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Electrospinning Fabrication of Chitosan/Polyvinyl Alcohol Nanofibers: Surface Morphology and Optical Properties Investigation"

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Abstract
In this study nanofiber PVA/Chitosan with volume ratio 7:3(V/V) have been prepared by electrospinning method. Electrospinning was performed at a voltage 18 kV, with a tip-collector distance of 10 cm and flow rate 1ml/h. The structure of PVA/Chitosan was examined by FTIR, XRD and SEM, Tensile strength and contact angle (CA) were also investigated. The results showed that PVA/Chitosan nanofiber was successfully formed with small beads with smooth surface. SEM observation showed that the fiber had several hundred nanometers about 230nm.

Keywords: *Electrospinning, Nanofibers, Chitosan, Poly (vinyl alcohol), optical energy gap*

1.INTRODUCTION

With the rapid development of nanoscience and nanotechnology over the last two decades, great progress has been made not only in preparation and characterization of nanomaterials, but also in their functional applications. As an important one-dimensional nanomaterial, nanofibers have extremely high specific surface area because of their small diameters, and nanofiber membranes are highly porous with excellent pore interconnectivity. These unique characteristics plus the functionalities from the polymers themselves impart nanofibers with many desirable properties for advanced applications.

For many biomedical applications, the most important parameters that should be considered are biocompatibility and mechanical performance. In comparison with synthetic materials, natural biopolymers usually show better biocompatibility and hence, are more suitable for use in the human body. Electrospun fibers of these biomaterials could therefore be of great interest in numerous biomedical and pharmaceutical applications, including tissue engineering, medical implants and body-implant interphases. Technically, electrospinning is a process that uses a strong electrical field to draw a polymer fluid into fine filaments. The basic electrospinning set-up, shown in Figure (1), consists of a needle nozzle, a high voltage power supply, a container for spinning fluid and an electrode collector. When a viscous fluid is charged with a high voltage, the electrostatic force draws the fluid into a liquid jet. Because of the interaction between the jet and external electric field and charge repulsion inside the jet, the charged jet undergoes a bending or whipping instability to stretch it thinner. Solvent evaporation from the filaments results in solid fibers in Figure(1). In most cases, the as-spun fibers deposit randomly on the electrode collector forming a non-woven nanofiber mat. Aligned nanofibers can also be produced using controlled fiber deposition techniques [1,2].

Until now, a large number of polymeric and inorganic materials have been electrospun into nanofibers. The nanofibers can have many different morphologies, such as porous-surface nanofibers [3,4], core-sheath[5,6]and side-by-side structures [7]

