10 min under a steady flow rate in the range 0.06–0.2 mL/h, depending on the sample, using the motorized injector. The distance between the needle tip and the collector was 15 cm (based on preliminary tests), and the voltage was varied for each sample depending on its properties. Pure PVA and PVP solutions were also electrospun under the same conditions that have been described above. The process was conducted at 25 °C and at 40% relative humidity (RH). All the solutions prepared for electrospinning were fixed to present a 10 wt% composition of polymer, either natural, synthetic or in combination of both of them according to the study carried out by Liu et al. [50].

**Table 4**
Diameter of the electrospun nanofibers made of MKS/PVA solution and their evaluation according to their morphology.

| Sample | Diameter (μm) | Fiber Evaluation |
|---|---|---|
| 1 | 0.315 ± 0.061 | Smooth fibers |
| 2 | 0.276 ± 0.047 | Smooth fibers |
| 3 | 0.280 ± 0.094 | Smooth fibers |
| 4 | 0.297 ± 0.048 | Smooth fibers |
| 5 | 0.233 ± 0.069 | Fair fibers |
| 6 | 0.189 ± 0.042 | Fair fibers (beaded fibers) |
| 7 | 0.250 ± 0.058 | Smooth fibers |
| 8 | 0.228 ± 0.056 | Smooth fibers |
| 9 | 0.191 ± 0.058 | Smooth fibers |
| 10 | 0.187 ± 0.038 | Beaded fibers |
| 11 | 0.180 ± 0.028 | Beaded fibers |
| 12 | 0.146 ± 0.018 | Beaded fibers |
| 13 | 0.163 ± 0.044 | Beaded fibers |
| 14 | 0.147 ± 0.053 | Beaded fibers |

#### 2.2.4. Preliminary studies

Due to the fact that mango kernel starch had never been studied in the electrospinning field, some preliminary studies were carried out before defining the definitive experimental of this work, where different compositions, voltages, distances and solvents were tested. In order to justify the selection of the conditions in Table 2 and Table 3, this preliminary research is presented here. Table 1 gathers all the electrospun nanofibers in this study, while Fig. 2 shows their FESEM morphologies at 1000× magnification.

Samples P1, P2 and P3 showed excellent fiber formation while P4 presented very poor fiber formation, which is ascribed to an excessive distance between the collector and the needle (18 cm). This first samples demonstrate that it is possible to obtain fibers successfully at 2 wt% starch combined with 8 wt% PVA and water as solvent.

On the other hand, P5, P6 and P7 did not gave successful electrospinning results, obtaining particles rather than fibers (electrospraying) [51]. This was ascribed probably to an excessive proportion of starch in the samples (8 wt%), making the blends too fluid to be electrospun. The solvent was changed to a mixture of 50%water/50%ethanol, but it did not make any positive effect. Similarly, samples P8, P9 and P10 were tested in different conditions for 10 wt% starch solutions (without any synthetic polymer), but the obtained results were not evaluable.

Finally, samples of 10 wt% PVP solutions were tested (P11, P12 and P13), in order to know if it would be possible to electrospun MKS/PVP fibers with water as solvent. The morphologies obtained indicate the presence of highly beaded fibers, which are not optimal for fiber applications and are considered "defective" [52]. 2 wt% starch and 8 wt% PVP were tested in sample P14, obtaining the same results as with pure PVP fibers.

From this study it can be concluded that the proportion of starch in the blend must not be excessive (below 8 wt%) to obtain smooth fibers, that 15 cm of D seems to be the optimal parameter in this experimental setup and that the optimal voltage for MKS/PVA fibers in water seems to be between 25 and 30 kV.

#### 2.2.5. Final conditions

Once the preliminary studies were carried out, the final conditions were chosen. Table 2 gathers the compositions and the electrospinning conditions of all the MKS/PVA based nanofibers, while Table 3 gathers all the compositions and electrospinning conditions for the MKS/PVP based nanofibers.

#### 2.2.6. Morphological characterization of nanofibers

The morphology of the electrospun nanofibers was observed by field emission scanning electron microscopy (FESEM) in a ZEISS ULTRA 55 from Oxford Instruments (Abingdon, United Kingdom). The samples were directly placed in the vacuum chamber, as they were electrospun in aluminum foil. An extra high tension (EHT) of 1.5 kV was applied to the electron beam. The average fiber diameter was determined via ImageJ Launcher v 1.41 software using, at least, 20 FESEM images.

The roughness profile of some of the obtained nanofibers was studied using an atomic force and scanning tunnel microscope (Bruker, Multimode 8, Massachusetts, EEUU). The Antimony (n) doped Si cantilever had a tip of 3 μm radius. Surface roughness was evaluated using Nanoscope Analysis software and the arithmetic mean value ($R_a$), which is related to the integral of the absolute value of the roughness profile height over the analysed length approximated by a trapezoidal rule, was calculated.

## 3. Results and discussion

### 3.1. MKS/PVA nanofibers

FESEM images for each sample of electrospun MKS/PVA nanofibers are presented in Fig. 3, while Table 4 gathers the diameter of the fibers in each sample and a short evaluation of the morphology of the fibers. They have been classified as smooth, fair of beaded fibers.

