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Green and Sustainable Production of Edible Pullulan/Whey Nanofibers Loaded With Black Cumin (*Nigella sativa*) Waste Extract

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ABSTRACT

In this study, the electrospinning method was used to create green synthesis sustainable pullulan and whey protein nanofibers loaded with black cumin waste extract. FE-SEM imaging, FTIR, XRD, DSC, total phenolic content determination, total antioxidant activity determination, and antibacterial activity assessment were used to characterize the nanofibers. The diameter of the obtained nanofibers ranged from 240.7 to 600.4 nm. It was found that the antioxidant activity of the nanofibers containing black cumin waste extract varied between 273.59 and 437.90 $\mu\text{mol TE/g}$ dry matter. While the nanofibers containing black cumin waste extract exhibited antioxidant activity, those without the extract did not exhibit antioxidant activity. The total phenolic content of the nanofibers was determined to be between 4.93 and 31.52 mg GAE/g dry matter. The 5% BWE PN (100:0) nanofiber containing black cumin waste extract demonstrated antibacterial activity against *Staphylococcus aureus* with a zone diameter of 11.23 mm. The study results indicate that the green synthesis nanofibers produced with black cumin waste extract exhibit antioxidant and antibacterial properties, providing them appropriate for food coating and packaging applications. Additionally, in terms of sustainability and waste valorization, they are regarded as a promising nanomaterial for future applications.

1 | Introduction

Nanoscience and nanotechnology involve the production and examination of materials at the atomic, molecular, and macromolecular levels. Because they have a larger surface area per unit mass, biological components become more active than larger components with the same chemical structure [1–4]. This technology utilizes the distinct properties of objects operating within a size range of 1–1000 nm, corresponding to the scale of various biological structures, including antigens, receptors, subcellular components of the immune system, and microorganisms [5]. Because of their high surface-to-volume ratios, nanomaterials enhance the properties of the host materials in which they are

incorporated. Cosmetics, food, textiles, pharmaceuticals, electronics, and computer equipment are among the leading sectors that benefit from the properties of nanomaterials [6].

Nanotechnology enables the use of materials and devices more rapidly, on a smaller scale, with greater power, efficiency, and versatility. We anticipate more effective, specific, and flexible applications, and more customized, versatile, and cost-effective devices and drugs derived from nanotechnology [7].

Electrospinning is one of the nanotechnological methods that can produce nanostructures with rich content for many biomedical applications [8]. Electrospinning technology facilitates the

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preparation of environmentally sustainable biopolymers from natural materials and supports the advancement of functional packaging solutions [9]. To produce nanofibers through electrospinning, you require a suitable polymer solution, an electrode in contact with the polymer solution, a high-voltage direct current power source, and a grounded collector plate [10]. Electrospinning also has emerged as an effective method for encapsulating and stabilizing bioactive compounds, owing to its capacity to generate nanofibrous structures that provide a high surface area and protective polymeric matrices. Nanofibers have the capability to encapsulate sensitive bioactives, including phenolics, flavonoids, and essential oils, thus providing protection against environmental factors such as heat, light, oxygen, and humidity [11, 12]. Electrospun nanofibers facilitate the controlled release of encapsulated actives, proving advantageous for applications in active packaging and nutraceutical delivery systems [13].

Black cumin (*Nigella sativa* L.) is a spice traditionally used in medicine by many Asian, Middle Eastern, and Far Eastern countries [14]. Black cumin mainly contains fixed and volatile oils, carbohydrates, proteins, alkaloids, vitamins, and minerals [15]. Black cumin oil contains numerous important bioactive compounds. These include thymoquinone (30%–48%), thymohydroquinone, dithymoquinone, cymene (7%–15%), carvacrol (6%–12%), 4-terpineol (2%–7%), t-anethole (1%–4%), pinene, and thymol. Thymoquinone (TQ) is recognized as the most significant aromatic component of black cumin oil among all bioactive compounds [16]. Black cumin is known for its analgesic and anti-inflammatory effects [17, 18], anticancer properties [19, 20], antibacterial activity [20, 21], antifungal effects [20, 22], and antioxidant capacity [23, 24]. Electrospinning is known to load black cumin oil or extract into polymeric nanofibers [15].

Seeds of oil crops, especially those with a high content of polyunsaturated fatty acids, are considered to be abundant in antioxidants [25]. Oilseed cakes are byproducts of oilseed extraction, characterized by their high content of proteins, carbohydrates, mineral substances, fiber, and some lipids. From an economic perspective, oilseed cakes appear to be neglected despite their potential as nutraceuticals and functional food ingredients, aligning with current market trends that seek products rich in bioactive compounds, particularly antioxidants, omega-3 fatty acids, and dietary fiber [24]. Black cumin waste, a by-product, is also a source of valuable bioactive compounds, such as hydroxybenzoic, syringic, and p-coumaric acids. Additionally, the production of edible films can successfully utilize black cumin waste, a by-product rich in protein [26]. A research comparing the antioxidant levels in flaxseed, evening primrose, and black cumin seed (*Nigella sativa*) waste revealed that the extract from black seed waste exhibited the second highest antioxidant content of the three [24]. In a separate investigation, black cumin seed waste extract was included in yogurt, and after 14 days of storage, all yogurts containing the extract exhibited greater inhibition activity than those lacking the extract [23]. An additional study employing several extraction techniques confirmed the existence of antioxidant activity in black cumin waste extract [27]. Current knowledge suggests that black cumin waste may serve as an economical antioxidant in biopolymer blends due to its abundance of beneficial bioactive components and health advantages [26].

Because they are biologically compatible, biodegradable, and customizable to achieve desired properties, natural and modified polysaccharides serve as advantageous materials for the encapsulation of bioactive components. Because their delivery systems are based on carbohydrates, they can bind and encapsulate both hydrophilic and hydrophobic bioactive components. This gives them a wide range of carrier properties. They also have higher thermal stability compared to protein- and lipid-based delivery systems [28]. Electrospinning frequently uses cellulose [29, 30], alginate [31, 32], chitosan [33, 34], pullulan [35, 36], starch [37, 38], and dextran [39]. Among these polysaccharides, pullulan is a commercially obtained microbial, edible, linear exopolysaccharide derived from fungi. Food packaging materials widely use this water-soluble biopolymer with simple film-forming properties [6]. Pullulan and its derivatives serve not only in food packaging and coating but also as binders, encapsulating agents, fat substitutes, and thickeners, among other applications [40].

Proteins are the main components of the human body, have valuable nutritional content, and serve as functional food additives, so we prefer them over synthetic polymers for the encapsulation of bioactive components. However, due to their complex secondary and tertiary structures, it is quite challenging to obtain nanofibers solely from proteins via the electrospinning method. Globular proteins have very few internal interactions to entangle and mix during the spinning process [28]. It was suggested that using the appropriate solvent, adding denaturing agents to the polymer solution, or heating the surface can simplify protein electrospinning [41]. Another method for enabling protein electrospinning is to mix them with a carrier polymer with high electrospinning capacity [42]. Proteins like gelatin [43, 44], collagen [45, 46], elastin [47, 48], casein [49, 50], amaranth [51, 52], and zein [53, 54] can be given as examples of proteins used during electrospinning. Whey protein isolate, recognized for its emulsifying properties, serves as an effective carrier for encapsulating bioactive components. Whey protein isolate is a beneficial way to control the release of bioactive components because it does not react with stomach enzymes and is easier to digest in the small intestine [55].

This research examined the production of sustainable, bio-based food packaging through the production of edible nanofibers derived from black cumin waste via electrospinning techniques. The study integrated black cumin extract into pullulan and pullulan/whey matrices to preserve bioactive compounds and enhance waste valorization. A comprehensive characterization was performed, including morphological, chemical, thermal, antioxidant, and antibacterial analyses, to assess the functional properties of the nanofibers and their applicability in active packaging.