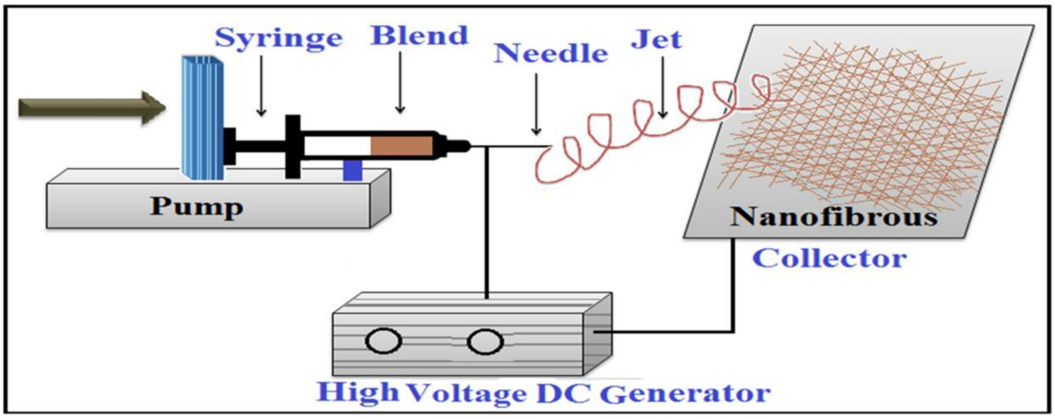


Figure (1): Schematic diagram for the electrospinning process to produce

Chitosan (CS) is a product of natural polysaccharide chitin with acetyl groups removed, and it has a variety of physiological functions including the biodegradability, biocompatibility, non-toxicity and antibacterial ability [8,9,10]. Therefore, electrospun nanofibers with chitosan have unique advantages in biomedical applications since they mimic the natural extracellular matrix (ECM) as a structure to support and regulate cell behavior [11,12]. However, CS is a cationic polyelectrolyte that makes it positively charged when only dissolved in solution, which leads to poor electrospinning properties [13].

Repulsive interactions between like-charged groups destroy Taylor cone, thus making it difficult for the fiber to form when electrospinning [14]. Researchers have used different methods to improve the electrospinability of CS. Schiffman et al. used trifluoroacetic acid (TFA) to dissolve chitosan for one-step electrospinning [15].Anionic polyelectrolyte has been utilized to neutralize cations in chitosan solution, like hyaluronic acid [16,17]. Some researchers also attempted to mixing CS with other polymers with good electrospinability, such as polyvinyl alcohol (PVA), polylactide co-glycolide (PLGA) [18] polyethylene oxide (PEO)[19] .

PVA is a widely recognized biodegradable man-made polymer that is soluble in water. PVA possesses astonishing characteristics such as biocompatibility, non-toxicity, biodegradability, adhesive properties, film-forming capability, good mechanical strength, good chemical resistance and excellent charge storage capacity [20]. PVA has also been employed in controlled drug delivery systems, packaging, dialysis, nanofltration, implantable medical devices and membrane preparations [21]. The presence of reactive functional groups in PVA makes them prominent in research. These functional groups increase the intermolecular interactions between constituent polymers. PVA's strong hydrophilicity is a drawback in many applications because it causes molecular aggregation via hydrogen bonding. The introduction of organic or inorganic components into the PVA matrix achieves the desired properties and features that are not exhibited by the constituent components individually [22].However, utilization of toxic solvents is not optimal, especially in biomedical applications. As a non-toxic, hydrophilic and biocompatible synthetic polymer, PVA has been used in various biomedical fields [23]. Related studies have reported some examples of nanofibers prepared by electrospinning with PVA and chitosan [24,25]. The people trained in this profile should know about the business, computational tools, and statistical analysis and interpretation. Among then objectives of Information Science is to provide a means for making relevant information available to individuals, groups, and organizations involved with science and technology [39].

In the present work, conventional electrospinning method was employed to synthesize PVA/CS nanocomposite fibers. The morphology and diameter of PVA/CS nanocomposite fibers were characterized by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The functional groups of PVA/CS nanocomposite fibers were characterized by Fourier Transform infrared spectroscopy.

2. Characterization Techniques

2.1. Fourier transform infrared (FT-IR) spectroscopy

Characterization of polymers using FTIR (Fourier Transform Infrared Spectroscopy) type Bruker OPTIK Gmbh&Co.KG Model ALPHAII 113369(Germany) is a widely used technique in polymer science and engineering. FTIR provides information about the chemical structure and functional groups present in the polymer, helping researchers understand its properties and behavior. FTIR spectroscopy is a versatile tool that aids in understanding the structure, properties and behavior of polymer. Here in this study this technique was used to characterized and identify the reactive functional groups present in the prepared polymers and polymer composites. The spectra were vector normalized over the entire wavelength range of 400-4000cm^{-1}.

2.2. X-ray diffraction

X-ray diffraction (XRD) is an analytical technique primarily used for phase identification of a crystalline material. The samples were scanned on a Diffractometer system XPERT-PRO, which uses Cu-k-alpha X-rays at 40 kV and 30 mA.

