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Preparation and Characterization of Polyvinyl Alcohol/Starch/Bio Oil Extraction Blends Coating for Food Packaging Using Electrospinning Technique

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Abstract

In this project, natural and synthetic polymers were combined to create a film using the electrospinning technique. Starch was used as a natural biodegradable polymer in this study, and mixtures of this polymer were made with polyvinyl alcohol polymer in various proportions as an industrial polymer to improve pumpability, and cress seed gum was used to improve the behavior of the nanofiber fabric towards water using the fabric's wetting angle. In addition to measuring the viscosity of the solutions and the volumetric concentrations of the solutions used for pumping, the electrospinning technique was used to prepare nanofiber fabric from different ratios of mixtures under pumping conditions (applied voltage 20 kV, pumping rate 1 ml/hour, pumping distance 20 cm, and needle diameter 0.4 mm). The best volume ratio according to wettability was (PVA:STA (40:60) and (PVA:CSM (40:60), which were produced in two groups with varied volume ratios. The two best ratios were then combined. Finally, the chemical structure of these ideal blends nanofibers was detected using Fourier transform infrared spectroscopy, and the shape was evaluated using a field emission scanning electron microscope. The glass transition temperature was measured using a thermal differential scanning microscope to test the miscibility of polymeric mixtures. The results showed that the wetting angle of the pva:sta mixture (40:60) was 66 degrees and that the mixture was miscible according to the glass transition temperature obtained using differential thermal microscopy. The results of the PVA: CSM mixture, on the other hand, showed that the mixing ratio was also the best in terms of the wetting angle (40: 60), where it was 69 degrees, but the mixture was immiscible according to the differential scanning microscope, which revealed two degrees of glass transition. Adding cress seed oil to a mixture (PVA: STA) improves polymer compatibility and changes the mixture's viscosity behavior. In terms of raising the wetting angle to 74 degrees and optimizing the behavior of the electrospinning solution, the optimal mixing ratios (PVA: STA: CSM) were (40:30:30). The results reveal that the best volume ratios are shrinking in diameter, as determined by the Digimizer Image Analysis Software application, Furthermore, the FTIR results suggest that substantial hydrogen bonding interactions occurred between the starch and PVA components, improving their compatibility. These findings show that STA and CSM could be used as a new biopolymer source for the production of nanofibers that could be used in a range of applications, including food packaging..

Keyword: Electrospinning, Cress seed mucilage, Starch, Poly (vinyl alcohol) , food packaging.

1. Introduction

The development of microbiological creatures has led to the advent of new strains that are more resistant to standard bactericides, allowing them to avoid excessive growth, particularly when they are preserved in their nutrients for a long time. Micro-antibacterial filters can now be used instead of traditional films, thanks to advances in nanotechnology [1]. Petroleum-derived polymers are the most common type of packaging used in the food business. However, research into the development and characterization of biodegradable films has increased dramatically, owing to a desire to reduce the environmental impact of synthetic packaging materials. [2-3]. Because of their outstanding Biodegradable polymer films have long been regarded as tempting alternatives to plastic packaging due to their biodegradability, biocompatibility, edibility, and the variety of their potential applications. Biopolymers (e.g. starch, cellulose, pullulan, and chitosan) based on proteins and polysaccharides (e.g. starch, cellulose, pullulan, and chitosan) are widely utilized in the food industry to make biodegradable and environmentally friendly packaging films. [4]. The hydrophilic feature gives the material biodegradability and ensures that the wetness angle is smaller than 90 degrees [5] . The substance gets increasingly hydrophobic as the

