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Effect of Solution Properties of Cellulose Acetate/ Chitosan/Poly (vinyl alcohol) on The Morphology of Electrospun Nanofibers

Yerdawu Z. Gebremaryam*[a], Mikael A. Jimma[a], Roman O. Olekhnovich[b] and Mayya V. Uspenskaya[b, c]

[a]Chemical Engineering Center, ITMO University
Saint Petersburg, Russian Federation
[b]Saint Petersburg State University
Saint Petersburg, Russian Federation
[c]Peter the Great St. Petersburg Polytechnic University
Saint Petersburg, Russian Federation

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Abstract. Electrospinning is attracting significant research interest due to its versatility, adaptability, ease to use and economic feasibility. To meet strict requirements, efforts are being made to generate multi-characteristic nanofibers by blending various polymers. In the present work, electrospun nanofibers were fabricated from a mixture of CA/CS/PVA using a solution of acetic acid and water. At this time, the characterization of the polymer solutions (rheological properties, conductivity and pH value), as well as the morphology and diameters of the nanofibers was conducted. Thus, the various solutions with different total concentrations (5, 6, 7 and 8 % w/v) and ratios of CA/CS/PVA were utilized to assess their effects on the characteristics of the resulting nanofibers. The results show that, a total concentration of 8 % with polymer ratios of CA/CS/PVA (1.6/1.6/4.8 wt.%) exhibited the best solution, and nanofibers were successfully obtained. The electrospinning was conducted under the following parameters; voltage 30 kV, feed rate 0.5 mL/h, and tip-to-collector distance 115 mm resulting in a mean dimeter of 277 nm.

Keywords: electrospinning; cellulose acetate; chitosan; morphology; nanofiber; fiber diameter.

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* Corresponding author E-mail address: yirdaw25@gmail.com

Влияние свойств раствора ацетата целлюлозы/хитозана/поливинилового спирта на морфологию электроспряденых нановолокон

Е. З. Гебремарьям[а], М. А. Химма[а], Р. О. Олехнович[б], М. В. Успенская[б, в]

[а]Химико-технологический центр, Университет ИТМО
Российская Федерация, Санкт-Петербург
[б]Санкт-Петербургский государственный университет
Российская Федерация, Санкт-Петербург
[с]Санкт-Петербургский политехнический университет Петра Великого
Российская Федерация, Санкт-Петербург

Аннотация. Электроспиннинг привлекает значительный исследовательский интерес благодаря своей универсальности, технологичности, простоте в использовании и экономической целесообразности. Чтобы соответствовать строгим требованиям, предпринимаются усилия по созданию нановолокон с различными характеристиками путем смешивания различных полимеров. В настоящей работе были изготовлены электроспряденые нановолокна из смеси CA/CS/PVA с использованием раствора уксусной кислоты и воды. В это время была проведена характеристика растворов полимеров (реологические свойства, электропроводность и значение pH), а также морфология и диаметр нановолокон. Таким образом, для оценки их влияния на характеристики полученных нановолокон были использованы различные растворы с различной суммарной концентрацией (5, 6, 7 и 8 % по массе) и соотношениями CA/CS/PVA. Результаты показывают, что наилучшее решение было получено при общей концентрации 8 % и соотношении полимеров CA/CS/PVA (1.6/1.6/4.8 мас.%), и нановолокна были успешно получены. Электроформование проводилось при следующих параметрах: напряжение 30 кВ, скорость подачи 0,5 мл/ч и расстояние от наконечника до коллектора 115 мм, в результате чего средний диаметр составлял 277 нм.

Ключевые слова: электроспиннинг, ацетат целлюлозы, хитозан, морфология, нановолокно, диаметр волокна.