2.3 Scanning Electron Microscopy (SEM)

The morphology of the fiber mat produced by the conventional static collector assembly and SBMS setup was studied using SEM (NOVA NANOSEM450) A small section of fiber mat was mounted onto the sputtered. Image processing software was used to measure the diameter of the nanofiber captured in the SEM micrograph.

2.4. Mechanical strength /Tensile strength

The tensile strength of the prepared electrospun nanofibers was measured using Universal Mechanical Tester (Zwick/Roll by Zwick GmbH & Co. KG) with a 0.21MPa load cell under standard atmospheric condition. The samples were cut into 40×10 mm size and placed in grips of the testing machine. Tensile property of the nanofiber samples was measured with grip separation. The samples were stretched with cross head speed as 0.1 mm/min, that attempted to pull the specimen apart and the test was run until the sample broke under the load. To ensure reliable result the process was performed twice for each sample.

3.MATERIALS AND METHODS

3.1 Materials

CS (2000-3500cps)very high molecular weight was purchased from Glentham LIFE SCIENCES Ltd and PVA (molecular weight 14000 g/mol) from THOMAS BAKER (India). Glacial acetic acid (99.7%) was purchasedfrom LOBA CHEMIE PVT.LTD.

3.2. Preparation of electrospun nanofibers

10wt% aqueous solution of PVA was prepared and 2wt% CS solution was prepared by dissolving CS in acetic acid water (90% v/v). The PVA / CS solution was mixed with 7:3 volume ratios. The solution was stirred at room temperature for about 24 hrs. until homogenous clear of PVA/CS solutions was obtained. Then the polymer solution was transferred into 10ml plastic syringe (21G), a high voltage (18 kV) was applied by copper electrode connected with syringe needle with a feed rate of 1 ml /hr., while the needle distance from target was 10 cm.

4. RESULTS AND DISCUSSION

4.1. FTIR of PVA/Chitosan Nanofiber (PVA/CS NF)

Figure(2) ,show the FTIR of PVA ,strong and broad absorption band at 3305cm^{-1} due to the stretching vibration of hydroxyl group , also absorption band at 2906 ,2854cm^{-1} refer to stretch vibration of CH_2 asymmetric and symmetric , absorption broad bands around 1700cm^{-1} due to carbonyl groups(C=O) ,band at 1579,1434cm^{-1} corresponding to C-H bending vibration of CH_2 , and absorption band at 1084cm^{-1} due to stretching vibration of C-O stretching of acetyl groups .Figure(3) the FTIR spectrum of Chitosan (CS) shows the characteristic absorption bands at 3360cm^{-1} due to stretching vibration of OH groups presented in their structure overlaped with N-H group .Another absorption band at 2861cm^{-1} is attributed to aliphatic C-H ,also bands 1660,1583cm^{-1} corresponds to $CONH_2$ and NH_2 groups and bands at 1072,1012cm^{-1} due to the stretching vibration of C-O group . Figure(4) For Chitosan / PVA nanofiber blend ,it is noticeable that the spectrum induded bands and overlaped bands due to Chitosan and PVA , and depending on the polymer concentration ,the band of Hydroxyl group of chitosan also was shifted to strong broad bands at 3288cm^{-1} ,also strong bands appear at 2920cm^{-1} was related to antisymmetric CH_2 stretching of PVA .On the other hand small and shoulder bands at 1729cm^{-1} vibration of C=O bands present in acetale unit in PVA. Additionally, it was notice a shift of bands in spectrum of Chitosan /PVA compare with spectrum of Chitosan alone, at 1647,1538cm^{-1} which due to $CONH_2$ and NH_2 group. Finally, the two absorption bands of Chitosan at 1072,1012cm^{-1}was overlaped in Chitosan / PVA blend at one strong absorption band at 1082cm^{-1} which indicated of C-O group in their structures [26,27].