Samples 1, 2 and 3 are made of pure PVA, which exhibited an excellent fiber formation and homogeneity. These results were expected, as PVA has been widely studied in the field of electrospinning and its effectiveness has already been proven [53,54]. The fiber diameter decreased from 0.315 μm in sample 1 to 0.276 μm and 0.280 μm in samples 2 and 3, respectively. This trend is related to an increase in the applied voltage, which results in fibers with smaller diameters due to a higher compression of the fibers exerted by higher electrostatic forces in the tip of the needle when conducting electrospinning [55]. The fiber diameters obtained for these samples are in accordance with the range observed by Koski et al. [54], who studied electrospun fibers of pure PVA of different molecular weights, ranging from 0.25 to 2 μm. The addition of 2 wt% of MKS and 8 wt% PVA leads to the formation of fibers of samples 4, 5 and 6. Sample 4 presented quite good copolymer nanofibers with some droplets, while samples 5 and 6 showed fibers not as homogeneous as sample 4 and with a higher concentration of droplets, which can be attributed to an excess of the applied electric potential, as it was observed by Fong et al. [56] when they studied the formation of beaded fibers in poly(ethylene oxide) solutions. Again, a decrease in fiber diameter is observed from 0.297 μm in sample 4 to 0.233 and 0.189 μm in samples 5 and 6, respectively, which is also attributed to an increase in the applied potential (from 25 to 30 kV). It could be inferred that at this concentration, 25 kV are optimal with regard to fiber morphology. Samples 7, 8 and 9 correspond to the addition of 3 wt% of MKS and 7 wt% of PVA. In all three copolymer samples, smooth and uniform fibers were obtained, again experiencing a decrease in diameter (from 0.250 to 0.191 μm) with the applied voltage (from 25 kV to 29 kV), as it has been aforementioned. A similar experiment was carried out by Sukyte et al. [40], who developed nanofibers based on potato starch (3 wt%) and PVA (7 wt%), with fiber diameters of 0.275 μm approximately. Totally in contrast, the samples containing 4 wt% of starch and 6 wt% of PVA (samples 10, 11 and 12) show a clear detriment in the nanofibers, producing poor beaded fibers in the three cases. This beaded morphology is ascribed to a reduction in the viscosity of the solution [17], which is a direct consequence of the decrease in the PVA content and the increase in starch content. Nonetheless, the fiber diameters have been measured and they follow the same trend as before, decreasing the diameter from 0.187 μm to 0.146 μm for an increase in voltage from 32 to 36 kV. An increase in voltage in relation to the previous samples was needed in order to produce evaluable fibers, as the low viscosity of the solution required a higher electrostatic force to evaporate the solvent. Finally, samples 13 and 14 contained 5 wt% of MKS and 5 wt% of PVA. Similar to what has been observed for the previous samples, these are beaded fibers, as a result of the increase of starch content and decrease of PVA content, thus demonstrating that mango kernel starch is generally detrimental for the electrospinnability of polymer blends, as it has been reported in other studies [57]. In this

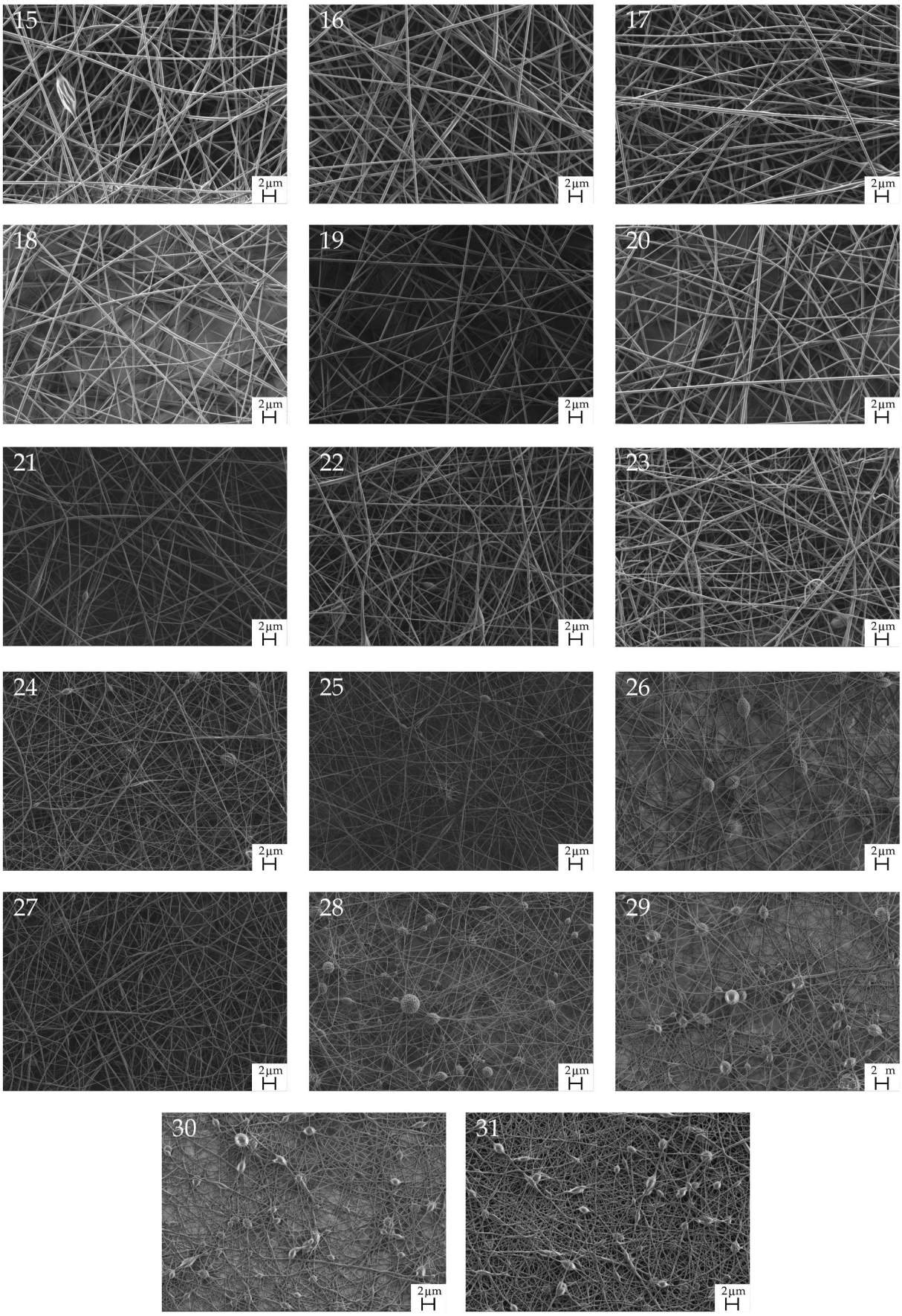


**Fig. 4.** FESEM images at 2500× magnification of all the MKS/PVP fibers specified in Table 5.

**Table 5**
Diameter of the electrospun nanofibers made of MKS/PVP solutions.

| Sample | Diameter (μm) | Fiber evaluation |
|---|---|---|
| 15 | 0.339 ± 0.050 | Smooth fibers |
| 16 | 0.282 ± 0.076 | Smooth fibers |
| 17 | 0.241 ± 0.069 | Smooth fibers |
| 18 | 0.316 ± 0.073 | Smooth fibers |
| 19 | 0.299 ± 0.067 | Smooth fibers |
| 20 | 0.260 ± 0.069 | Smooth fibers |
| 21 | 0.243 ± 0.104 | Smooth fibers |
| 22 | 0.216 ± 0.057 | Smooth fibers |
| 23 | 0.174 ± 0.039 | Smooth fibers |
| 24 | 0.079 ± 0.036 | Smooth fibers |
| 25 | 0.110 ± 0.039 | Smooth fibers (beaded fibers) |
| 26 | 0.105 ± 0.052 | Fair fibers (beaded fibers) |
| 27 | 0.099 ± 0.031 | Smooth fibers |
| 28 | 0.071 ± 0.021 | Fair fibers (beaded fibers) |
| 29 | 0.121 ± 0.049 | Fair fibers (beaded fibers) |
| 30 | 0.073 ± 0.037 | Fair fibers (beaded fibers |
| 31 | 0.080 ± 0.040 | Fair fibers (beaded fibers) |