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Introduction

Electrospinning is a process that creates nanofibers from polymer solutions or molten polymers by applying a high electric field. This simple and versatile technique of producing nanoscale fibers has attracted significant research attention due to its economic viability, ease of use, diversity, and

adaptability [1–5]. These nanofibers offer several benefits compared to conventional fibers, including a higher surface-to-volume ratio, lightweight characteristics, porosity, flexibility, and the possibility of incorporating chemical functionalities relative to other known materials [6–8]. Consequently, due to these characteristics, the use of nanofiber materials has recently increased across a wide variety of applications, including scaffolds for tissue engineering, filtration media, controlled drug release, biosensors, wound dressings, specialized membranes, and antimicrobial activities [3, 9].

Although electrospun nanofibers have been used in various applications, the mechanical properties of these nanofibers play a critical role in determining their performance and success. The mechanical properties of nanofibers, particularly their stiffness, significantly influence cell behavior and tissue regeneration by affecting cell migration and elongation, which are crucial for tissue engineering applications [10]. In addition, the mechanical strength of the fibers can influence the rate of drug release, which is critical for controlled drug delivery applications [11]. To enhance the properties of nanofibers and achieve the desired properties of the nanofibrous material, it is better to blend two or more polymers. This is especially important when producing nanofibers based on biopolymers due to their low mechanical properties.

Among natural polymers, chitosan has attracted considerable interest due to its advantageous biological properties [12]. It has been extensively researched for use in biomedical and separation technologies due to its unique features, which include biocompatibility, hydrophilicity, bioactivity, and low toxicity [13, 14], as well as excellent antimicrobial, antioxidant properties, moderate thermal degradation [14, 15]. Nevertheless, chitosan nanofibers exhibit poor chemical, and mechanical stability, and they swell excessively in water, which reduces their mechanical strength and makes them unstable [16]. Furthermore, the mechanical properties of the fibers depend on their orientation; an increase in fiber disorientation leads to a decrease in these properties. Thus, chitosan contributes to the disorientation of the nanofibers [17]. On the contrary, chitosan is a promising absorptive material for various applications due to the abundance of free amine ($–NH_2$) groups, which allow for the absorption of heavy metals [15, 17, 18].

Cellulose is another the most abundant naturally occurring polymeric material, characterized by its polyfunctional macromolecular structure and environmentally benign nature. The CA has many desirable properties that make it ideal for a variety of applications such as good mechanical properties, biodegradability and biocompatibility with high thermal degradation characteristic. The fabrication of electrospun nanofiber based on cellulose is nevertheless challenges due to complexity of cellulose structure and properties, its solubility [1, 3, 19–22], and its lack of reactive functional [17]. Thus, blending chitosan and cellulose acetate can improve the mechanical and absorption properties of the nanofibrous material, as cellulose acetate has excellent mechanical properties, while chitosan contains primary amine (–NH2) and hydroxyl (–OH) groups, and the hydroxyl (OH) groups in cellulose acetate (CA) can better reaction groups for the sorption of metals [12]. Besides, during the preparation of the polymer solution using a binary solvent of acetic acid and water. The cellulose acetate is insoluble in water due to strong intermolecular hydrogen bonding [13], but mixing of acetic acid with water enhance the solubility of cellulose acetate. On the other hand, the electrospinnability of chitosan and cellulose acetate can be improved by blending with other polymers such as polyvinyl alcohol, polycaprolactone and polylactic acid or demineralize, deproteinize, and deionized water as a solvent [13, 23, 24]. Poly (vinyl alcohol) (PVA) is a water-soluble polymer with

several hydroxyl groups in its side chains [25]. PVA solution is a synthetic polymer that is non-toxic, air soluble, and has excellent thermal, gas permeability, and chemical resistance properties [26]. Blending PVA with polysaccharides such as cellulose and chitosan enhances the spinnability in electrospinning [25–27]. Besides, the structural matrix formed by the consolidation of chitosan and cellulose shows remarkable mechanical performance and biocompatibility, making them idea for biomedical application [28, 29].