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wetting angle rises above 90 degrees [6]. Hand casting [7], spray coating [8], and electrospinning of fibers [10-11-12] are all ways for obtaining homogeneous coatings from polymeric and Nano composites materials. Electrospinning of fibers is the most dependable and effective process for creating polymeric coatings [12] PVA (polyvinyl alcohol) is a semi-crystalline, synthetic, healthy, biocompatible, and water-soluble polymer with unusual physical properties due to the presence of many –OH groups and hydrogen-bond formation [13]. PVA also has outstanding film-forming properties, making it one of the best bio-based polymers for packaging [14-15]. Starch (STA) is an intriguing raw material for creating edible films because of its low cost, non-toxicity, ease of availability, and excellent film-forming properties. STA has been extensively used in the development of biofilms for use in food packaging. [16]. Mucilage is a long-chain polysaccharide with a high molecular weight that is a type of hydrocolloid. It's used in the food industry. The (Brassicaceae) family's cress seed mucilage (Lepidium sativum L.) grows in Iran, India, North America, and portions of Europe. It is anionic in nature and contains a significant amount of mucilaginous chemicals with a molecular weight of roughly 540 kDa. [17-18]. Polymeric blends are an essential component of modern technology. It refers to the process of combining two or more polymers to create a new product with better qualities. Miscible and immiscible polymeric blends are the two types of polymeric mixes. The ability to blend polymers is determined by various aspects, including the structural similarity of the polymers, the size similarity of the polymers molecules, and the thermal orientation similarity of the polymers. The higher the similarity between these polymers, the greater the chance of miscibility [19] .Many prior research have included fiber electrospinning technology in the preparation of packaging films. Jurgita et al. (2012) used the electrospinning technique with two types of rotating electrodes to create bicomponent nanofibres from poly(vinyl alcohol) (PVA) and modified cationic starch (CS) mixed solutions (PVA/CS mass ratio 3/1) with different total solids concentrations in water (8, 10 and 12 wt. percent) (a plain cylindrical electrode and an electrode withtines). The best results were obtained with a PVA/CS solution containing an 8 wt% solid concentration. The viscosity of the 12 wt. percent spinning fluid was much higher than that of the 8 wt. percent solution. These changes in viscosity had a substantial impact on the electrospinning process, since the less viscous fluid produced thinner nanofibres. When compared to the cylindrical electrode, the electrode with tines performed better because the diameter distribution was more uniform [20]. Susmita et al. published a study in 2015 on the use of a starch and PVA blend to improve starch tensile strength. PVA (Polyvinyl Alcohol) is a biodegradable and biologically safe synthetic polymer. This blend's nanocomposite was made with 1 percent, 2 percent, and 3 percent nanoclay loading. This polymer's thermal characteristics have been investigated. This mix and its nanocomposites were thermally characterized using Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA) in this paper. The nano fillers are well disseminated in the polymer matrix and exfoliated, according to XRD data, since there is no peak of nano clay layers till 10° angle in 1 percent, 2 percent, and 3 percent loading. The nano clay dispersion is optimal [21]. Pokhrel et al. 2019 published a paper on starch, a renewable, biodegradable, and low-cost natural biopolymer with high availability. The starch from Solanum tuberosum (potatoes) was isolated and its preliminary properties were investigated. Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy were used to examine the structure of the extracted starch (SEM). Using glycerol as a plasticizer and citric acid as a cross-linking agent, a series of starch-based biodegradable blends with polyvinyl alcohol (PVA) were made using the solution casting process and their biodegradable qualities were investigated. Fourier transform infrared spectroscopy (FTIR) and differential scanning calorimetry were used to determine the blend's structure (DSC). The morphology of potato starch reveals that it has an oval or ellipsoid form with a size of [22]

In this study, a combined natural starch polymers and mucilage with PVA polymers that are easy to spin, biodegradable, and have strong chemical, physical, and thermomechanical properties, resulting in the creation of a suitable solution for electrospinning nanofibers. The novelty of this work depends in the use of cress seed oil to modify the viscosity behaviour of polymeric mixtures by making them more compatible, which aids in the regularity of the process of pumping polymeric solutions into the electrospinning system, resulting in more uniform and efficient fiber production as well as changing the fabric's water behaviour.

2. Experimental

2.1 Materials

Poly vinyl alcohol with a molecular weight of 13000-23000 g/mol and an 88 percent hydrolysis rate, according to central drug house (P) Ltd., was used to prepare samples. Alpha Chemika, product corn starch (STA), and cress seed were obtained from

an Iraqi market. The electrospinning solutions were made with distilled water as the solvent.