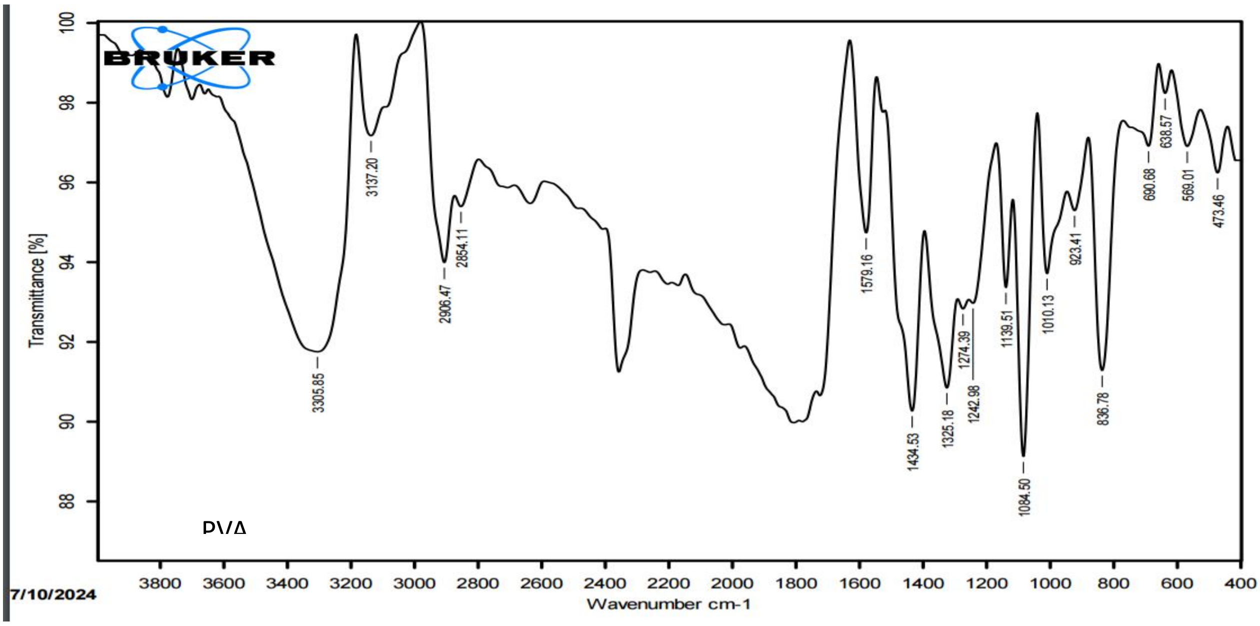


Figure (2): FTIR for PVA

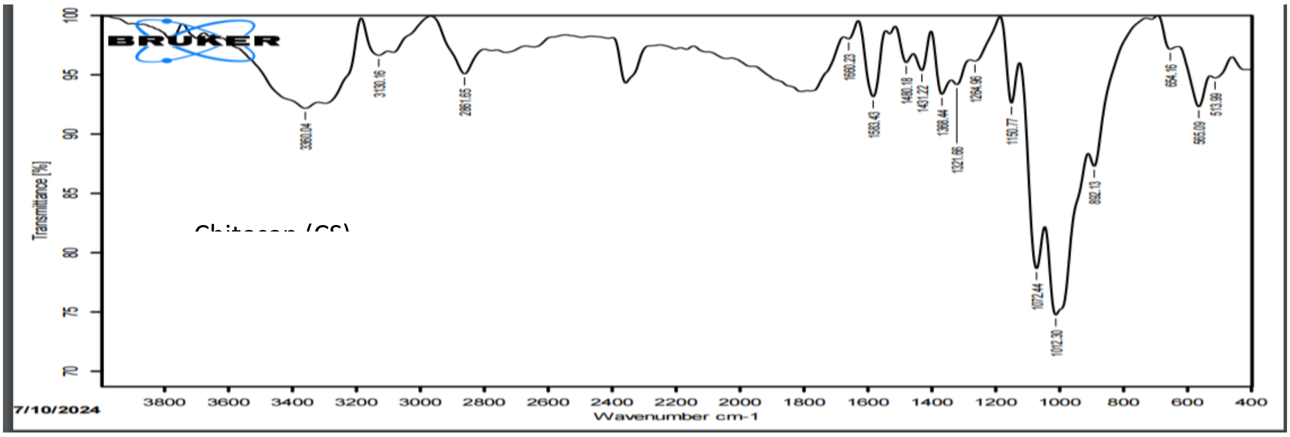