case, the fiber diameters are practically the lowest of all MKS/PVA nanofibers, being 0.163 and 0.147 μm, respectively. Interestingly, fiber diameter seems to decrease as the content of starch increases, wich could be probably ascribed to the lower viscosity of the solutions with higher content in starch, as they flow more easily from the injector to the collector. A similar phenomenon was observed by Kong et al. [58].

From these results it can be concluded that 3 wt% MKS/7 wt% PVA is the optimal combination for obtaining smooth nanofibers with true applicability in fields such as biomedicine or food industry [59]. These fibers are quite similar to those of pure PVA, whose effectivity has been widely proven, and with lesser diameters, which could prove to be really useful in applications that require great interfacial area. Moreover, 3 wt % of starch seems to be the concentration threshold in which good nanofibers are obtained in combination with PVA, as at higher concentrations of starch, the fibers turn to beaded fibers with too many droplets in their morphology.

All in all, successful MKS/PVA starch nanofibers have been obtained, with a relatively wide range in electric voltage that allows to modify their diameter according to the function they will be destined for, maintaining the homogeneity of the fibers.

### 3.2. MKS/PVP nanofibers

Fig. 4 shows the FESEM morphologies of all the electrospun MKS/PVP fibers, while Table 5 gathers the average diameter of all the samples and their corresponding evaluation according to the quality of the obtained fibers. Samples 15, 16 and 17 are related to neat PVP nanofibers using methoxyethanol as solvent at different voltages (18, 20 and 22 kV, respectively). In spite of having some droplets, quite homogeneous and smooth fibers have been obtained with diameters ranging from 0.339 to 0.241 μm, respectively. Similarly to what was observed in the case of PVA fibers, the diameter of the fibers diminishes as the voltage increases, due to an electrostatic compression phenomenon in the tip of the needle [60], which makes the fiber thinner with the increase in the electrostatic potential. Vongsetskul et al. [61] also observed this effect in pure PVP fibers when varying the applied electrical voltage. The diameters observed are similar to the ones obtained by Holopainen et al. [49], who used 10 wt% PVP solutions to obtain nanofibers with hydroxyapatite. Next, samples 18, 19 and 20 correspond to MKS/PVP solutions with 2 wt% of MKS and 8 wt% of PVP. These prove to be homogeneous fibers without the presence of droplets, with diameters ranging from 0.316 to 0.260 μm, for a change in voltage from 18 to 22 kV, respectively. Interestingly, the morphology is quite similar to the morphology of pure PVP fibers, which demonstrates in this case that it is possible to successfully obtain functional starch/PVP nanofibers at this composition. Considering that there is hardly any information regarding the combination of starch with PVP to obtain fibers by electrospinning, it is an intriguing finding. In a similar manner, samples 21, 22 and 23 show nanofibers with 3 wt% of MKS and 7 wt% of PVP. The diameter also descends with the increase of potential in this case from 0.243 to 0.174 μm. There seems to be some concentration of droplets in sample 22, but it does not affect the homogeneity of the fibers, which are quite good in general terms. The observed diameters are lower than those of the higher concentration of PVP, which again seems to indicate that the higher the concentration of starch, the lower the diameter of the fibers. This fact is directly related to a decrease in the viscosity of the solution. The following samples, 24, 25, 26 and 27 correspond to fibers with 4 wt % of MKS and 6 wt% of PVP, with applied potentials of 16, 18, 20 and 22 kV, respectively. In this case, 16 kV was added because the fibers did not seem to be homogeneous and due to the change in starch concentration, another potential was considered. Samples 24, 25 and 26 presented some droplets, especially sample 26. On the other hand, sample 27 presented quite homogeneous fibers. This seems to point out to the fact that when the concentration of starch increases in the solution, a higher voltage is needed to avoid the formation of beaded fibers, which could be ascribed to the need of a higher evaporation rate of the solvent in the tip of the needle [59]. Diameters ranging from 0.079 to 0.110 μm have been obtained, which turn to be considerably smaller than the ones obtained for the previous solutions with lower concentration of starch. This reduction in diameter could be related to a marked decrease in the viscosity of the solution when going from 3 up to 4 wt% of MKS, at the same time that PVP is decreased from 7 to 6 wt%. Finally, samples 28, 29, 30 and 31 stand for solutions with 5 wt% and 5 wt% of PVP. As it was expected, beaded fibers were obtained, being the worst results for MKS/PVP nanofibers, and again demonstrating that starch is not optimal for electrospinning at high concentrations, as it has been also reported by Hemamalini et al. [15]. Diameters of 0.071, 0.121, 0.073 and 0.08 μm were obtained, which are the smallest diameters obtained for nanofibers in this study, although these nanofibers are not optimal for actual applications, due to their high concentration of beads. Thus, 4 wt% could be considered the concentration threshold from which total homogeneous nanofibers cannot be developed and beaded fibers appear.

According to these results, smooth MKS/PVP nanofibers have been successfully produced at different concentrations of starch and PVP, which is quite interesting given the fact that there are no publications of starch/PVP fibers obtained by electrospinning technique, especially referring to MKS. Moreover, smooth fibers in a wide range of diameters have been reported, even reaching less than 100 nm, which could be of special use in applications were high interfacial area may be needed. Particularly, in terms of starch fibers, fibers from 2 wt% and 3 wt% starch have shown very positive results, as well as sample 27, corresponding to a 4 wt% starch solution electrospun at 22 kV, which presents very thin smooth fibers.