Despite this, preparing the appropriate solution of the three polymers for electrospinning is the main challenge, as the properties of the polymer solution affect the results of the electrospinning process. The polymer solution parameters (polymers molecular weight, viscosity and conductivity), processing conditions (voltage, the distance from the spinneret tip to the collector, flow rate, etc.), and ambient conditions (relative humidity and temperature) can influence electrospinning and the morphology of the resulting fibers. Thus, understanding these parameters enables the construction of electrospinning configurations that can affect the final fibers' shape, size, and alignments [30–32]. Therefore, the main objective of this work was to study the possibility of obtaining electrospun nanofibers from a solution of cellulose acetate, chitosan and polyvinyl alcohol using acetic acid and water as components of binary solvents. In addition, characterization of the morphology and diameters of electrospun nanofibers was conducted.

1. Materials and Methods

1.1. Materials

The materials utilized for this study were Chitosan (CS) (M_w = 200 kDa), Cellulose acetate (CA) (the average M_w = 31.65 kDa), and Polyvinyl alcohol (M_w = 25.6 kDa). The chosen solvents were acetic acid (> 99 %) and distilled water. These solvents were selected based on the significantly solubility of chitosan, cellulose acetate and polyvinyl alcohol in them.

1.2. Nanofabrication of CA/CS/PVA

1.2.1. Solution Preparation

The solution of CA/CS/PVA with different concentrations (5, 6, 7 and 8 %) and ratios of the polymers were utilized with the mixture of acetic acid/distilled water (70/30 Vol.%) as a solvent. In the same way, the different ratios of the polymers (0.5/0.5/4, 0.75/0.75/3.5, 0.6/0.6/4.8, 0.9/0.9/4.2, 0.7/0.7/5.6, 1.05/1.05/4.9, 0.8/0.8/6.4, 1.2/1.2/5.6, 1.4/1.4/4.2, 1.75/1.75/3.5, 1.6/1.6/4.8 and 2/2/4 wt.%) CA/CS/PVA respectively were used. Hence, at this time the polymers were dissolved step by steps first cellulose acetate, chitosan and polyvinyl alcohol were added successively. Thus, in order to achieve the homogenous solutions for each solution carried out 300 rpm using magnetic stirring for 3 h and with temperature of 60 °C.

1.2.2. Characterization of the Solution

After each solution were prepared conductivity and pH value of the solutions were measured by SevenCompact Duo pH/Conductivity meter S213 (METTLER TOLEDO) with a temperature of 25 ± 1 °C. In the same way, the non-Newtonian behavior of the solution was analyzed using a Rheometer MCR502 (Anton Paar) to determine the kinematic viscosity of the solution. For each measurement were made at room temperature (25 °C) and the shear rate rage was 0.1 to 955 s^{-1}.

1.2.3. Electrospinning Process

The electrospinning of CA/CS/PVA nanofibers was performed using NANON-01A (MECC CO., LTD., Fukuoka, Japan). The electrospinning undertaking was carried out at a temperature of 25 ± 2 °C and a controlled relative humidity of 50 ± 2 %. Hence, in order to obtain the nanofibers without defects the technical parameters of electrospinning were changed as follows; applied voltage from 20 to 30 kV, feed rate from 0.1 to 1 mL/h, and tip-to-collector distance was set to 115mm. Then, the fibers were collected on a plate collector covered by aluminum foil.

1.3. Morphology and Mean Diameter of Nanofibers

The morphology of CA/CS/PVA electrospun fibers was observed under optical microscope Olympus STM6 (OLYMPUS Corporation, Tokyo, Japan). To improve colorfulness and contrast the result of the fibers differentially interferential contrasting technique (DIC) was utilized. The diameters were analyzed by ImageJ software (National Institutes of Health, Bethesda, MD, USA).

1.4. Statistical Analysis

The results were conducted by selecting hundreds of fibers samples, in order to determine the mean diameter of the nanofibers. The data were analyzed using one-way ANOVA, p-value of less than 0.05 was considered statistically significant.