2.2 Cress seed mucilage (CSM) extraction

Mucilage was extracted by cleaning the seed to remove any contaminants. The cress seed (5 to 100 (w/w)) was then added to deionized water, which was mixed with a magnetic stirrer and kept at room temperature for 15 minutes. After that, a cloth filter was used to separate the seed from the mucilage in the solution. The mucilage solution is then allowed to cool before being utilized in the experiment. The constituents detected in this mucilage were analyzed in the Babylon agriculture directorate/central laboratory, as shown in the table (1)

Table 1: illustrates the characteristic of the cress seed mucilage

Ca (ppm)	Mg (ppm)	K (ppm)	Na (ppm)	pH
250	**25**	**236.5**	**4.1**	**6.1**

2.3 Preparation of samples :

Table 2 shows the blended polymer and solution concentration ratios under electrospinning setti***The***

No.	Contents ratios **%**	**Con. w/v**	Θ^o
S1	PVA 100%	0.08	40
S2	PVA:STA (60:40)	0.058	58
S3	PVA:STA (50:50)	0.092	55
S4	PVA:STA (40:60)	0.047	66
S5	PVA:CSM (60:40)	0.068	64
S6	PVA:CSM (50:50)	0.65	58
S7	PVA:CSM (40:60)	0.062	69
S8	PVA:STA:CSM (40:30:30)	0.054	74

Electrospinnig conditions : applied voltage =20kv, electrospinning distance=20 cm, needle diameter=0.4 mm , rotate speed of collector 600 rpm , Flow rate = 1ml/hr

The first step was to dissolve (8g) PVA in (100 ml) of hot distilled water at 80oC for 90 minutes under magnetic stirrer to obtain a concentration of (0.08w/v), and then to prepare the starch gelatinization solution by dissolving (2.5g) starch in (100 ml) of hot distilled water at 90 oC for 15 minutes (0.025 con. w/v). PVA-STA solutions were then created by combining PVA and STA in various volume ratios (60:40, 50:50, and 40:60). The second stage was to make a cress seed mucilage solution with a concentration of 5g, then blend PVA with the mucilage solution for 1 hour using a magnetic stirrer to obtain homogenies solutions with varying volume ratios (60:40, 50:50, and 40:60). The ideal volume ratio based on the highest contact angle was (40:60) for PVA-STA and (40:60) for PVA-CSM from the first and second steps. Finally, using a magnetic stirrer, the two processes from PVA-STA-CSM were combined for 1 hour at the ideal volume ratio (40:30:30). Because these are miscible mixtures, the concentrations were determined using the mixing rule. Table 2 shows all of these steps.

3. Results and Discussion

3.1 Solutions result:

As shown in figure (1-a) and table 3, the concentration of ideal blends drops after introducing STA in a modest volume amount, according to the rule of mixture of blends. This is also demonstrated by the results of viscosity of solutions, as shown in figure (1-b), which show a decrease in viscosity (η) after adding STA because the concentration and volume fraction of STA are lower than those of PVA, and this blend is miscible, as shown in the DSC curve of (PVA + STA) fig 5a. Susmita et al 2015, Shanta 2019 [21-22] and RH095 [23]

Table 3. The physical properties of solution blends

Con%	η (cP)	δ (μS/cm)	S.T (mN/m)
0.08	348	821	35.58
0.047	170	445	38.46
0.062	420	934	40.51
0.054	390	695	38.6

However, because the blend is immiscible, viscosity increases with tiny amounts of CSM, as demonstrated in DSC curve of (PVA + CSM) mix fig 5b, and this corresponds with RH095, [23]. However, observe how the viscosity reduces with little cP when blending (PVA+CSM+STA), as shown in fig 1c. This is because the presence of STA between PVA and CSM promotes the blend's compatibility. This is in line with the rationale given by RH095 [23]. As demonstrated in Fig 1 d, adding STA to pure PVA

lowers the electrical conductivity (δ) because STA reduces the amount of free ions in the solution due to the presence of polar covalent connections between PVA and STA. Meanwhile, due to the ionic nature of CSM's mucilage, the electrical conductivity increased with the addition of CSM, which agrees with the results published by [24]. The CSM has a lot of free ions in its structure, as shown in table 1. As a result, the electrical conductivity of the solution increases