### 3.3. AFM analysis of the MKS/PVA nanofibers and MKS/PVP nanofibers

After evaluating all fibers by field emission scanning electron microscopy (FESEM), the optimal solutions have been selected for a deeper analysis by means of atomic force microscopy (AFM). Two samples from the 3 wt% starch 7 wt% PVA solution have been selected (samples 7 and 9, electrospun at 25 and 29 kV, respectively). On the other hand, sample 20, electrospun at 22 kV, has been chosen from the 2 wt% starch 8 wt% PVP solution, sample 21, at 18 kV, from the 3 wt% starch 7 wt% PVP solution and sample 27, at 22 kV, from the 4 wt% starch 6 wt% PVP solution. Fig. 5 shows FESEM images of these samples at 5000× magnification as well as AFM images with a scan size of 4 μm. Additionally, Fig. 6 gathers the fiber diameter distribution of the selected nanofibers, where it can be seen how the average diameter coincides with the diameter showed in Tables 4 and 5

AFM is a very useful technique to study the topography of the fibers. As it can be observed, the AFM images are in good agreement with the FESEM images. First, sample 7 and 9, which correspond to MKS/PVA

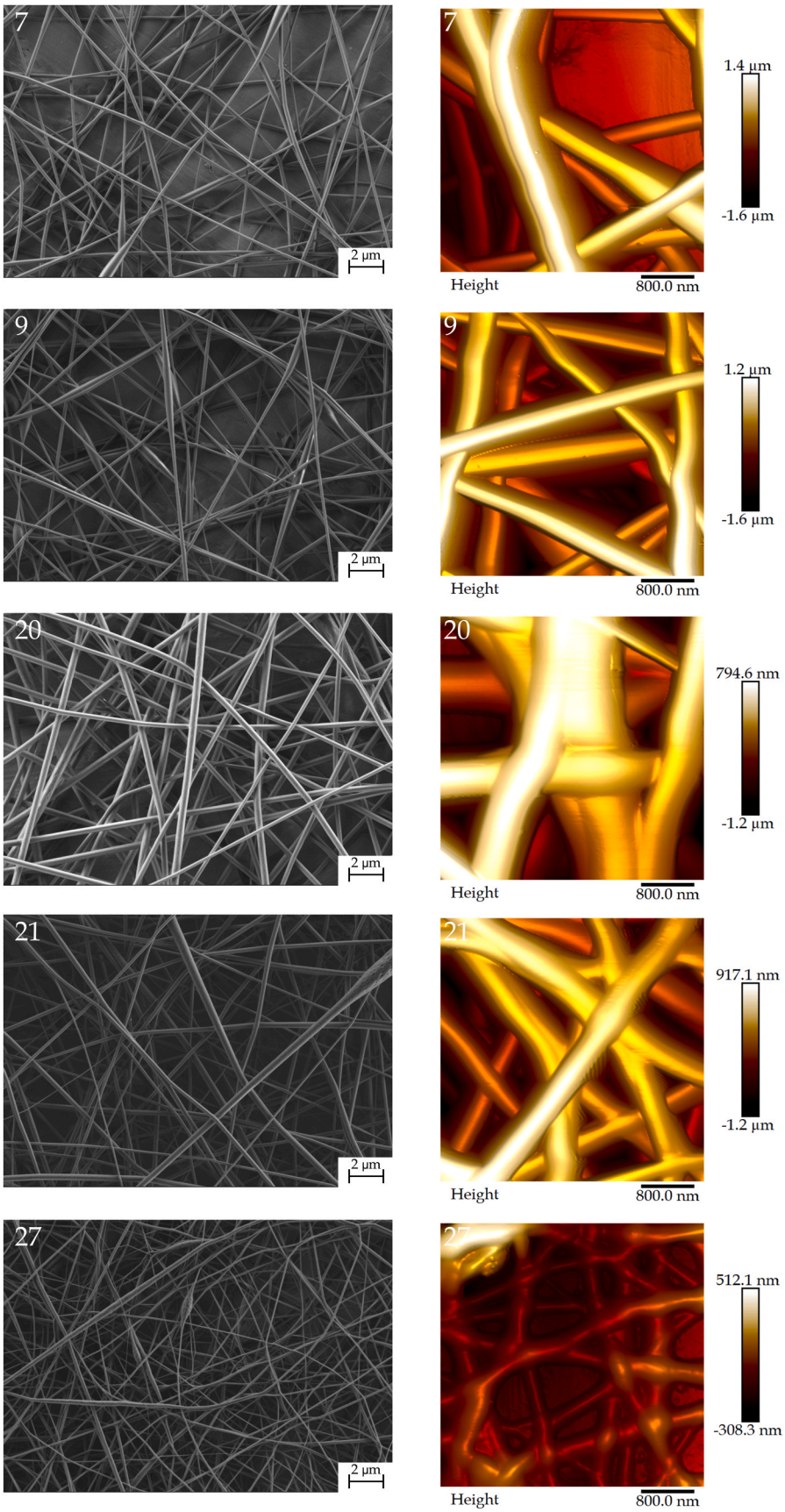


**Fig. 5.** FESEM images at 5000× magnification and AFM images at a scan size of 4 μm of the optimal electrospun MKS/PVA and MKS/PVP obtained fibers (samples 7, 9, 20, 21 and 27).