2. Result and Discussion

The formation of fibers and morphology of fibers are significantly influenced various solution parameters including concentration, molecular weight, viscosity and conductivity. The pH value significantly influences chemical and physical properties of the fiber, but has less effect on spinnability in electrospinning [33]. In Table 1, various solution of CA/CS/PVA polymer ratios were presented with the total concentrations ranging from 5 % to 8 % using acetic acid/water (70/30) solvent system. Similarly, the physicochemical parameters such as viscosity, conductivity and pH value of the solution were carried out. Hence, when the concentration and the polymer ratio of CA/CS increase the parameters such as conductivity and viscosity of the solutions increase. Although the total concentration percentage remained constant, the viscosity and conductivity of the solution increased due to rising amounts of cellulose acetate and chitosan. On the other hand, as the concentration and polymer ratio increase the pH value decrease.

Electrical conductivity plays a crucial role in shaping the formation and structure of electrospun fibers. It determines charge mobility, which influences electrostatic repulsion force and impacts the morphology of electrospun fibers. The increase of electrical conductivity of the solution, leads to a significant decrease in diameter, whereas at low conductivities, the jet elongation is inadequate to produce uniform fibers [34, 35]. In this place, the conductivity of the solution was carried out by increasing the ratios of CA/CS, while simultaneously decreasing the ratio of PVA. The relationship between the total concentration and polymer ratios of CA/CS/PVA on electrical conductivity was conducted based on the outcomes of the solutions presented in Table 1.

In Fig. 1 shown, the total concentration of CA/CS increases, the electrical conductivity of the solution increased from 647 μS/cm to 1036 μS/cm. Chitosan acts as a partial polyelectrolyte and increases the conductivity of polymer solutions [17]. Furthermore, the electrical conductivity increases

Table 1. Solution parameter results in different total concentration and polymer ratios

Total concentration% (w/v)	Polymer ratios (CA/CS/PVA, wt.%)	Conductivity (σ) in μS/cm	Viscosity (η) in mPa.s	pH value
5	0.5/0.5/4	647	743.51	3.18
	0.75/0.75/3.5	769	787.34	3.11
6	0.6/0.6/4.8	762	764.65	3.15
	0.9/0.9/4.2	805	806.23	3.07
7	0.7/0.7/5.6	801	790.61	3.01
	1.05/1.05/4.9	924	832.73	2.90
8	0.8/0.8/6.4	863	805.68	2.95
	1.2/1.2/5.6	975	858.82	2.87
7	1.4/1.4/4.2	925	1312.50	2.91
	1.75/1.75/3.5	1013	1965.72	2.72
8	1.6/1.6/4.8	1006	2528.85	2.82
	2/2/4	1036	2736.43	2.68

with the addition of CA due to the larger amount of oxygen in CA molecules [36]. Fig. 2 present the concentration increased from 5 to 8 %, and increasing the polymer ratio of cellulose acetate and chitosan, while decreasing polyvinyl alcohol. In this case, the viscosity of the solution has increased from 743.51 mPa.s to 2736.43 mPa.s, with a shear rate range of 0.1 to 955 s^{-1}, and at room temperature (25 °C). The viscosity is significantly enhanced due to the hydrogen bonding interaction between the -NH_2 and -OH groups of chitosan and the -OH groups of PVA, along with the entanglement of polymers and the random orientation of the polymer chains [37]. As the shear rate increases, the viscosity decreases, which can be explained by the influence of exposed -NH_3 on electrostatic and steric repulsion, as well as the reorganization of the polymeric structure[38, 39]. An increasing in polymer concentration