2 | Materials and Methods

2.1 | Materials

Pullulan was donated by Hayashibara Co. Ltd., while whey protein concentrate was donated by Malkara Birlik Süt ve Süt Mamulleri A.Ş. We obtained black cumin waste from a Bursa-based company that uses the cold-press method to produce black cumin oil. Ethanol ($\geq 99\%$) was supplied by Isolab, Folin reagent (2N) by Merck, Na_2CO_3 by Sigma-Aldrich, 2,2-diphenyl-1-picrylhydrazyl (DPPH) by Sigma-Aldrich, 6-hydroxy-2,5,7,8-tetramethylchroma

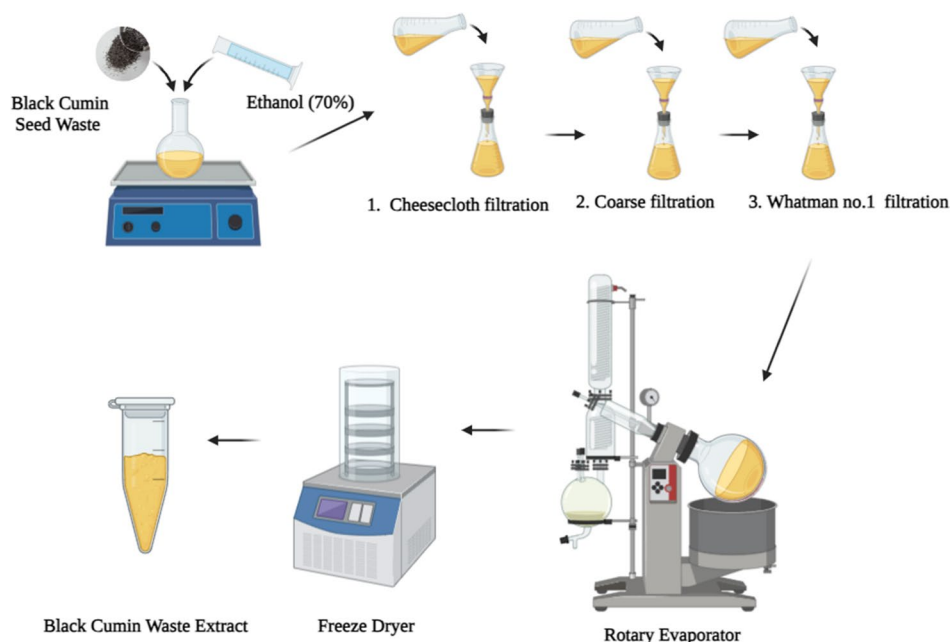


FIGURE 1 | Preparation stages of black cumin waste extract. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

n-2-carboxylic acid (Trolox) by Sigma-Aldrich, methanol ($\geq 99\%$) by Carlo Erba, gallic acid by Carl Roth GmbH + Co. KG, nutrient broth by Merck, Luria broth by Himedia, TSB broth by Merck, and agar-agar by Sigma-Aldrich.

2.2 | Methods

2.2.1 | Production of Black Cumin Waste Extract

Black cumin waste samples were ground using a laboratory-type grinder (Lavion HC-100, Turkey). The ground samples were then sieved through a 500-mesh sieve and vacuum-packed. The packed black cumin waste samples were stored in a freezer at -18°C (Uğur, UED 7266) until use. 500 g of ground sample powder was extracted with 2000 mL of 70% (v/v) ethanol by stirring on a magnetic stirrer for 6 h. The resulting extract was sequentially filtered through cheesecloth, coarse filter paper, and finally Whatman No. 1 filter paper. The remaining solid residue was re-extracted with 1000 mL of 70% ethanol by shaking on a laboratory shaker (Wiseshake SHO-1D, Korea) for an additional 6 h. This second extract was filtered following the same procedure. The combined filtrates were concentrated at 50°C using a rotary evaporator (Büchi R-114, Switzerland). The concentrated extract was then frozen at -80°C for 24 h and subsequently lyophilized at -50°C using a freeze dryer (Thermo Savant Modulyo, USA). The obtained extracts were stored in a deep freezer at -18°C (Uğur, UED 7266) until use [56]. The preparation steps of the black cumin waste extract are shown in Figure 1.

2.2.2 | Production of the Feed Solution Consisting Black Cumin Waste Extract

Polymer solutions (20% w/w) were formulated with pure water to include pullulan and whey protein in the ratios of 100:0 (w/w), 50:50 (w/w), and 25:75. The mixture was agitated using

a magnetic stirrer (Daihan MSH-20A, Korea) at 800 rpm, after which black cumin waste extract (5% w/w) was included. The solution was agitated until it achieved homogeneity. The homogeneous solution was maintained at room temperature for 24 h to eliminate air bubbles. The control sample solutions were made using the identical manner [35]. The viscosity (Brookfield DV-II Viscometer, USA) and electrical conductivity (Jenco-3177, USA) measurements of the resulting solutions were performed.

2.2.3 | Electrospinning Process

The electrospinning apparatus employed in this study includes a blunt-tip needle with a diameter of 0.8 mm, an aluminum collector, a syringe pump, and a power supply (Matsusada, 40 kV) (Figure 2). Nanofiber production was carried out using the prepared feed solutions at a power of 15 kV, with a distance of 15–20 cm between the needle tip and the collector and flow rates varying between 0.2 and 0.6 mL/h. The nanofibers obtained under these conditions were collected on parchment paper. The conditions for the produced nanofibers are provided in Table 1.

2.2.4 | Morphology Analysis

The surface morphology of the nanofibers was examined using a Zeiss Supra 40 VP model scanning electron microscope (SEM) equipped with an Energy Dispersive Spectroscopy (EDS) unit. Prior to obtaining SEM images, a thin conductive layer was applied to the samples using the sputter coating technique. This conductive layer, composed of 80% gold and 20% palladium, was implemented to improve image clarity while preserving the integrity of the nanofibers. The imaging of the conductivity-enhanced fibers was performed under a vacuum of 10^{-6} torr and an acceleration voltage of 10 kV. The average diameter of the nanofibers was measured using the Image J software, based on 100 randomly

selected nanofibers. The homogeneity coefficient of the fibers was calculated using Equations (1) and (2). Histograms of the nanofibers were generated using the Origin Pro 9.0 software.

$$A_n = \frac{\sum n_i d_i}{\sum n_i} \text{ (Number Average)} \quad (1)$$

$$A_w = \frac{\sum n_i d_i^2}{\sum n_i d_i} \text{ (Weight Average)} \quad (2)$$

Here, d_i represents the nanofiber diameter, while n_i represents the number of nanofibers. The $\frac{A_w}{A_n}$ ratio gives the fiber homogeneity coefficient [57].

2.2.5 | The Fourier Transform Infrared Spectroscopy

Fourier Transform Infrared Spectroscopy (FTIR) analysis was conducted to determine the chemical composition of the nanofibers produced using black cumin waste extract, pullulan, and whey protein under the conditions listed in Table 1. This analysis was performed using the ATR technique with a Thermo Fisher

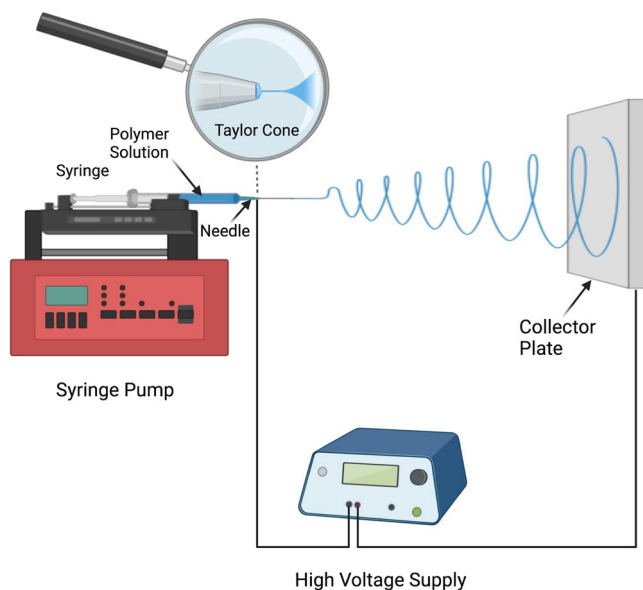


FIGURE 2 | Laboratory-sized electrospinning unit [34]. [Color figure can be viewed at wileyonlinelibrary.com]

Scientific Nicolet ISFT-IR model FTIR spectrophotometer, in the wavelength range of 4000–400 cm^{-1} and with a spectral resolution of 4 cm^{-1} . At least 16 scans were performed for each spectrum.