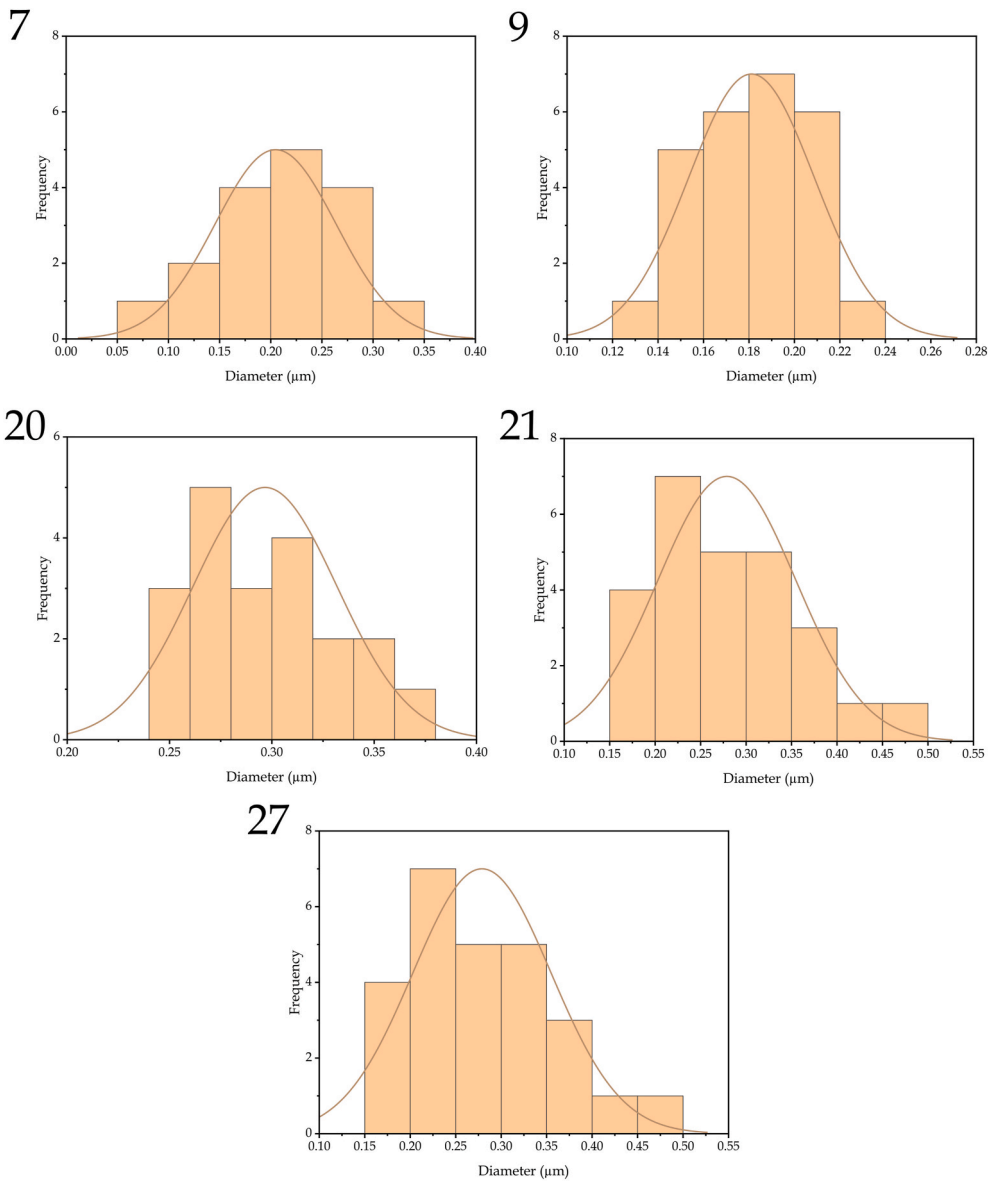


**Fig. 6.** Fiber diameter distributions of the optimal electrospun MKS/PVA and MKS/PVP fibers (samples 7, 9, 20, 21 and 27).

**Table 6**
Roughness measurements of the MKS/PVA and MKS/PVP electrospun nanofibers.

| Sample | Roughness $R_a$ (nm) |
|---|---|
| 7 | 339 |
| 9 | 343 |
| 20 | 286 |
| 21 | 260 |
| 27 | 80 |

fibers, seem to maintain the same fiber diameter relation as in the previous section, although sample 9 presents a smaller diameter as a result of having been electrospun at 29 kV, while sample 7 was electrospun at 25 kV. Similarly, samples 20, 21 and 27 also seem to match the previously analysed results. Those samples presented fiber diameters of 260, 243 and 99 nm, respectively. It can be seen how AFM images show that sample 27 has a considerably smaller diameter than the rest of the samples, while sample 21 has also a clearly smaller diameter than fibers in sample 20. However, the diameter observed in AFM images seems to be larger than in FESEM, this is because the scan size in AFM was quite limited due to cantilever and tip sensibility. Thus, very few fibers could be observed by AFM at the same time. Nonetheless, the diameter relation is clearly observed. Additionally, the scale in the right of the AFM images is representative for the height of the image, obtained from the oscilation of the tip of the microscope. Observing this scales it can be observed how sample 27 presents a less profound scale (from −300 nm to 500 nm approximately) in terms of depth, which is another indicative of the smaller diameter of the MKS/PVP fibers at 4 wt% of MKS and 6 wt % of PVP, as this scale is quite lower than that of the rest of the samples. AFM allowed to measure the roughness of the samples, measured as $R_a$, which is the arithmetic average of the absolute values of the roughness profile ordinates [62]. It can be observed how the roughness of MKS/PVA fibers is quite higher than that of MKS/PVP fibers, which could be ascribed to the difference in fiber diameter previously commented, as it was also reported by Adeli et al. [63] in a study about PVA/chitosan/starch electrospun nanofibers by AFM. Particularly in the case of sample 27, showing a roughness of 80 nm, while the rest of the samples exhibited roughnesses in the range 260–343 nm (see Table 6).

The results presented here suggest that MKS/PVP nanofibers present lower roughnesses than MKS/PVA nanofibers, and that increasing the

concentration of MKS in MKS/PVP fibers up to 4 wt% allows to obtain especially thin fibers. Interestingly, this fact makes the obtained fibers suitable for applications in tissue engineering for cell attachment and cell growth. The roughness range aforementioned proves to be favourable for osteoblast proliferation [64] and endothelial [65] and neuronal cells growth and proliferation [66] according to several studies.

## 4. Discussion and conclusions

In this study, smooth ultrathin fibers in the nano scale based on starch extracted from mango kernel and PVA and PVP were successfully obtained by means of the electrospinning technique and evaluated by FESEM and AFM. Several experimental conditions were tested for MKS/PVA and MKS/PVP fibers. MKS/PVA nanofibers' diameter ranged from 0.146 to 0.315 μm, while MKS/PVP nanofibers's diameter ranged from 0.08 to 0.339 μm. It was observed how the diameter of the electrospun nanofibers decreased with an increase in the applied voltage, as a result of higher compression electrostatic forces in the tip of the needle. Additionally, it was demonstrated that in general, starch is detrimental for the electrospinning technique, establishing a concentration threshold for which smooth fibers are obtained in each blend. In the case of MKS/PVA fibers, starch concentrations superior to 3 wt% provoked the appearance of droplets in their morphology, leading to beaded fibers. On the other hand, MKS/PVP fibers with starch concentrations higher than 3 wt% also caused the appearance of beaded fibers, although there was one sample at 4 wt% of starch that exhibited smooth and homogeneous fibers at a voltage of 22 kV. MKS/PVP nanofibers proved to be considerably thinner than MKS/PVA nanofibers when 4 wt % or superior concentrations of starch were added. This fact could prove to be quite interesting from a scientific point of view for certain applications, were higher interfacial area is required. AFM analysis allowed to determine the topologhy of the optimal nanofiber samples, which were 3 wt% starch solutions electrospun at 25 and 29 kV, respectively, for MKS/PVA blends and 2, 3 and 4 wt% starch concentration solutions electrospun at 22, 18 and 22 kV, respectively. The roughness of the samples followed a similar trend compared to fiber diameter, being the MKS/PVP sample with 4 wt% of starch the one with the lowest roughness ($R_a$ = 80 nm). AFM images confirmed the results observed by FESEM.