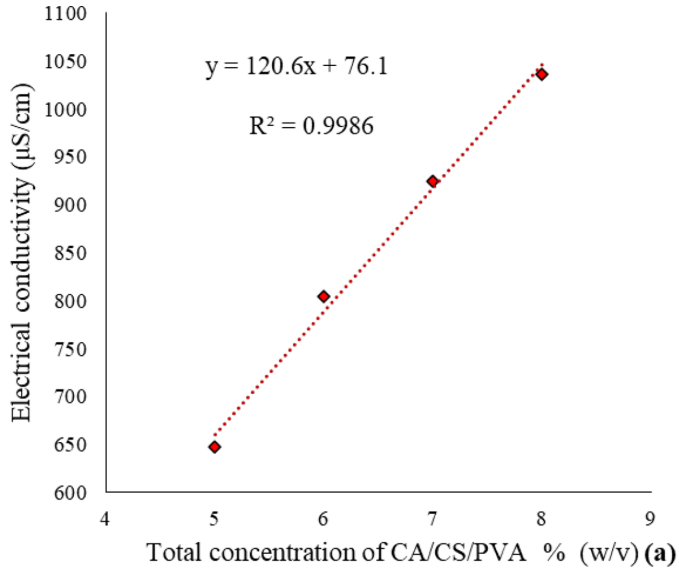

Fig. 1. Effect of the total conecntration of CA/CS/PVA on electrical conductivity of the solution

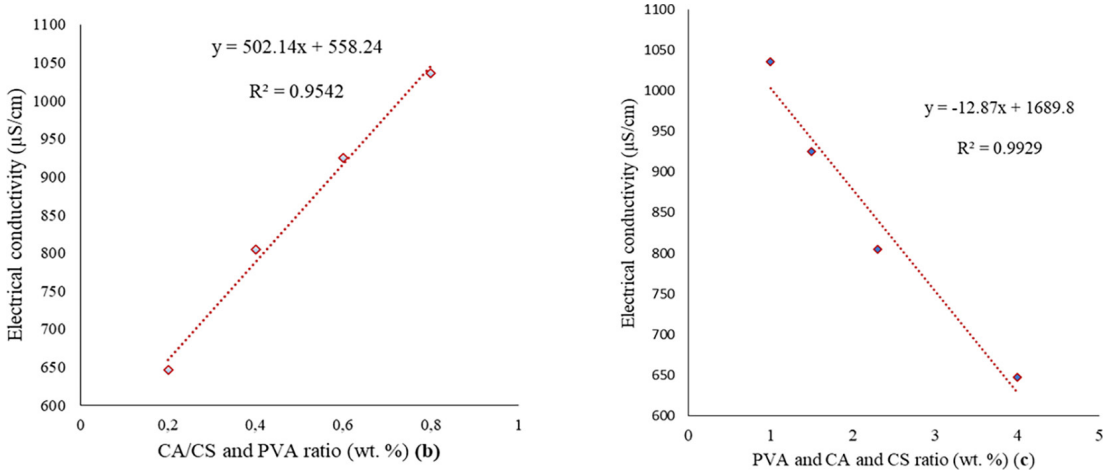

Fig. 2. Effect of polymer ratio on the electrical conductivity of the solution

leads to higher viscosity and more chain entanglements. This makes the greater viscoelastic force to counterbalance the Coulombic streatching force leaing to the formation of polymeric nanofibers with fewer or no beads [40].

If the CA/CS total concentration increase and the decreasing in PVA (Fig. 3 (e)) drastically increased the viscosity of the solution. In fact, the viscosity of a polymer solution is influenced by both the concentration of the polymer and its molecular weght. Hence, the molecular weights of CA/CS are much higher than that of PVA, which results in an increased viscosity of the solution.-Table 2 presented the process of fabricating nanofibers through electrospinning utilizing the different ratios of CA/CS/PVA wt.%. In addition, the total concentrations varied from 5–8 %, along with technical parameters of the electrospinning process such as applied voltage, feed rate and tip-to-collector distance. The tip-to-collector distance was set at 115 mm, a feed rate range of 0.1–1 mL/h and a voltage range of 20 kV-30 kV. Based on the data presented in Table 2, fibers were not obtained when the total concentration was 5 %. However, when the total concentration increased to 6 % with voltages of 25 and 30 kV, electrospinning began to obtain the fibers with several defects. On the