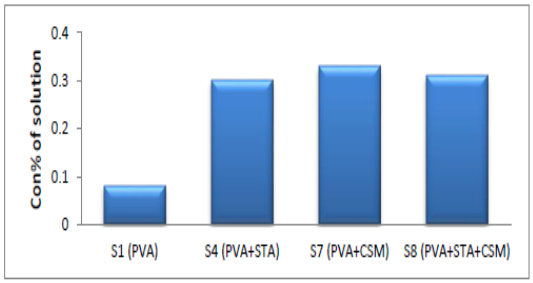


Fig. (1-a): show con% of the optimal volume ratio for blends

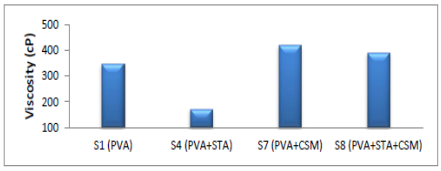


Fig. (1-b): show viscosity of the optimal volume ratio for blends

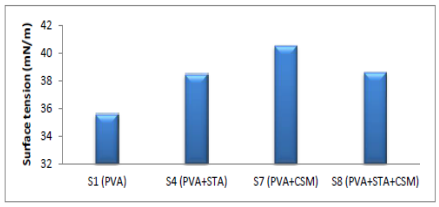


Fig. (1-c): show surface tension of the optimal volume ratio for blends

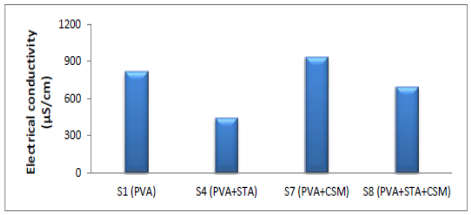


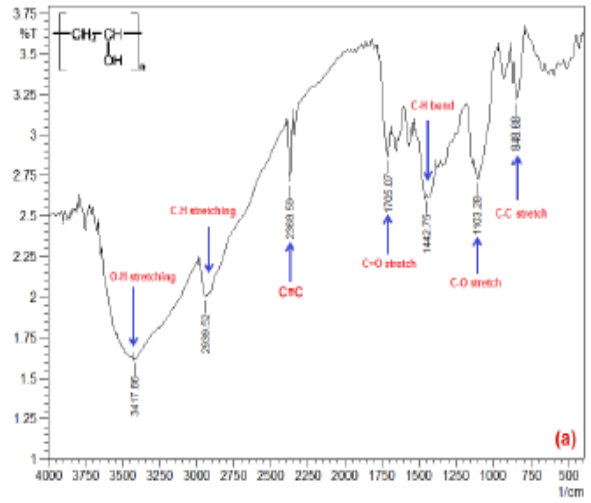
Fig. (1-d): show electrical conductivity of the optimal volume ratio for blends

Fig (1. a-d): illustrates the physical properties of optimal blends/ (a) concentration, (b)/ viscosity, (c)/ surface tension and (d)/ electrical conductivity

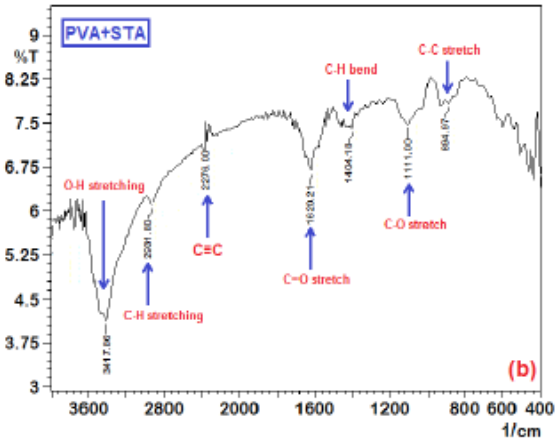
3.2 FTIR results:

FTIR was used to determine the chemical structure and functional groups of (PVA pure), (PVA+STA), (PVA+CSM), and (PVA+STA+CSM). An FTIR study was carried out to see if there were any interactions between CSM and PVA in electrospun nanofibers (Fig. 2a) and table 3. show the absorption peaks that presented with pure PVA. The occurrence of an ABS band for both components at approximately 3600-3300 cm-1, as shown in Fig 2. b, is owing to the vibration of the OH groups, as demonstrated. Amylase and amylopectin, both found in starch, exhibit a distinct peak for their C-H stretch vibration at 2900 cm-1. Because of the closely linked water molecules, the peak for isolated starch was 1500 cm-1. Changes in starch crystallinity can be seen throughout this band. The CH2OH side chain in amylase is given a sophisticated mode at 1150 cm-1 in extracted and commercial starch, which agrees with. [22] In the CSM spectra, the ABS bands 1129 cm^{-1} 164 (C-O stretch), 1400 cm^{-1} (symmetrical stretch of carboxyl groups), 1618 cm^{-1} 165 (asymmetrical stretch of carboxyl groups or intramolecular bound water), and 2923 cm^{-1} 166 (asymmetrical stretching of COOH groups) were identified (stretch symmetric-asymmetric of C-H). PVA's distinctive peaks were discovered to be 848 cm^{-1} 168 (C-C vibrations), 1095 (C-O stretch of residual acetate groups in the PVA matrix), 1438 (C-H_2 groups), 169 1735 (C =O), and 2940 cm^{-1} (C-O stretching of residual acetate groups in the PVA matrix) (stretching C-H of alkyl groups). This is also in line with claims made by Fahima and others. [23]. In the FTIR spectrum of PVA + CSM + starch, broad