All in all, the results obtained in this work proved that it is possible to obtain completely natural, biocompatible and biodegradable electrospun mango kernel starch nanofibers in combination with PVA and PVP with great potential in applications related to food industry or the biomedical field, such as tissue engineering. Moreover, the diameter of the fibers can be controlled within a wide range varying the conditions of the electrospinning setup, thus allowing to adjust the morphology of the fibers depending on the application they will be destined for. The high natural content of the fibers presented here makes them environmentally friendly and quite cost effective, as starch is extracted from mango, water is used as a solvent and electrospinning is a relatively simple technique. Considering that mango kernel starch has never been previously tested in electrospinning, and that PVP has not been used in combination with starch in this technique, this work opens a whole new path towards the development of novel mango kernel starch fibers with other synthetic polymers or even with other natural polymers. The next steps of the investigation could be centered in optimizing the electrospinning conditions of MKS/PVA and MKS/PVP nanofibers and giving them an actual application, such as tissue regeneration or drug delivery systems for example, given their biocompatibility.

## Author contributions

**Jaume Gomez-Caturla**: Investigation, Writing – original draft, Writing – review & editing; **Juan Ivorra-Martinez**: software, methodology; **Diego Lascano**: visualization, formal analysis, writing – original draft; **Rafael Balart**: supervision, writing – review & editing, validation; **Daniel García-García**: conceptualization, data curation, supervision; **Franco Dominici**: investigation, visualization, supervision; **Debora Puglia**: supervision, software, conceptualization; **Luigi Torre**: project administration, resources, funding acquisition.

## Funding

This research is a part of the grant PID2020-116496RB-C22 funded by MCIN/AEI/10.13039/501100011033.

## Data availability statement

The raw/processed data required to reproduce these findings cannot be shared at this time due to technical or time limitations.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Acknowledgements

Authors also thank Generalitat Valenciana-GVA, grant number AICO/2021/025 for supporting this work. J. Gomez-Caturla wants to thank Generalitat Valenciana-GVA, for his FPI grant (ACIF/2021/185) and grant FPU20/01732 funded by MCIN/AEI/10.13039/501100011033 and by ESF Investing in your future. J. Ivorra-Martinez wants to thank FPU19/01759 grant funded by MCIN/AEI/10.13039/501100011033 and by ESF Investing in your future. Microscopy Services at UPV are also acknowledged by their help in collecting and analyzing images.

## References

[1] V. Thavasi, G. Singh, S. Ramakrishna, Electrospun nanofibers in energy and environmental applications, Energy Environ. Sci. 1 (2008) 205–221.
[2] W. Liu, S. Thomopoulos, Y. Xia, Electrospun nanofibers for regenerative medicine, Adv. Healthc. Mater. 1 (2012) 10–25.
[3] A. Chinnappan, C. Baskar, S. Baskar, G. Ratheesh, S. Ramakrishna, An overview of electrospun nanofibers and their application in energy storage, sensors and wearable/flexible electronics, J. Mater. Chem. C 5 (2017) 12657–12673.
[4] M.H.A. Zanin, N.N. Cerize, A.M. de Oliveira, Production of nanofibers by electrospinning technology: overview and application in cosmetics, Nanocosmet. Nanomed. (2011) 311–332.
[5] A. Rezaei, A. Nasirpour, M. Fathi, Application of cellulosic nanofibers in food science using electrospinning and its potential risk, Compr. Rev. Food Sci. Food Saf. 14 (2015) 269–284.
[6] F. Sharifi, A.C. Sooriyarachchi, H. Altural, R. Montazami, M.N. Rylander, N. Hashemi, Fiber based approaches as medicine delivery systems, ACS Biomater. Sci. Eng. 2 (2016) 1411–1431.
[7] M. Busolo, S. Torres-Giner, C. Prieto, J.M. Lagaron, Electrospraying assisted by pressurized gas as an innovative high-throughput process for the microencapsulation and stabilization of docosahexaenoic acid-enriched fish oil in zein prolamine, Innovat. Food Sci. Emerg. Technol. 51 (2019) 12–19.
[8] L. Quiles-Carrillo, N. Montanes, J.M. Lagaron, R. Balart, S. Torres-Giner, Bioactive multilayer polylactide films with controlled release capacity of gallic acid accomplished by incorporating electrospun nanostructured coatings and interlayers, Appl. Sci. 9 (2019) 533.
[9] T.J. Sill, H.A. Von Recum, Electrospinning: applications in drug delivery and tissue engineering, Biomaterials 29 (2008) 1989–2006.
[10] H.M. Ibrahim, A. Klingner, A review on electrospun polymeric nanofibers: production parameters and potential applications, Polym. Test. 90 (2020) 106647.
[11] N. Bhardwaj, S.C. Kundu, Electrospinning: a fascinating fiber fabrication technique, Biotechnol. Adv. 28 (2010) 325–347.
[12] M. Hajiabbas, I. Alemzadeh, M. Vossoughi, A porous hydrogel-electrospun composite scaffold made of oxidized alginate/gelatin/silk fibroin for tissue engineering application, Carbohydr. Polym. 245 (2020) 116465.
[13] S. Cesur, S. Ulag, L. Ozak, A. Gumussoy, S. Arslan, B.K. Yilmaz, N. Ekren, M. Agirbasli, O. Gunduz, Production and characterization of elastomeric cardiac tissue-like patches for Myocardial Tissue Engineering, Polym. Test. 90 (2020) 106613.
[14] H.A. Pawar, S.R. Kamat, P.D. Choudhary, An overview of natural polysaccharides as biological macromolecules: their chemical modifications and pharmaceutical applications, Biol. Med. 7 (2015) 1.