2.2.6 | X-Ray Diffraction

X-ray diffraction (XRD) analyses were conducted at room temperature to determine the crystallinity of the nanofibers produced using black cumin waste extract, pullulan, and whey protein under the conditions listed in Table 1. The analyses were performed using a GNR APD PRO 2000 model x-ray diffractometer with Cu $K\alpha$ (1.5406 Å) radiation as the x-ray source. The XRD device was operated at 40 kV and 30 mA for the measurements. For nanofiber samples, the scanning range was set between $2\theta = 10^\circ$ – 70° , and for powdered samples, between $2\theta = 5^\circ$ – 90° with a step size of 0.1° and an integration time of 2 s per angle value.

2.2.7 | Differential Scanning Calorimeter (DSC) Analysis

Differential Scanning Calorimetry (DSC) analysis was conducted to assess the thermal properties of the nanofibers using a Netzsch Synchronous Thermal Analyzer (STA 449 F5 Jupiter) device, manufactured in Germany. In this analysis, samples ranging from 2 to 3 mg were heated from 20°C to 600°C at a rate of $5^\circ\text{C}/\text{min}$ under a nitrogen gas atmosphere. The melting point (T_m) and degradation temperature (T_d) of the nanofiber samples were determined based on the endothermic and exothermic curves observed in the DSC diagram.

2.2.8 | Total Phenolic Content and Total Antioxidant Activity

Before determining the total phenolic content and total antioxidant activity, the nanofiber samples were dried in a drying oven (Memmert UN 30 plus, Germany) at 40°C for a duration of 2 h. 0.1 g of dried nanofiber samples and 1 g of solution samples were weighed, to which 10 mL of distilled water was added. The fiber and solution samples were stirred on a magnetic stirrer at 800 rpm until completely dissolved. Afterwards, the extracts to be used for the total phenolic content and antioxidant activity determination were prepared by centrifuging at 9000 rpm for 15 min at 4°C .

TABLE 1 | The process conditions for electrospinning of feed solutions.

Sample code	Black cumin waste extract content (% w/w)	Pullulan:whey protein isolate ratio (w/w)	Applied voltage (kV)	Tip to collector distance (cm)	Flow rate (mL/h)
PN (100:0)	0	100:0	15	15	0.2
5% BWE PN (100:0)	5	100:0	15	15	0.3
P/WN (50:50)	0	50:50	15	20	0.2
5% BWE P/WN (50:50)	5	50:50	15	15	0.2
P/WN (25:75)	0	25:75	15	15	0.6
5% BWE P/WN (25:75)	5	25:75	15	15	0.5

TABLE 2 | Some properties of feed solutions and nanofibers.

Sample code	Feed solution properties		Nanofiber properties		
	Viscosity (Pa s)	Conductivity ($\mu\text{S cm}^{-1}$)	Nanofiber diameter (nm)	Nanofiber uniformity coefficient	Nanofiber morphology
PN (100:0)	2.88 ± 0.39^a	60.65 ± 0.17^a	536.8 ± 36.9^a	1.005	Smooth
5% BWE PN (100:0)	3.50 ± 0.69^b	242.0 ± 1.15^b	600.4 ± 76.4^b	1.016	Smooth
P/WN (50:50)	0.30 ± 0.01^c	2090 ± 33.16^c	240.7 ± 31.7^c	1.017	Smooth
5% BWE P/WN (50:50)	0.28 ± 0.01^c	2037.0 ± 15.00^d	257.7 ± 42.4^d	1.025	Smooth
P/WN (25:75)	0.05 ± 0.01^c	2835 ± 23.80^e	Not calculated	—	Beads
5% BWE P/WN (25:75)	0.06 ± 0.01^c	3120 ± 17.30^f	Not calculated	—	Beads

Note: Different letters indicate significant differences between means in columns for each data at $p < 0.05$ level.

The total amount of phenolic compounds was determined through modifications to the Folin–Ciocalteu method, with the results expressed as mg GAE/g of dry sample in terms of gallic acid equivalent [58]. Antioxidant activity determination was performed using the DPPH method based on the capacity to reduce the DPPH (2,2-diphenyl-1-picrylhydrazyl) free radical, with the results expressed as mmol TE/g of dry sample in terms of trolox equivalent [59].

2.2.9 | Antibacterial Activity

The antibacterial activity analysis of the nanofibers was performed using a modified version of the disk diffusion method [60]. Antibacterial activity was tested against the indicator microorganisms *Micrococcus luteus* NCIMB and *Staphylococcus aureus* ATCC29113. LB agar medium was used for *M. luteus* and Nutrient agar medium for *S. aureus*. Before placing the nanofiber samples onto the Petri dishes, they were sterilized under UV light for 30 min on each side, for a total of 1 h per sample. Nanofibers with a diameter of 5 mm and a weight of 20 mg were then placed onto the Petri dishes. The dishes were incubated at 37°C for 24 h. The zone diameters were measured using the Image J software.

2.2.10 | Statistical Analysis

The experimental data were analyzed using IBM SPSS 23 software. The multiple Duncan test was applied to determine the differences between total phenolic content, total antioxidant activity, rheological properties, electrical conductivity, and fiber diameters ($p < 0.05$). All analyses were performed in triplicate and with two parallels, and the results are expressed as mean $\pm$ standard error (SE).

3 | Results and Discussion

3.1 | Feed Solution Properties

The apparent viscosity and electrical conductivity values of the feed solutions are presented in Table 2. It was determined that the viscosity values of the feed solutions ranged between 0.05 and 3.50 Pa s, with the highest viscosity observed in the

5% BWE PN (100:0) solution. It was found that the addition of whey protein to the feed solutions reduced their viscosity ($p < 0.05$). The results of the research where nanofibers were obtained using pullulan and whey protein support our findings [61]. An increase in viscosity was observed in the 5% BWE PN (100:0) solution, which was obtained by adding 5% (w/w) black cumin waste extract to the PN (100:0) solution, compared to the PN (100:0) solution ($p < 0.05$). During the electrospinning process, very low viscosity values limit the formation and continuity of nanofibers, while very high viscosity values make it difficult to discharge the polymer solution from the syringe [62].

The electrical conductivity values of the feed solutions used in our study ranged between 61 and 3120 $\mu\text{S cm}^{-1}$ (Table 2). Electrical conductivity is another parameter that affects nanofiber formation. Homogeneous and continuous nanofibers can be obtained when the electrical conductivity value of the polymer solution is high enough to overcome the electric field force created by the voltage applied by the electrospinning setup. Therefore, solutions without electrical conductivity cannot be used in nanofiber production [62]. It was indicated that the addition of whey protein and extract to the solutions led to an increase in electrical conductivity values ($p < 0.05$). This is one of the reasons for the decrease in the diameter of the nanofibers obtained from the solutions. It was indicated that an increase in solution viscosity typically leads to a rise in fiber diameter, whereas an increase in electrical conductivity coefficients tends to decrease fiber diameter [61].