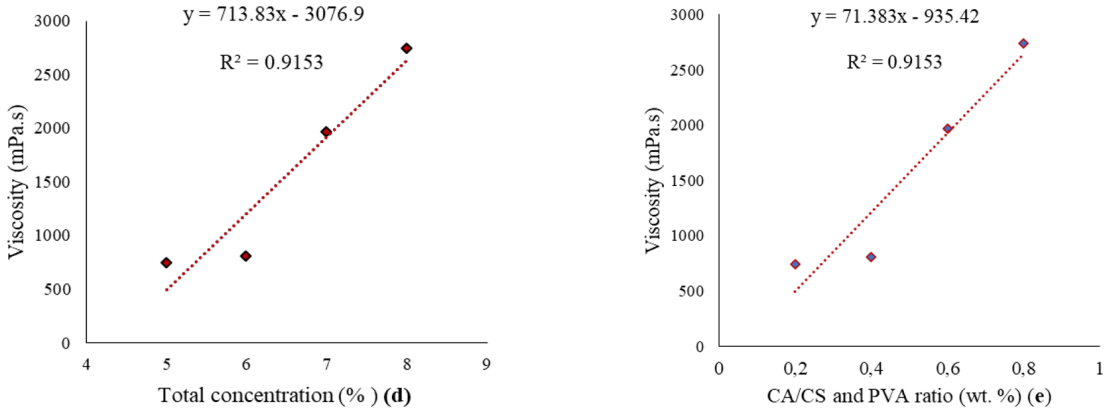

Fig. 3. Effect of concentration (d) on viscosity (e) of the solution in different ratio of CA/CS/PVA

Table 2. Results of electrospinning nanofabrication at various total concentration/CA/CS/PVA ratio

Tip-to-collector distance (mm)			Freed rate (mL/h)									
Total concentration % (w/v)	CA/CS/PVA, wt.%	Applied voltage (kV)	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1
5	0.5/0.5/4	20	_	_	_	_	_	_	_	_	_	_
		25	_	_	_	_	_	_	_	_	_	_
		30	_	_	_	_	_	_	_	_	_	_
	0.75/0.75/3.5	20	_	_	_	_	_	_	_	_	_	_
		25	_	_	_	_	_	_	_	_	_	_
		30	_	_	_	_	_	_	_	_	_	_
6	0.6/0.6/4.8	20	_	_	_	_	_	_	_	_	_	_
		25	_	_	_	_	_	_	_	_	_	_
		30	_	_	_	_	_	_	_	_	_	_
	0.9/0.9/4.2	20	_	_	_	_	_	_	_	_	_	_
		25	_	_	_	±	±	±	±	_	_	_
		30	_	_	_	_	_	_	±	±	±	±
7	0.7/0.7/5.6	20	_	_	_	_	_	_	_	_	_	_
		25	_	_	_	_	_	_	_	_	_	_
		30	_	_	_	_	_	±	±	±	±	±
	1.05/1.05/4.9	20	_	_	_	_	±	±	±	_	_	_
		25	_	±	±	+	+	+	±	±	±	±
		30	_	±	±	±	+	+	+	+	+	±
	1.4/1.4/4.2	20	_	_	_	_	_	_	_	_	_	_
		25	_	_	±	±	+	+	+	±	±	±
		30	_	_	±	±	+	+	+	+	+	±
	1.75/1.75/3.5	20	_	_	±	±	±	±	±	_	_	_
		25	_	_	±	±	+	+	+	+	±	±
		30	_	_	_	±	±	+	+	+	±	±
8	0.8/0.8/6.4	20	_	_	_	_	_	_	_	_	_	_
		25	_	_	±	±	±	±	_	_	_	_
		30	_	_	_	_	±	±	±	±	_	_
	1.2/1.2/5.6	20	_	_	_	±	±	±	±	_	_	_
		25	_	±	±	+	+	+	+	+	+	±
		30	_	±	±	±	+	+	+	+	+	+
	1.6/1.6/4.8	20	_	_	_	±	±	±	_	_	_	_
		25	_	_	±	+	+	+	+	+	±	±
		30	_	_	_	_	+	+	+	+	+	±
	2/2/4	20	_	_	±	±	±	±	±	_	_	_
		25	_	_	±	±	+	+	+	±	±	±
		30	_	_	_	±	±	+	+	+	±	±

() particles, (±), Nanofibers with defects, (+) Nanofibers without defects

other hand, as the total concentration increased to 7 and 8 %, better fiber results were obtained with electrospinning voltages at 25 and 30 kV.