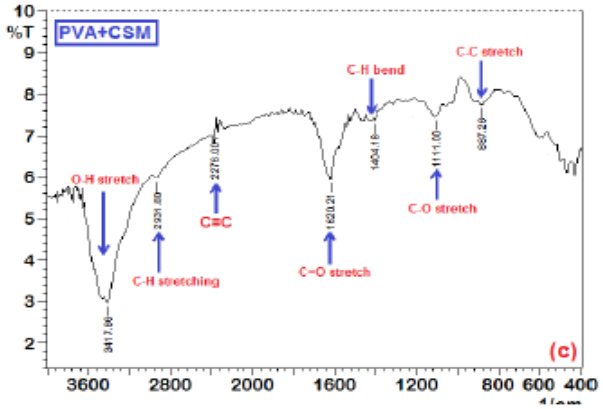
spectra were detected at approximately 3000cm-1 in O-H stretching (Fig. 2.d). Asymmetric ring vibrations were also found at 1150 cm^{-1}. In spectra between 1080 and 960 cm^{-1}, stretching vibrations in C-O groups were observed. At 3200 cm1 and 2880–2900 cm^{-1}, all of the processed film samples had bands that proved the C-H group, as well as bands that confirmed the CH2 group at 1245, 1405–1465, 2855, and 2916–2936 cm^{-1}. Films with starch nanocrystals were generated in chia-seed mucilage nanocomposites, and the FTIR spectra of the wide absorption peak at 3290 cm^{-1} related to the OH (hemicellulosic) group were obtained. Protein structures showed different peaks at 1400, 1500, and 1600 cm^{-1} . [25]



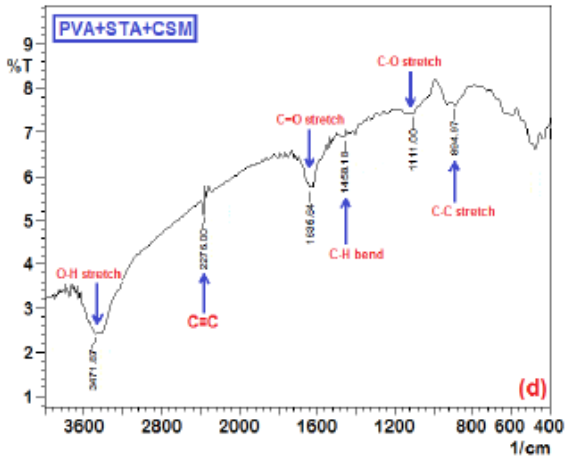
2.a



2.b



2.c



2.d

Figure (2): FTIR spectra of a: (pure PVA), b: (PVA+STA), c: (PVA+CSM) d:(PVA+STA+CSM)

Table 3: Bands wavenumber variation for pure PVA [14]

Type of bond	Stander transmission peak/cm^{-1}	Experimental transmission Peak/cm^{-1}
OH stretching	3475	3417
CH_2 asymmetric stretching	2933	2939
C≡C	2000-2400	2368
C=O stretching	1713	1705
Symmetric bending of CH_2	1432	1442
C—O stretching	1096	1103
CC stretching vibrations	844	848

3.3 Contact angle

Figure 3 and table 4 show the optimal volume ratios for nanofiber blends that show an increase in contact angle after adding STA and CSM but still in the range of hydrophilic character due to the OH bonds that were presented through mixing of solution samples that lead to the hydrophilic nature of its structure for these additions, which is in agreement with Abdullah et al. 2019 [26].

Table 4 shows the contact angles of the ideal volume ratios for mixes. Figure 3 is also included.