3.2 | Morphology of Electrospun Nanofiber

The properties of the nanofibers produced in our study (nanofiber diameter, homogeneity coefficient, and nanofiber morphology) are presented in Table 2, while their FESEM images and histograms are shown in Figure 3. The diameters of the obtained nanofibers ranged from 240.7 to 600.4 nm, and their homogeneity coefficients ranged from 1.005 to 1.025. It was determined that the nanofiber diameter of the PN (100:0) nanofiber was 536.8 nm, and its homogeneity coefficient was 1.005. The nanofiber diameter of the 5% BWE PN (100:0) nanofiber, which was obtained by adding 5% (w/w) black cumin waste extract to

the PN (100:0) feed solution, was found to be 600.4 nm, and its homogeneity coefficient was 1.016. This increase in nanofiber diameter due to the addition of black cumin waste extract was thought to be the combined effect of the increase in viscosity of the feed solution and the increased flow rate during nanofiber production. In a study on polyethylene oxide nanofibers, it was observed that the diameters of the nanofibers increased as viscosity rose [63]. Similarly, when producing cyclodextrin nanofibers, thicker fibers were obtained at higher flow rates, while other parameters remained constant [64].

In our study, nanofibers were also produced under different conditions from solutions prepared using pullulan and whey protein in 50:50 w/w and 25:75 w/w ratios. The properties of the nanofibers obtained with the inclusion of whey protein into the solutions (nanofiber diameter, homogeneity coefficient, and nanofiber morphology) were changed (Table 2, Figure 3). The nanofiber diameter of the P/WN (50:50) nanofiber was 240.7 nm, while the diameter of the 5% BWE P/WN (50:50) nanofiber, which included 5% (w/w) black cumin waste extract, was 257.7 nm. The homogeneity coefficients of these fibers were determined to be 1.017 and 1.025, respectively. The incorporation of whey protein into the solutions led to reduced viscosity values and enhanced electrical conductivity values when compared to polymer solutions containing only pullulan. Therefore, nanofibers produced using pullulan: whey protein (50:50 w/w) had smaller diameters compared to nanofibers without whey protein in their structure ($p < 0.05$). In a study on polylactic acid, it was stated that a decrease in polymer solution viscosity and an increase in electrical conductivity led to the formation of thinner nanofibers [65]. In another study on polyvinyl alcohol and pectin blends, it was noted that an increase in the electrical conductivity of the polymer solution resulted in a reduction in fiber diameter [66].

As the value of the homogeneity coefficient approaches 1, the homogeneity of the nanofibers is observed to increase [57, 67]. In this case, it can be stated that the most homogeneous nanofiber was the PN (100:0) nanofiber, while the least homogeneous was the 5% BWE P/WN (50:50) nanofiber. Beaded nanofibers were obtained from the solutions prepared using pullulan and whey protein in a 25:75 w/w (pullulan:whey protein) ratio. It was determined that the homogeneity coefficient increased when whey protein was added to the structure of the fibers, but when the whey protein content exceeded a certain ratio, defects in the fiber morphology occurred (Figure 3). The reason for this is that whey protein, like all other proteins, has a globular structure and lacks sufficient entanglements and interchain bonds. Therefore, it is not possible to use aqueous solutions of whey protein alone in the electrospinning process. To achieve an optimal electrospinning process and obtain nanofibers with a more regular morphology, the polymer solution should be supplemented with an appropriate carbohydrate polymer [68].

3.3 | FTIR Results

When the FTIR spectra of the black cumin waste extract were examined, characteristic bands belonging to the black cumin extract were observed at 3298, 2925, 1653, 1601, 1515, 1374, 1263, 992, 923, 876, and 548 cm^{-1} (Figure 4). The spectra of black

cumin were consistent with the studies conducted which utilized black cumin oil [69, 70]. The peak at 3298 cm^{-1} represents the symmetric C—H stretching vibration of cis-olefinic double bonds. The sharp peak at 2925 cm^{-1} corresponds to the asymmetric stretching vibration of the C—H bonds in the aliphatic CH_2 triglyceride groups. The peak at 1651 cm^{-1} is related to the C=C stretching vibration of cis-olefins. The peak at 1371 cm^{-1} represents the symmetric bending vibration of the C—H bonds in the CH_2 groups of triglycerides [69].

When the FTIR spectra of pullulan polymer were examined, characteristic bands of pullulan were observed at 3319, 2925, 1653, 1418, 1339, 1146, 1076, 927, 848, 753, 567, and 510 cm^{-1} (Figure 4). The vibrations observed at 848 and 927 cm^{-1} correspond to the α -glucopyranosyl units and α -1-6 glycosidic bonds, respectively, while the vibrations between 1200 and 1030 cm^{-1} represent the C—O bonds. The adsorption band at 1146 cm^{-1} indicates the 1-4 glycosidic bond, the peak at 3319 cm^{-1} indicates the O—H vibration, whereas the vibration around 2925 cm^{-1} reflects the C—H bond structure. The obtained spectra were in agreement with the literature [68, 71–73].

As seen in Figure 4, the characteristic bands of whey protein were observed at 3273, 2925, 1629, 1521, 1447, 1394, 1311, 1238, 1153, 1072, and 514 cm^{-1} . The peak at 3273 cm^{-1} in the whey protein spectrum corresponds to the N—H stretching band, whereas the peaks at 1629 and 1238 cm^{-1} are associated with the Amide I and Amide II vibrations, respectively, which are definitive markers of whey protein. The Amide I vibration originates from the C=O stretching vibration and is used as an indicator in infrared spectroscopy to detect the secondary structure of proteins. The Amide II vibration is expressed as a combination of NH bending and CN stretching vibrations. The spectra of whey protein were also consistent with the literature [68, 74, 75].

It is imperative to assess whether encapsulation is successful following the electrospinning process by determining whether it is based on physical or chemical entrapment. In the event of physical entrapment, the FTIR spectra of the primary components, including the polymer and extract, exhibit no or minimal alterations in terms of characteristic peaks. Changes in the characteristic peaks of the FTIR spectra are observed when a chemical interaction occurs between the extract and the polymers used [76]. In the FTIR spectra of the nanofibers obtained in our research, characteristic peaks belonging to pullulan, whey protein, and black cumin waste were observed. This indicates that the black cumin waste extract in the structure of the obtained nanofibers was effectively encapsulated by the biopolymers without any negative interaction.

3.4 | XRD Results

The x-ray diffraction (XRD) profiles of black cumin waste extract, pullulan polymer, and whey protein polymer are shown in Figure 5, while the XRD profiles of the nanofibers obtained from the extract and polymers are presented in Figure 6.

In the XRD profile shown in Figure 5, the broad peaks observed at 8° and 20° for the black cumin waste extract indicated that the extract has an amorphous structure [77, 78]. When examining