Figure (3): FTIR for Chitosan

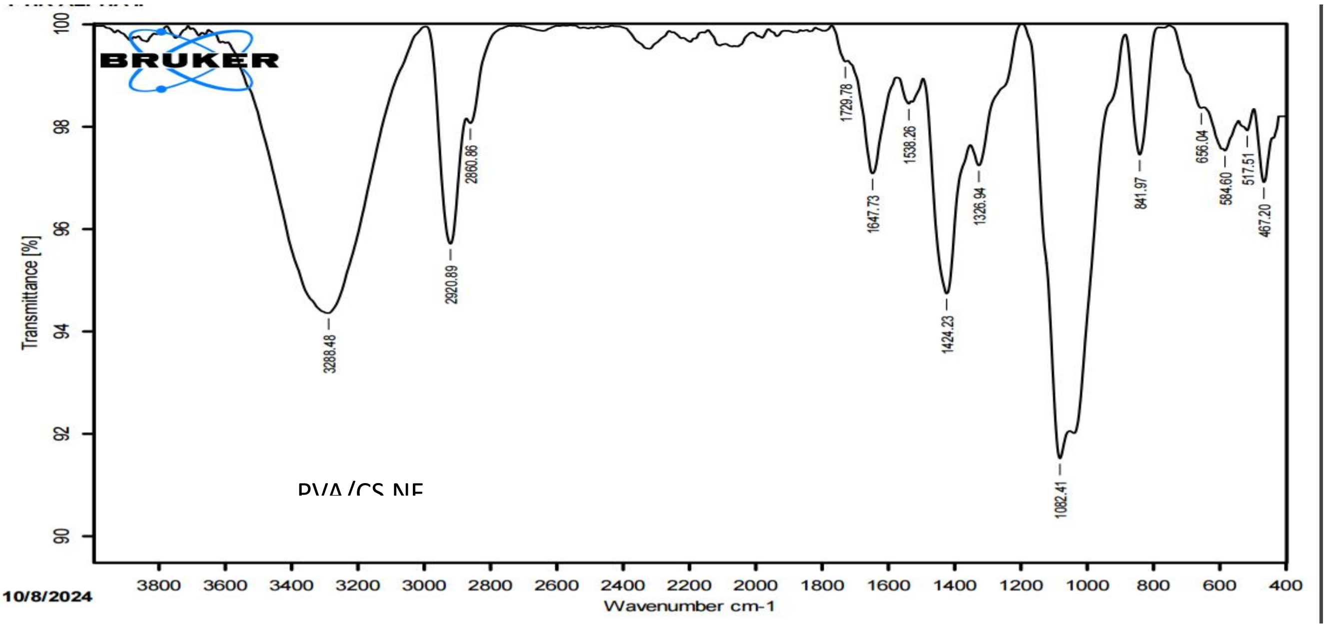
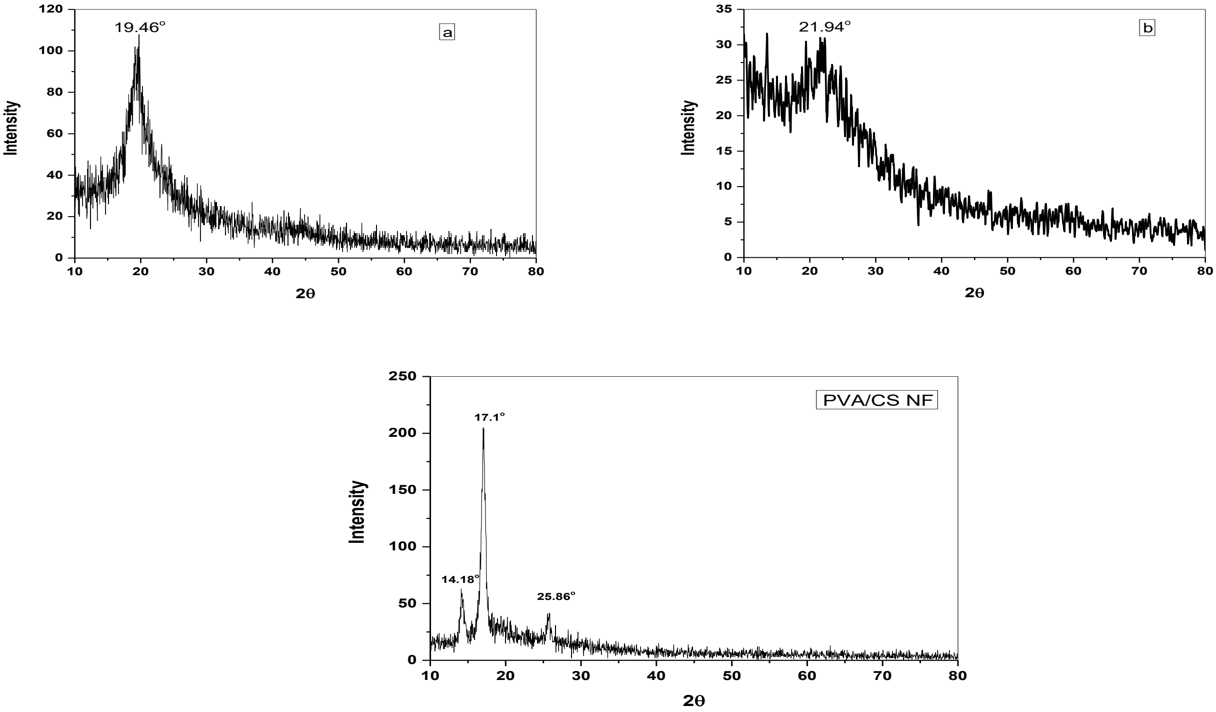