[15] T. Hemamalini, V.R.G. Dev, Comprehensive review on electrospinning of starch polymer for biomedical applications, Int. J. Biol. Macromol. 106 (2018) 712–718.
[16] S.R.D. Petroudy, S.A. Kahagh, E. Vatankhah, Environmentally friendly superabsorbent fibers based on electrospun cellulose nanofibers extracted from wheat straw, Carbohydr. Polym. 251 (2021) 117087.
[17] G. Liu, Z. Gu, Y. Hong, L. Cheng, C. Li, Electrospun starch nanofibers: recent advances, challenges, and strategies for potential pharmaceutical applications, J. Contr. Release 252 (2017) 95–107.
[18] F.G. Torres, S. Commeaux, O.P. Troncoso, Starch-based biomaterials for wound-dressing applications, Starch-Stärke 65 (2013) 543–551.
[19] S. Doi, J.H. Clark, D.J. Macquarrie, K. Milkowski, New materials based on renewable resources: chemically modified expanded corn starches as catalysts for liquid phase organic reactions, Chem. Commun. (2002) 2632–2633.
[20] R. Hoover, D. Hadziyev, Characterization of potato starch and its monoglyceride complexes, Starch-Stärke 33 (1981) 290–300.
[21] V.R. Williams, W.-T. Wu, H.Y. Tsai, H.G. Bates, Rice starch, varietal differences in amylose content of rice starch, J. Agric. Food Chem. 6 (1958) 47–48.
[22] R. Tester, S. Debon, M. Sommerville, Annealing of maize starch, Carbohydr. Polym. 42 (2000) 287–299.
[23] T. Min, X. Sun, L. Zhou, H. Du, Z. Zhu, Y.J.C.P. Wen, Electrospun pullulan/PVA nanofibers integrated with thymol-loaded porphyrin metal− organic framework for antibacterial food packaging 270 (2021) 118391.
[24] E. de Morais Teixeira, A.L. Da Roz, A.J.F. de Carvalho, A.A. da Silva Curvelo, Preparation and Characterisation of Thermoplastic Starches from Cassava Starch, Cassava Root and Cassava Bagasse, Macromolecular symposia, Wiley Online Library, 2005, pp. 266–275.
[25] H. Makroo, P.K. Prabhakar, N. Rastogi, B. Srivastava, Characterization of mango puree based on total soluble solids and acid content: effect on physico-chemical, rheological, thermal and ohmic heating behavior, LWT (Lebensm.-Wiss. & Technol.) 103 (2019) 316–324.
[26] J. Banerjee, R. Singh, R. Vijayaraghavan, D. MacFarlane, A.F. Patti, A. Arora, A hydrocolloid based biorefinery approach to the valorisation of mango peel waste, Food Hydrocolloids 77 (2018) 142–151.
[27] J.K. Mutua, S. Imathiu, W. Owino, Evaluation of the proximate composition, antioxidant potential, and antimicrobial activity of mango seed kernel extracts, Food Sci. Nutr. 5 (2017) 349–357.
[28] A. Nawab, F. Alam, A. Hasnain, Mango kernel starch as a novel edible coating for enhancing shelf-life of tomato (Solanum lycopersicum) fruit, Int. J. Biol. Macromol. 103 (2017) 581–586.
[29] R. Sridhar, R. Lakshminarayanan, K. Madhaiyan, V.A. Barathi, K.H.C. Lim, S. Ramakrishna, Electrosprayed nanoparticles and electrospun nanofibers based on natural materials: applications in tissue regeneration, drug delivery and pharmaceuticals, Chem. Soc. Rev. 44 (2015) 790–814.
[30] W. Li, Z. Zhang, L. Wu, Z. Zhu, Z. Xu, Improving the adhesion-to-fibers and film properties of corn starch by starch sulfo-itaconation for a better application in warp sizing, Polym. Test. 98 (2021) 107194.
[31] M.A. Asl, S. Karbasi, S. Beigi-Boroujeni, S.Z. Benisi, M. Saeed, Evaluation of the effects of starch on polyhydroxybutyrate electrospun scaffolds for bone tissue engineering applications, Int. J. Biol. Macromol. 191 (2021) 500–513.
[32] Y. Hong, G. Liu, Z. Gu, Recent advances of starch-based excipients used in extended-release tablets: a review, Drug Deliv. 23 (2016) 12–20.
[33] H. Jukola, L. Nikkola, M. Gomes, R. Reis, N. Ashammakhi, Electrospun starch-polycaprolactone nanofiber-based constructs for tissue engineering, in: AIP Conference Proceedings, American Institute of Physics, 2008, pp. 971–974.
[34] S. Mahalingam, G. Ren, M. Edirisinghe, Rheology and pressurised gyration of starch and starch-loaded poly (ethylene oxide), Carbohydr. Polym. 114 (2014) 279–287.
[35] J. Sunthornvarabhas, P. Chatakanonda, K. Piyachomkwan, K. Sriroth, Electrospun polylactic acid and cassava starch fiber by conjugated solvent technique, Mater. Lett. 65 (2011) 985–987.
[36] B.D. Ulery, L.S. Nair, C.T. Laurencin, Biomedical applications of biodegradable polymers, J. Polym. Sci. B Polym. Phys. 49 (2011) 832–864.
[37] P. Jaiturong, B. Sirithunyalug, S. Eitsayeam, C. Asawahame, P. Tipduangta, J. Sirithunyalug, Preparation of glutinous rice starch/polyvinyl alcohol copolymer electrospun fibers for using as a drug delivery carrier, Asian J. Pharm. Sci. 13 (2018) 239–247.
[38] M. Arrieta, C.L. de Dicastillo, L. Garrido, K. Roa, M.J. Galotto, Electrospun PVA fibers loaded with antioxidant fillers extracted from Durvillaea Antarctica algae and their effect on plasticized PLA bionanocomposites, Eur. Polym. J. 103 (2018) 145–157.
[39] E.-R. Kenawy, F.I. Abdel-Hay, M.H. El-Newehy, G.E. Wnek, Controlled release of ketoprofen from electrospun poly (vinyl alcohol) nanofibers, Mater. Sci. Eng., A 459 (2007) 390–396.