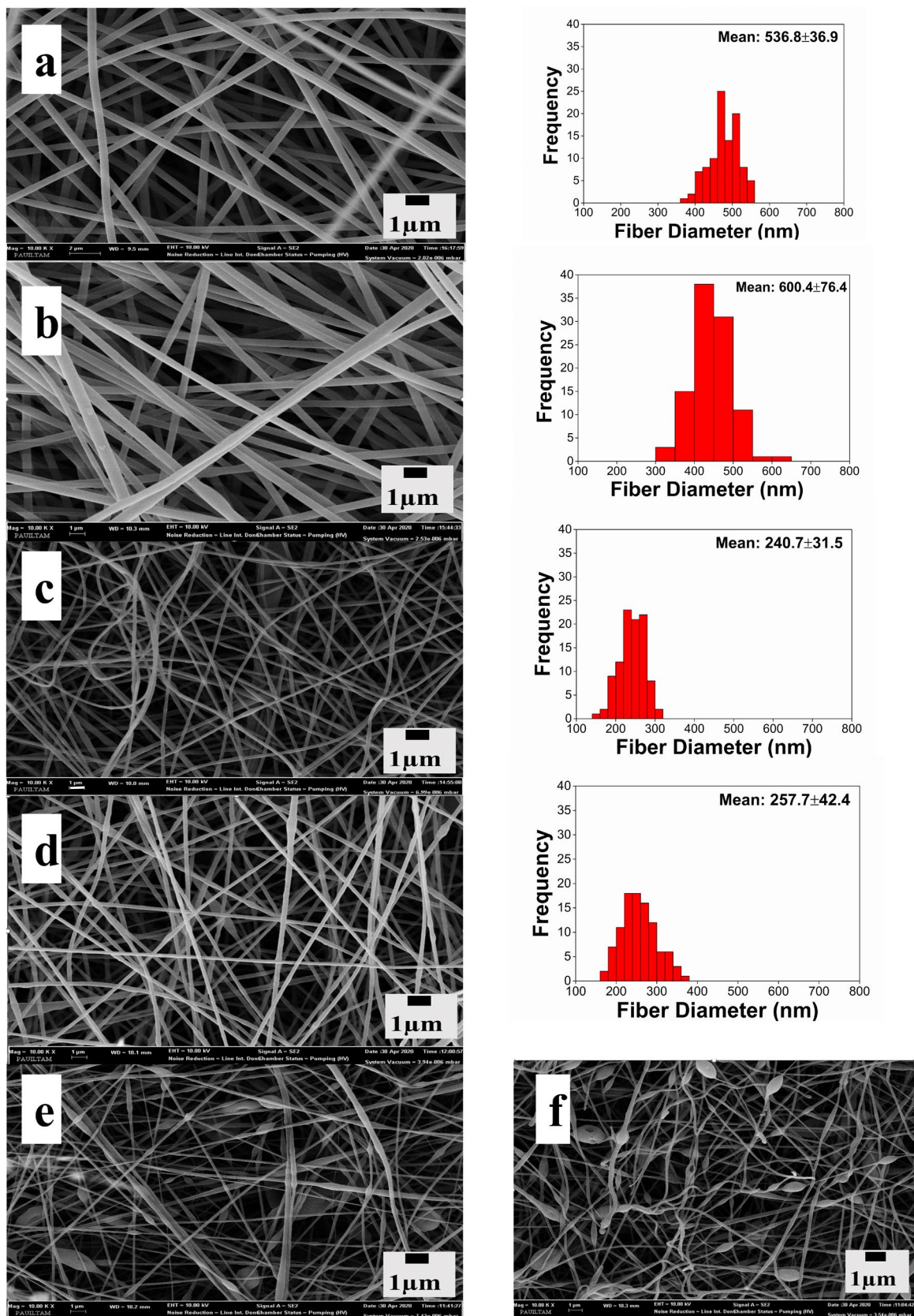


FIGURE 3 | FE-SEM images (10,000 $\times$) and fiber diameter distributions with an average fiber diameter of nanofibers (a) PN (100:0), (b) 5% BWE PN (100:0), (c) P/WN (50:50), (d) 5% BWE P/WN (50:50) and FE-SEM images (10,000 $\times$) of nanofibers, (e) P/WN (25:75), and (f) 5% BWE P/WN (25:75). [Color figure can be viewed at wileyonlinelibrary.com]

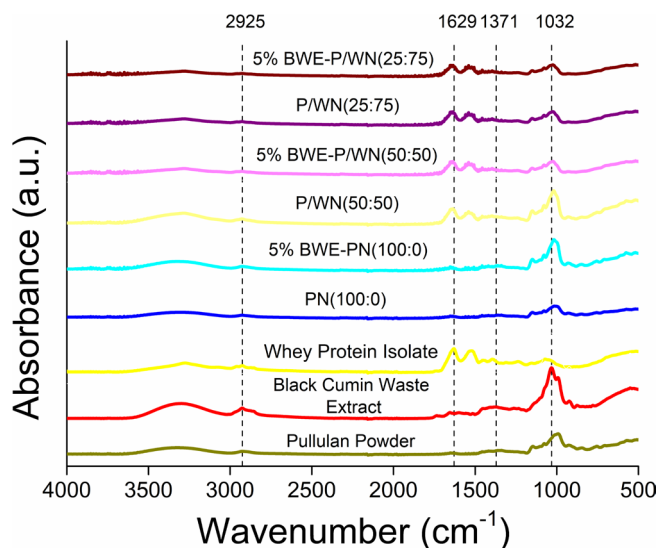


FIGURE 4 | FTIR spectra of pullulan, whey, black cumin waste extract, and nanofibers; PN (100:0), 5% BWE PN (100:0), P/WN (50:50), 5% BWE P/WN (50:50), P/WN (25:75), and 5% BWE P/WN (25:75). [Color figure can be viewed at wileyonlinelibrary.com]

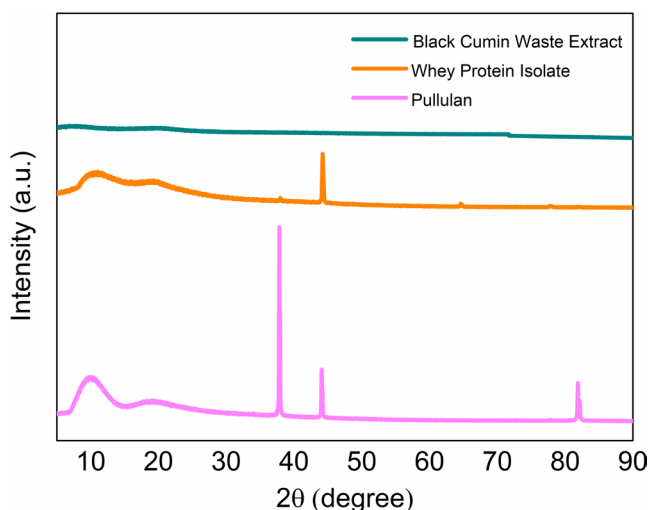


FIGURE 5 | X-ray diffraction profiles of pullulan powder, whey, and black cumin waste extract. [Color figure can be viewed at wileyonlinelibrary.com]

the XRD profile of the pullulan polymer, it exhibited a strong and sharp peak at 37.8° and a weak but sharp peak at 44° (Figure 5). The sharp peaks in the XRD profile of pullulan indicated that the pullulan polymer has a crystalline structure [79]. As for the XRD profiles of whey protein, a weak and broad peak was observed at 11° , while a sharp and strong peak was seen at 44° (Figure 5). Therefore, it can be said that whey protein also has a crystalline structure [80].

The nanofibers produced with pullulan and whey protein polymers, both with and without black cumin waste extract, exhibited broad but weak peaks at roughly 16° and broad but strong peaks at 23° . It was determined that the nanofibers obtained with or without extract using pullulan and whey protein have an amorphous structure. As can be inferred from the results, the crystalline structure of the polymers transformed

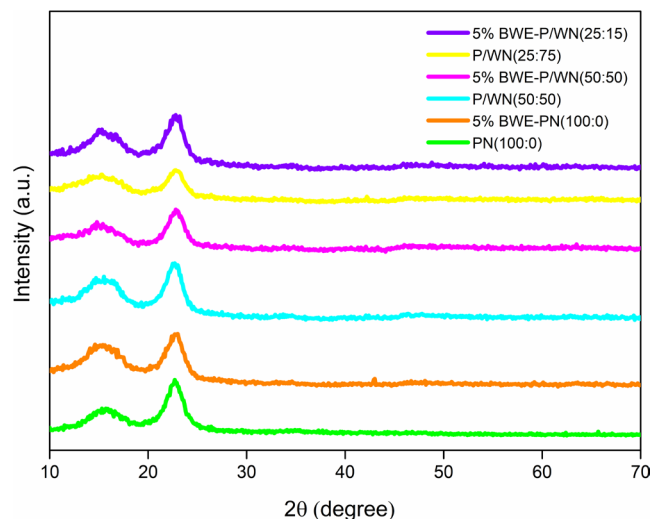


FIGURE 6 | X-ray diffraction profiles of nanofibers; PN (100:0), 5% BWE PN (100:0), P/WN (50:50), 5% BWE P/WN (50:50), P/WN (25:75), and 5% BWE P/WN (25:75). [Color figure can be viewed at wileyonlinelibrary.com]