Indeed, the electrospinnability of a polymer solution to be electrospun into fibers, depends on various intrinsic factors, such as the rheological properties of the spinning dope and conductivity, as well as environmental factors such as air flow rate, humidity and temperature. In numerous instances, when the spinning dope lacks adequate molecular entanglement, it tends to break into separate droplets instead of creating a continuous jet, leading to the formation of particles rather than fibers [41–43]. As previously mentioned, the PVA is a common polymer to utilized in electrospinning and to enhance spinnability of other polymers. Therefore, in this work the total concentration of CA/CS polymers were increased until they were possible for processing in electrospinning and allowing for the production of defect-free nanofibers. Thus, to obtain defect-free nanofibers, the parameters and the setup of the electrospinning process should be controlled. The microstructural characteristics of the electrospun CA/CS/PVA polymer were examined. The morphology and nanofibers mean diameter are affected by the total concentration and the ratio of polymer. Despite utilized various ratio of CA/CS/PVA polymers and total concentration, however the results of electrospinning were defects when below and above the specific ranges.

In this work, a good fiber result was obtained at the total concentration of 8 %. Fig. 4 shows the morphology and diameter distribution of CA/CS/PVA fibers at a total concentration of 8 % with polymer ratios of 1.2/1.2/5.6 and 1.6/1.6/4.8 wt.% respectively. Indeed, a polymer ratio of CA/CS/PVA (2/2/4 wt.%) was used to obtain fibers; but measuring their diameter is challenging because of the many imperfections in the fibers, which make it difficult to measure. Consequently, to successfully produce defect-free nanofibers through electrospinning, use a polymer ratios of 1.2/1.2/5.6 and 1.6/1.6/4.8 (CA/CS/PVA) along with the following technical electrospinning parameters; applied voltage of 25 kV and 30 kV, a feed rate of 0.5 mL/h, and a tip-to-collector distance of 115 mm. According to the literature as the CA concentration of increase, the diameter of result fibers also increases. This is due to the higher solid content and a higher viscosity that would resist the flow and elongation and result in less stretched jet [44, 45]. On the contrary, an increase in the CS concentration and higher voltages lead to the formation of smaller fiber diameters. This behavior between the diameter of nanostructured membranes and the concentration occurs, because CS acts as a partial electrolyte, increase conductivity of the polymer solution and thereby decreasing the diameter of the nanostructured membranes. Another reason to decrease the diameter can be observed, when increasing concentration due to the partial clogging or restrictions to flow at the tip of the needle [46–48]. The mean diameter in this study ranges from 232 nm to 294 nm.

3. Conclusion

This study investigated the possibility of production electrospinning nanofiber based on CA/CS/PVA and evaluation effects of solution properties of CA/CS/PVA on the morphology and mean diameter results of receiving fiber. During the production of nanofibers through electrospinning, several factors were taken in accounts such as solution total concentration, viscosity, conductivity, and polymer ratio, which significantly affect the final morphology of the electrospun nanofibers. The solutions were prepared with various ratios of CA/CS/PVA and total concentrations ranging from 5 to 8 % (w/v) using a solvent mixture of acetic acid and water (70/30 Vol.%). Despite this, a total concentration of 8 %