[40] J. Šukytė, E. Adomavičiūtė, R. Milašius, Investigation of the Possibility of Forming Nanofibres with Potato Starch, Fibres & Textiles in Eastern Europe, 2010.
[41] S. Woranuch, A. Pangon, K. Puagsuntia, N. Subjalearndee, V. Intasanta, Rice flour-based nanostructures via a water-based system: transformation from powder to electrospun nanofibers under hydrogen-bonding induced viscosity, crystallinity and improved mechanical property, RSC Adv. 7 (2017) 19960–19966.
[42] K.M. Koczkur, S. Mourdikoudis, L. Polavarapu, S.E. Skrabalak, Polyvinylpyrrolidone (PVP) in nanoparticle synthesis, Dalton Trans. 44 (2015) 17883–17905.
[43] V. Bühler, Excipients for Pharmaceuticals-Povidone, Crospovidone and Copovidone, Springer, Berlin Heidelberg New York, 2005.
[44] M. Teodorescu, M. Bercea, Poly (vinylpyrrolidone)–a versatile polymer for biomedical and beyond medical applications, Polym. Plast. Technol. Eng. 54 (2015) 923–943.
[45] X.-Y. Dai, W. Nie, Y.-C. Wang, Y. Shen, Y. Li, S.-J. Gan, Electrospun emodin polyvinylpyrrolidone blended nanofibrous membrane: a novel medicated biomaterial for drug delivery and accelerated wound healing, J. Mater. Sci. Mater. Med. 23 (2012) 2709–2716.
[46] C.P. Vidal, E. Velásquez, M.J. Galotto, C.L. de Dicastillo, Antimicrobial food packaging system based on ethyl lauroyl arginate-loaded core/shell electrospun structures by using hydrophilic and hydrophobic polymers, Polym. Test. 93 (2021) 106937.
[47] A. Nawab, F. Alam, M.A. Haq, A. Hasnain, Biodegradable film from mango kernel starch: effect of plasticizers on physical, barrier, and mechanical properties, Starch-Stärke 68 (2016) 919–928.
[48] J.L. Jane, T. Kasemsuwan, S. Leas, H. Zobel, J.F. Robyt, Anthology of starch granule morphology by scanning electron microscopy, Starch-Stärke 46 (1994) 121–129.
[49] J. Holopainen, E. Santala, M. Heikkilä, M. Ritala, Electrospinning of calcium carbonate fibers and their conversion to nanocrystalline hydroxyapatite, Mater. Sci. Eng. C 45 (2014) 469–476.
[50] Z. Liu, J.-H. He, Polyvinyl alcohol/starch composite nanofibers by bubble electrospinning, Therm. Sci. 18 (2014) 1473–1475.
[51] A. Jaworek, A.T. Sobczyk, Electrospraying route to nanotechnology: an overview, J. Electrost. 66 (2008) 197–219.
[52] W. Zuo, M. Zhu, W. Yang, H. Yu, Y. Chen, Y. Zhang, Experimental study on relationship between jet instability and formation of beaded fibers during electrospinning, Polym. Eng. Sci. 45 (2005) 704–709.
[53] J. Tao, S. Shivkumar, Molecular weight dependent structural regimes during the electrospinning of PVA, Mater. Lett. 61 (2007) 2325–2328.
[54] A. Koski, K. Yim, S. Shivkumar, Effect of molecular weight on fibrous PVA produced by electrospinning, Mater. Lett. 58 (2004) 493–497.
[55] S. Huan, G. Liu, G. Han, W. Cheng, Z. Fu, Q. Wu, Q. Wang, Effect of experimental parameters on morphological, mechanical and hydrophobic properties of electrospun polystyrene fibers, Materials 8 (2015) 2718–2734.
[56] H. Fong, I. Chun, D.H. Reneker, Beaded nanofibers formed during electrospinning, Polymer 40 (1999) 4585–4592.
[57] L. Kong, G.R. Ziegler, Fabrication of pure starch fibers by electrospinning, Food Hydrocolloids 36 (2014) 20–25.
[58] L. Kong, G.R. Ziegler, Quantitative relationship between electrospinning parameters and starch fiber diameter, Carbohydr. Polym. 92 (2013) 1416–1422.
[59] R. Ashraf, H.S. Sofi, A. Malik, M.A. Beigh, R. Hamid, F.A. Sheikh, Recent trends in the fabrication of starch nanofibers: electrospinning and non-electrospinning routes and their applications in biotechnology, Appl. Biochem. Biotechnol. 187 (2019) 47–74.
[60] V. Beachley, X. Wen, Effect of electrospinning parameters on the nanofiber diameter and length, Mater. Sci. Eng. C 29 (2009) 663–668.
[61] T. Vongsetskul, T. Chantarodsakun, P. Wongsomboon, R. Rangkupan, P. Tangboriboonrat, Effect of solvent and processing parameters on electrospun polyvinylpyrrolidone ultra-fine fibers, Chiang Mai J. Sci. 42 (2015) 436–442.
[62] S. Haq, R. Srivastava, Measuring the influence of materials composition on nano scale roughness for wood plastic composites by AFM, Measurement 91 (2016) 541–547.
[63] H. Adeli, M.T. Khorasani, M. Parvazinia, Wound dressing based on electrospun PVA/chitosan/starch nanofibrous mats: fabrication, antibacterial and cytocompatibility evaluation and in vitro healing assay, Int. J. Biol. Macromol. 122 (2019) 238–254.
[64] K. Hatano, H. Inoue, T. Kojo, T. Matsunaga, T. Tsujisawa, C. Uchiyama, Y. Uchida, Effect of surface roughness on proliferation and alkaline phosphatase expression of rat calvarial cells cultured on polystyrene, Bone 25 (1999) 439–445.
[65] T.-W. Chung, D.-Z. Liu, S.-Y. Wang, S.-S. Wang, Enhancement of the growth of human endothelial cells by surface roughness at nanometer scale, Biomaterials 24 (2003) 4655–4661.
[66] L. De Bartolo, M. Rende, S. Morelli, G. Giusi, S. Salerno, A. Piscioneri, A. Gordano, A. Di Vito, M. Canonaco, E. Drioli, Influence of membrane surface properties on the growth of neuronal cells isolated from hippocampus, J. Membr. Sci. 325 (2008) 139–149.