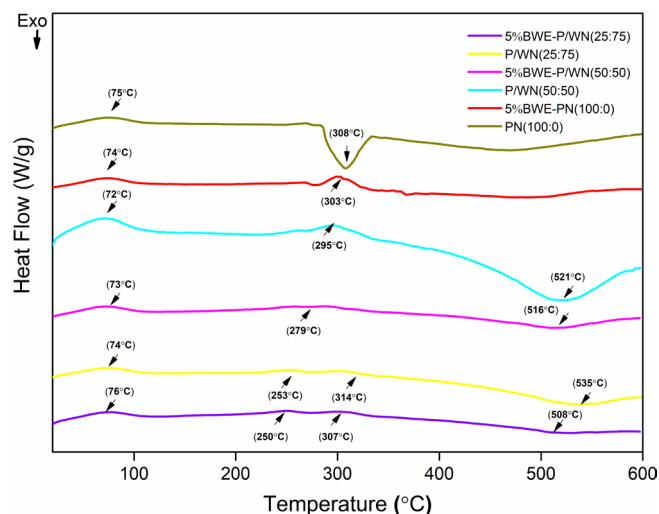


FIGURE 7 | DSC diagram of nanofibers; PN (100:0), 5% BWE PN (100:0), P/WN (50:50), 5% BWE P/WN (50:50), P/WN (25:75), and 5% BWE P/WN (25:75). [Color figure can be viewed at wileyonlinelibrary.com]

into an amorphous structure during the electrospinning process [72, 81, 82].

3.5 | Differential Scanning Calorimetry (DSC) Analyzes

In Figure 7, the DSC diagram of nanofibers produced using pullulan and whey protein polymers, with and without black cumin waste extract, is presented. All nanofibers exhibited an endothermic peak between 72°C and 76°C . This peak can be attributed to the evaporation of water present in the nanofibers [83, 84].

According to the DSC diagram shown in Figure 7, the PN (100:0) nanofiber started melting at 75°C and decomposed at 308.1°C .

TABLE 3 | The total phenolic content and the total antioxidant activity of feed solution and nanofibers.

Sample code	Processing step	Total phenolic content (mgGAE/g dm)	Total antioxidant activity ($\mu\text{mol TE/g dm}$)
PN (100:0)	Solution	0.62 ± 0.05^i	—
	Nanofiber	4.93 ± 0.27^h	—
5% BWE PN (100:0)	Solution	5.60 ± 0.60^{gh}	363.52 ± 4.90^d
	Nanofiber	6.71 ± 0.85^g	395.55 ± 19.06^c
P/WN (50:50)	Solution	11.21 ± 0.16^f	—
	Nanofiber	19.02 ± 0.50^e	—
5% BWE P/WN (50:50)	Solution	20.57 ± 0.20^d	465.67 ± 7.89^b
	Nanofiber	23.46 ± 0.31^c	437.90 ± 0.74^b
P/WN (25:75)	Solution	20.80 ± 0.08^d	—
	Nanofiber	24.36 ± 3.17^c	—
5% BWE P/WN (25:75)	Solution	25.95 ± 1.54^b	549.18 ± 13.42^a
	Nanofiber	31.52 ± 0.64^a	273.59 ± 8.37^e

Note: Different letter indicates significant difference between means in columns for each data at $p < 0.05$ level.

The decomposition temperature of pullulan nanofibers was reported to be 305.2°C [85]. The 5% BWE PN (100:0) nanofiber, containing 5% (w/w) black cumin waste extract, exhibited endothermic heat behavior (Figure 7). The results are consistent with the literature [86, 87]. According to the graph, the 5% BWE PN (100:0) nanofiber started melting at 73.5°C and decomposed at 302.6°C . The P/WN (50:50) nanofiber, produced from a 50:50 (w/w) mixture of pullulan and whey protein, started melting at 72°C and showed a second endothermic peak at 295°C . The final decomposition temperature of the P/WN (50:50) nanofiber was identified as 521.7°C . The 5% BWE P/WN (50:50) nanofiber started melting at 72.5°C and showed its second endothermic peak at 279.2°C (Figure 7). The third peak of the 5% BWE P/WN (50:50) nanofiber, showing exothermic heat behavior, occurred at 516.1°C . This temperature can be considered the final decomposition point of the nanofiber.

The P/WN (25:75) nanofiber, containing a 25:75 (w/w) mixture of pullulan and whey protein, started melting at 74°C and showed an endothermic peak at 253°C . Later, the P/WN (25:75) nanofiber exhibited a second endothermic peak at 314°C . The third peak, shown by the same nanofiber at 535°C , indicated final decomposition. The 5% BWE P/WN (25:75) nanofiber started melting at 76°C and showed its second and third peaks at 250°C and 307°C , respectively. The final decomposition peak was observed at 508°C .

It was reported that whey protein began to decompose around 270°C when examining the DSC diagrams of nanofibers produced from whey protein and rye flour [62]. It is thought that the peaks around 250°C in the DSC diagrams of nanofibers produced from mixtures of pullulan and whey protein at certain ratios indicate the decomposition of whey protein.

The melting and decomposition temperatures of nanofibers containing black cumin waste extract were lower compared to nanofibers without the extract. This suggests that the addition of

black cumin waste extract to the structure of nanofibers caused a plasticizing effect on the nanofibers [88, 89].

3.6 | Phenolic Content and Antioxidant Properties of Electrospun Nanofiber

Black cumin is a source of bioactive compounds important for antioxidant activity, such as thymoquinone, hydroxybenzoic acid, syringic acid, and p-coumaric acid. Considering this information, black cumin waste extract is a cheap antioxidant element for biopolymer blends because of its high-value bioactive components and nutritional value [26].

The total phenolic content (mgGAE/g dm) and total antioxidant activity ($\mu\text{mol TE/g dm}$) of the feed solutions and nanofibers are presented in Table 3. The phenolic content of the solution and nanofibers containing whey protein was found to be higher compared to those without whey protein (Table 3). It is believed that the total phenolic content of the solutions and nanofibers containing whey protein is due to amino acid residues such as isoleucine, leucine, lysine, methionine, phenylalanine, threonine, tryptophan, and valine. These amino acid residues are known to reduce the Folin-Ciocalteu reagent, which is used to determine total phenolic content [90].

The addition of black cumin waste extract to the solutions resulted in an increase in the phenolic content of all solutions (Table 3). The phenolic content of the nanofibers produced with black cumin waste extract was also higher than that of the nanofibers produced without the extract ($p < 0.05$) (Table 3). The total phenolic content of the 5% BWE PN (100:0) nanofiber, containing 5% (w/w) the extract from black cumin waste, did not exhibit any statistically significant difference when compared to the original solution from which it was derived ($p > 0.05$). This suggests that the electrospinning process effectively encapsulated the phenolic compounds present in the structure.