Table 4 shows the best samples' contact angles

No.	Contents ratios %	Contact angle (θ^o)
S1	PVA 100%	40
S4	PVA:STA (40:60)	66
S7	PVA:CSM (40:60)	69
S8	PVA:STA:CSM (40:30:30)	74

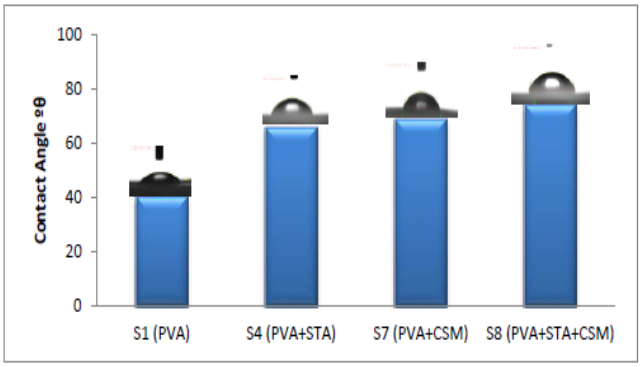
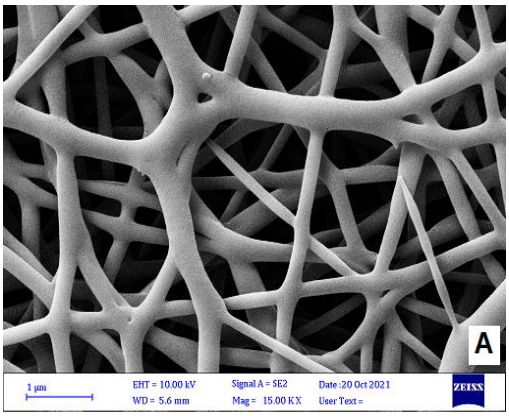


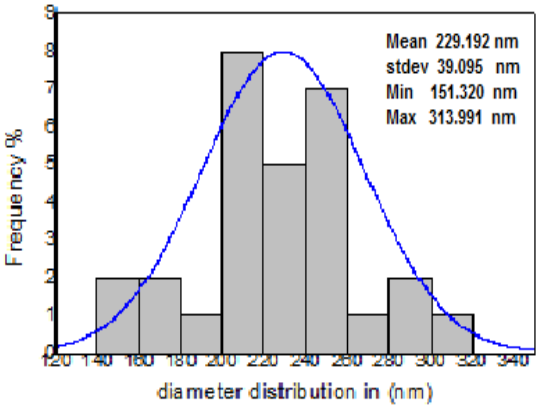
Fig. (3): show contact angle of the optimal volume ratio for samples.

3.4 FESEM results

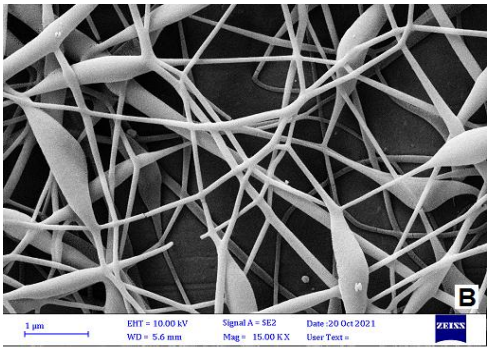
The morphological properties of nanofibers were investigated using FESEM. The average fiber diameter decreases after adding STA with bead formation, as seen in fig 4b, due to a drop in solution viscosity and instability of the electrospinning process. Figure (4C) shows that increasing the electrical conductivity of CSM causes nanofiber diameters to shrink significantly [27].



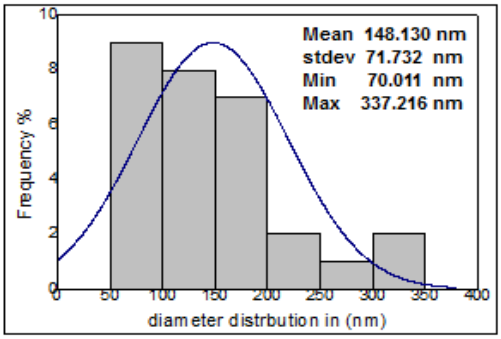
a.1 FESEM Images (PVA) NF.