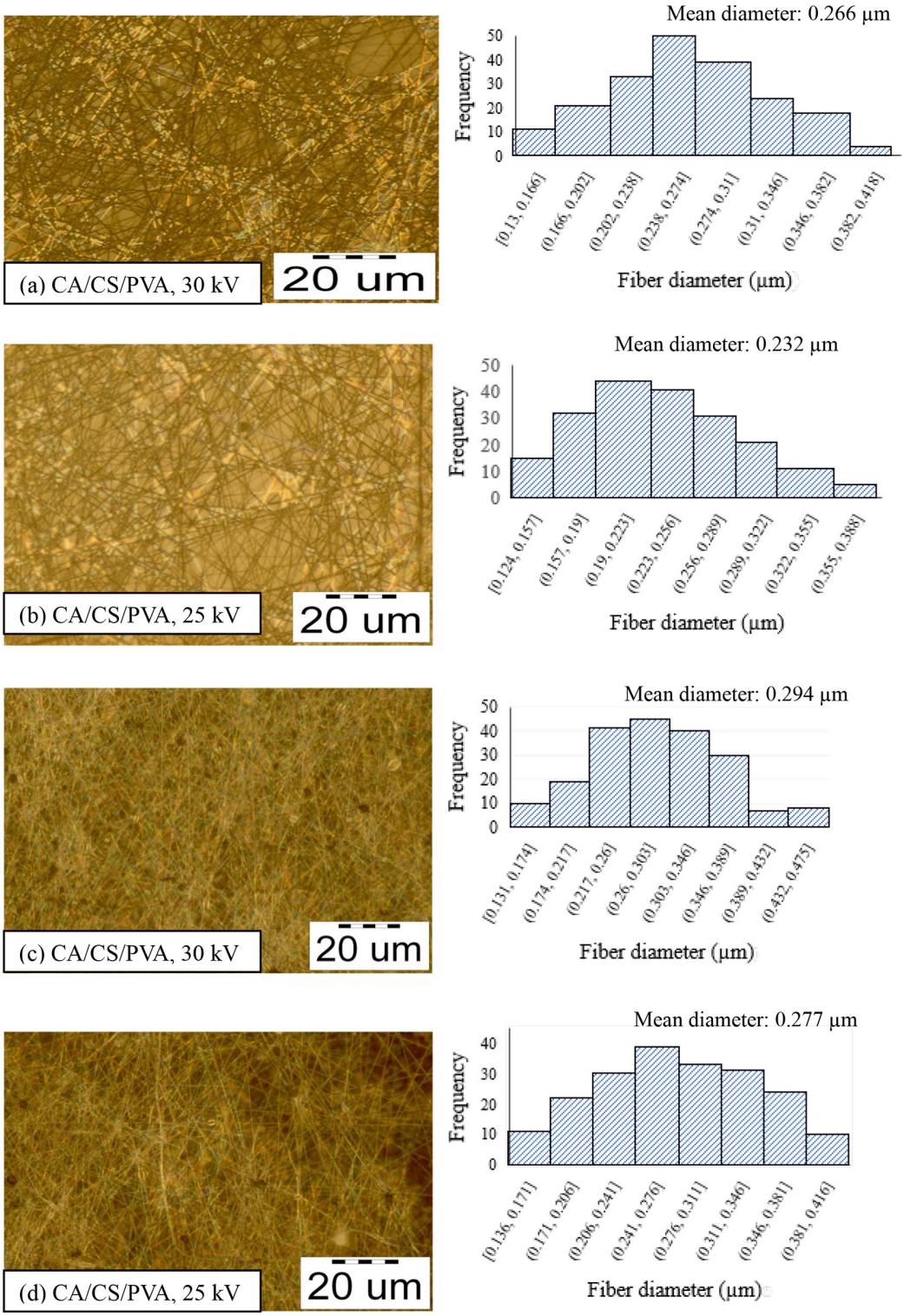

Fig. 4. Morphology and diameter distribution of nanofiber in different polymer ratio (a, b) CA/CS/PVA (1.2/1.2/5.6), and (c, d) CA/CS/PVA (1.6/1.6/4.8)

(w/v) with polymer ratio of CA/CS/PVA (1.6/1.6/4.8) successfully produced nanofibers. The mean fiber diameter varied from 232 nm to 294 nm at voltage of electrospinning in 25 kV and 30 kV, feed rate of 0.5 mL/h and a tip-to-collector distance of 115 mm.

Data Availability

Sufficient data have been included in the manuscript. Additional data can be kindly requested from the corresponding Author.

Conflicts of Interest

There is no conflict of interest to declare.

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