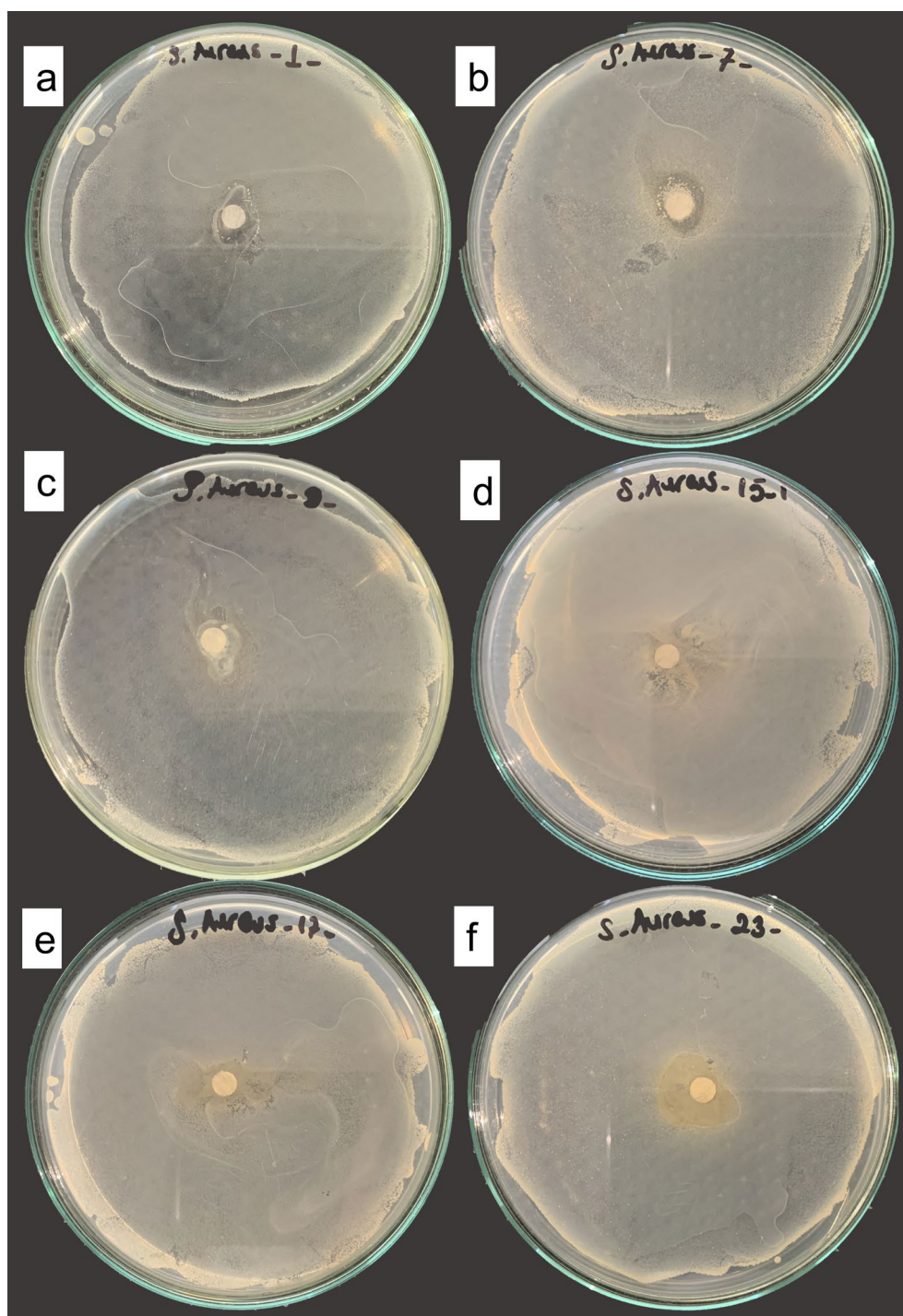


FIGURE 8 | Antibacterial activity of nanofibers; (a) PN (100:0), (b) 5% BWE PN (100:0), (c) P/WN (50:50), (d) 5% BWE P/WN (50:50), (e) P/WN (25:75), and (f) 5% BWE P/WN (25:75) against *S. aureus*. [Color figure can be viewed at wileyonlinelibrary.com]

However, it was observed that the total phenolic content of the 5% BWE P/WN (50:50) nanofiber increased by approximately 14% compared to the solution from which it was obtained (Table 3). Changing the conditions during nanofiber production causes changes in nanofiber morphology [91–94]. Changes in nanofiber morphology lead to changes in the properties of the nanofibers [95–97]. Therefore, the higher total phenolic content of the 5% BWE P/WN (50:50) nanofiber compared to the solution from which it was obtained is thought to be due to changes in nanofiber morphology.

As seen in Table 3, it was determined that nanofibers and polymer solutions without any extract had no antioxidant activity. All nanofibers produced with the addition of plant extracts exhibited antioxidant properties. The research involving the production of polyvinyl alcohol/gelatin nanofibers loaded with black cumin waste extract and zinc particles corroborates our findings [26]. According to Table 3, the 5% BWE PN (100:0) nanofiber, containing 5% (w/w) black cumin waste extract, exhibited 9% higher antioxidant activity compared to the solution from which it was obtained ($p < 0.05$). Nanofibers are superior materials with

a larger surface area-to-volume ratio, smaller pore sizes, and higher porosity [98]. When a material's structure is scaled down to the nanoscale, its properties can become much more effective. The higher antioxidant activity of the 5% BWE PN (100:0) nanofiber compared to its solution can be explained in this way. Similar results were also obtained in our previous study [35].

The antioxidant activity of the 5% BWE P/WN (50:50) nanofiber, produced from a 50:50 (w/w) pullulan: whey protein mixture with 5% (w/w) black cumin waste extract, did not exhibit a statistically significant difference compared to the solution from which it was obtained ($p > 0.05$). This suggests that the polyphenols present in the black cumin waste were effectively encapsulated through the electrospinning process. The study that nanofibers produced from PVA and PVA/ β -CD with grape seed extract supports our results [99].

However, the 5% BWE P/WN (25:75) nanofiber exhibited 50% lower antioxidant activity compared to the solution from which it was obtained ($p < 0.05$). As shown in Table 2 and Figure 3, the morphology of this nanofiber is significantly degraded. The dramatic decrease in antioxidant activity could be the result of the damaged nanofiber morphology. The previous study reported that the degradation in the morphology of polylactic acid nanofibers containing tea polyphenols led to reduced polyphenol release, negatively impacting antioxidant activity [100].

3.7 | Antibacterial Activity of Electrospun Nanofiber

The antimicrobial activity of nanofiber samples against *Staphylococcus aureus* and *Micrococcus luteus* is shown in

Figures 8 and 10. Nanofiber samples produced without loading the extract (negative control) did not exhibit antimicrobial activity against *S. aureus* and *M. luteus*. Among the nanofibers produced by adding 5% (w/w) black cumin waste extract to the polymer solution, only the 5% BWE PN (100:0) nanofiber exhibited antibacterial activity against *S. aureus* with a zone diameter of 11.23 mm (Figure 8). The antibacterial activity of the 5% BWE PN (100:0) nanofiber containing black cumin seed waste extract against *Staphylococcus aureus* was attributed to its active compound, thymoquinone, which disrupts the bacterial cell wall and membrane structure and inhibits biofilm formation by generating reactive oxygen species [16, 87]. (Figure 9).

Nanofibers produced using polylactic acid and polycaprolactone polymers in combination with 25% black cumin extract were reported to exhibit antimicrobial activity against *Staphylococcus aureus* and *Escherichia coli* [14]. The antimicrobial property of black cumin has been attributed to an active compound known as thymoquinone [101]. In our previous study, all of the clove extract-loaded pullulan and pullulan/whey nanofibers exhibited antibacterial activity against *S. aureus* and *M. luteus* [35]. Therefore, the low level of antibacterial activity observed against *S. aureus* and the lack of activity against *M. luteus* are thought to be due to the low thymoquinone content in the black cumin seed waste extract, rather than the release properties of the polymer matrix.