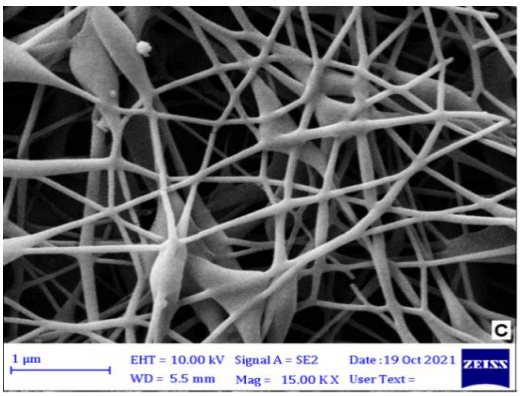
a.2 diameter distribution (PVA) NF



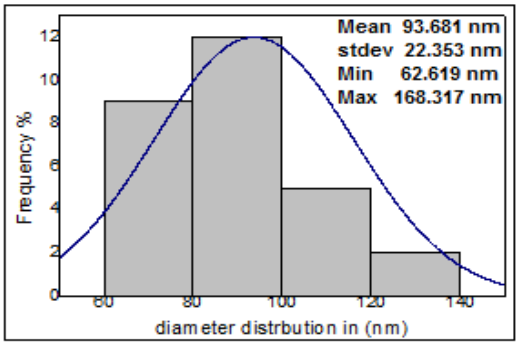
b. 1 FESEM Images (PVA+STA) NF



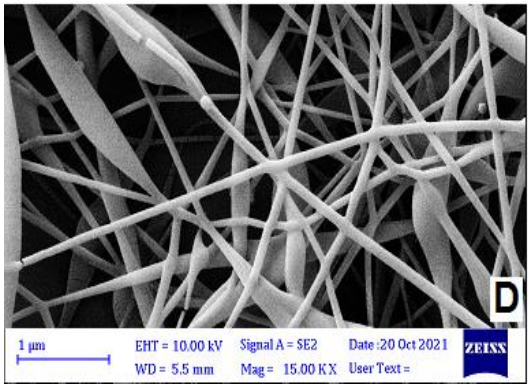
b.2 diameter distribution (PVA+STA) NF



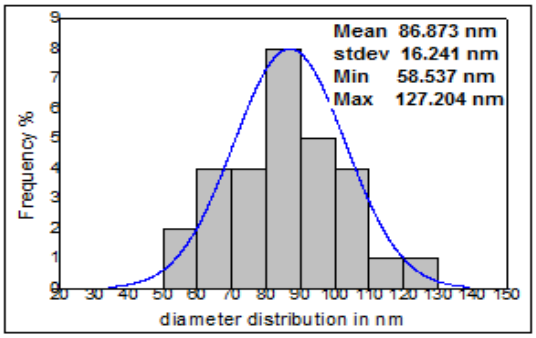
c. 1 FESEM Images (PVA+CSM) NF



c.2 diameter distribution (PVA+CSM) NF



d. 1 FESEM Images (PVA+STA+CSM) NF



d.2 diameter distribution (PVA+STA+CSM) NF

Figure (4): FESEM Micrographs the magnification power of 100 nm and Nano-fibers diameter distribution histograms for (A) S1(PVA), (B) S4 (PVA+STA),(C) S7 (PVA+CSM) and (D) S8(PVA+STA+CSM).

Figure (5a-d) and table (5) show directionality histograms and directionality analysis of the optimal blends, which show that changing the orientation from (-5.80) at (S1) to orientation angle (22.17) at (S8) with a change in the dispersion angle of optimal blends from (8.92o to 4.54°, and a decrease in the amounts of directed fiber for S4 and an increase for S7, as well as an increase in the goodness of orientation samples, indicates changing the orientation The ideal blends' alignment orientation as evaluated by (2D-FFT).

Table (5): The directionality analysis for optimal blends (by utilize Fourier components).

Sample No.	Direction (˚)	Dispersion (°)	Amount	Goodness
S1	-5.80	8.92	0.34	0.45
S4	-54.69	3.60	0.17	0.48
S7	3.75	4.90	0.37	0.56
S8	22.17	4.54	0.28	0.74

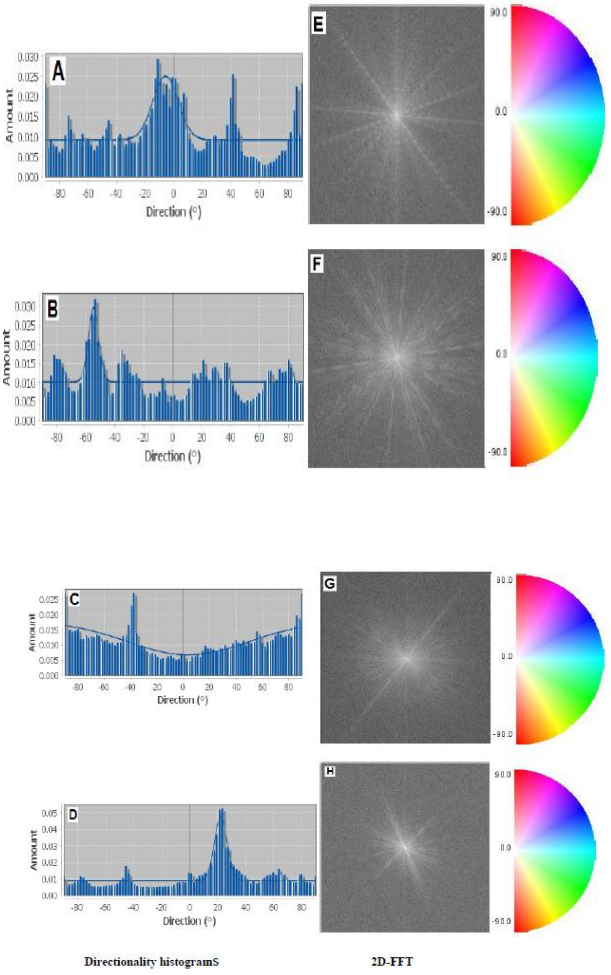


Figure (5): Directionality histogram, and 2D-FFT for (a) S1(PVA), (b) S4 (PVA+STA),(c) S7 (PVA+CSM) and (d) S8(PVA+STA+CSM).

3.5 DSC Results

Table 6 and Fig 6 a-d show the thermal analysis of PVA and its blends.

Figure 6a shows that pure PVA has a single Tg of 85.78 o C and a melting point of 222 oC, which agrees with Susmita 2015 [21]. When starch is added to PVA, the Tg of the blend rises to 89 degrees Celsius as in fig 6b. This is due to the presence of increased contact between the OH group and the water molecules, which causes more hindering of chains moving, raising the Tg. Because of the presence of OH interaction groups, the resulting sample is miscible mixtures, as shown by the

presence of only one Tg and only one Tm. While Tm drops from 222 to 212 degrees Celsius after adding starch to the blend, the fall in Tm was attributed to a decrease in the cohesive forces of attraction between the polymer chains, which agrees with Pokhrel 2019 [22].

Figure 6c shows that the resulting blend of PVA and CSM is immiscible, as evidenced by the presence of two Tg values, the first of which is around 40 oC and the second of which is around 85 oC. This could be attributed to structural differences between PVA and CSM, which result in an immiscible blend between them. We also saw the presence of CSM as a plasticizer for PVA, as evidenced by a decrease in the Tg of the PVA+CSM mix from 85. 78 o C to 83. 78 o C, as well as a dip in the onest peak from 85 o C to 74 o C. this is agrees with [23].

On the other hand, a mixture of three substances, including PVA+STA+CSM, results in a miscible blend, as shown in fig 6d. We can see that there is only one Tg about 89.48 o C, and only one Tm around 217 o C, indicating that the presence of STA between the PVA and CSM makes them more compatible, resulting in more blend miscibility via OH group interactions. These findings are crucial in explaining the behavior of viscosity, surface tensions, and concentrations in blend samples that correspond to RH095. [24]

Table 6 DSC results of blend samples

Sample	T_{g1} ^{o}C	T_{g2} ^{o}C	T_{m} ^{o}C	Type of mixture
PVA	85.78	-	222	Pure
PVA+STA	89.75	-	212	Miscible
PVA+ CSM	40	83.84	219	Immiscible
PVA+CSM+STA	89.48	-	217	Miscible

4. Conclusions

CSM was employed as a new ionic hydrocolloid to adjust solution viscosity for improving nanofiber properties and making the electrospinning process easier, as well as natural polymers such (STA) to examine biodegradability and biocompatibility nanofiber. The synthetic polymer PVA (polyvinyl alcohol) is used to improve spin ability. Different volume ratios were constructed, and the best volume ratio was established by looking at the contact angle that was the highest. According to FESEM, for these optimum combinations, the average fiber diameter should be lowered from (229-86 nm) with smooth fiber and a lower proportion of beads. Meanwhile, FTIR research demonstrated that all functional groups in blends have strong hydrogen bonds, and the electrospun nanofibers that arise can be used.

5. Conflicts of interest

There are no conflicts to declare

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