Edible milk protein films incorporated with black cumin essential oil demonstrated notable antimicrobial activity against *Staphylococcus aureus* and *Escherichia coli* [87]. In contrast, polyvinyl alcohol and gelatin films formulated with black cumin waste extract did not exhibit any antimicrobial activity against *E. coli*, *Klebsiella pneumoniae*, *Salmonella enterica*, *Micrococcus luteus*, *Listeria monocytogenes*, or *S. aureus* [26]. Similarly,

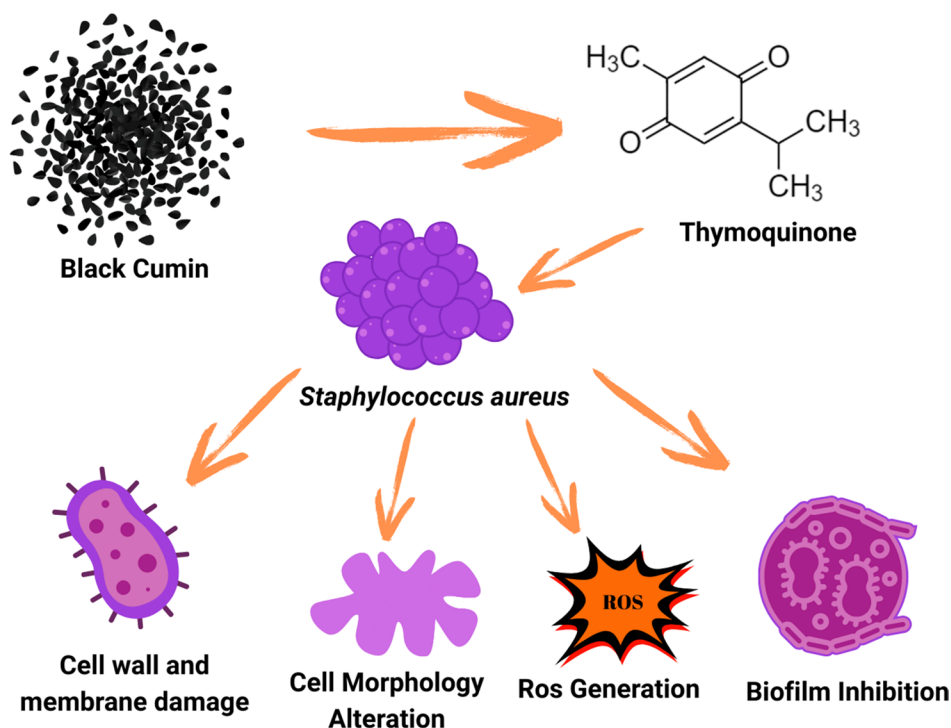


FIGURE 9 | The mechanism of the antibacterial activity of thymoquinone against *Staphylococcus aureus*. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

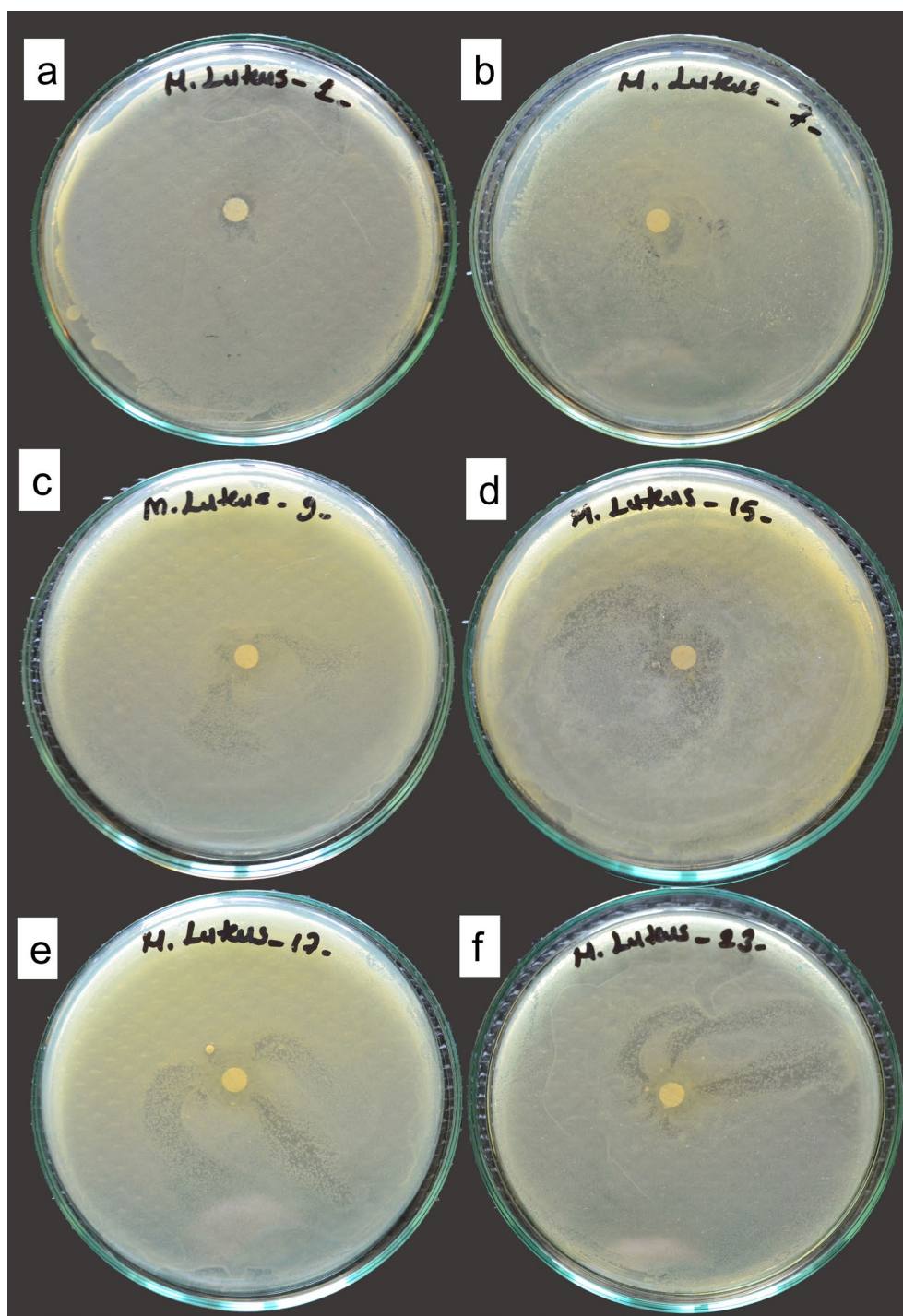


FIGURE 10 | Antibacterial activity of nanofibers; (a) PN (100:0), (b) 5% BWE PN (100:0), (c) P/WN (50:50), (d) 5% BWE P/WN (50:50), (e) P/WN (25:75), and (f) 5% BWE P/WN (25:75) against *M. luteus*. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

polyvinyl alcohol nanofibers enriched with black cumin extract showed antimicrobial efficacy against *S. aureus* and *E. coli*, which was attributed to the presence of bioactive constituents such as thymoquinone and thymohydroquinone [16] (Figure 10).

4 | Conclusion

In this study, green synthesis nanofibers were produced using the electrospinning method with black cumin waste extract and

pullulan: whey protein in ratios of 100:0 (w/w), 50:50 (w/w), and 25:75 (w/w). The nanofibers with a 100:0 (w/w) ratio of pullulan to whey protein exhibited the most uniform morphology, while the morphology of the nanofibers with a 25:75 (w/w) ratio was found to be distorted.

Analysis of the obtained nanofibers through FTIR, DSC, XRD, and FE-SEM imaging results indicated a compatible interaction between the black cumin waste extract and the polymers used in the nanofibers. It was observed that the addition of black cumin

waste extract improved the antioxidant properties of the nanofibers. Furthermore, it was determined that the 5% BWE PN (100:0) nanofiber exhibited antibacterial activity against *S. aureus*.

In light of this information, the development of various properties in the obtained nanofibers, the non-use of any materials that could harm the environment in nanofiber production, and the consideration of black cumin waste extract within the scope of waste management suggest that our study presents a promising contribution to the literature, with potential applications in additives, coatings, or packaging materials aimed at enhancing food safety.

Author Contributions

Özlem Zambak: conceptualization (equal), formal analysis (equal), investigation (equal), methodology (equal), writing – original draft (equal). **Sami Gökhan Özkal:** conceptualization (equal), formal analysis (equal), investigation (equal), methodology (equal), project administration (equal), supervision (lead), writing – original draft (equal).

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

Research data are not shared.

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