Figure (4): FTIR for PVA/CS NF

4.2. X-Ray diffraction

X-ray diffractogram was studied to assess the phase change during blend formation and crystalline nature of the prepared nanofiber. Figure (6a) shows the X-ray diffraction pattern of pure PVA which showed distinct semi-crystalline character peak at 2θ of 19.46° [28], while chitosan which showed distinct crystalline peaks at around 2θ values 21.94°[29] as shown in figure(6b). This is because of presence of plenty of –OH and –NH$_2$ groups in the chitosan structure, which could form stronger inter and intramolecular hydrogen bonds and the chitosan structure has certain regularity, so that the molecules form crystalline regions easily which was similar to the results reported by Duan and his co-workers. [30,32]. Figure(6c) show the PVA/CS blend nanofiber. Three peaks, small peak at 14.18° and a narrow peak at 2θ = 17.1° and a weak peak at 2θ = 25.86° indicating that the PVA/CS blend is semi-crystalline due to the strong intermolecular interaction between PVA chains and CS [33, 34].



Figure(6) : XRD analysis of (a) PVA (b) CS and (c) PVA/CS NF.

4.3. SEM of PVA/CS nanofiber

The SEM micrographs and diameter distribution of PVA/CS composite nanofibers was shown in Figure (7). Composite nanofibers with small beads and fine morphology with random direction was observed. The results suggest that the PVA/CS electrospinning solution could be successfully fabricate nanofibers. The average diameter of PVA/CS composite nanofibers was about 230.72nm

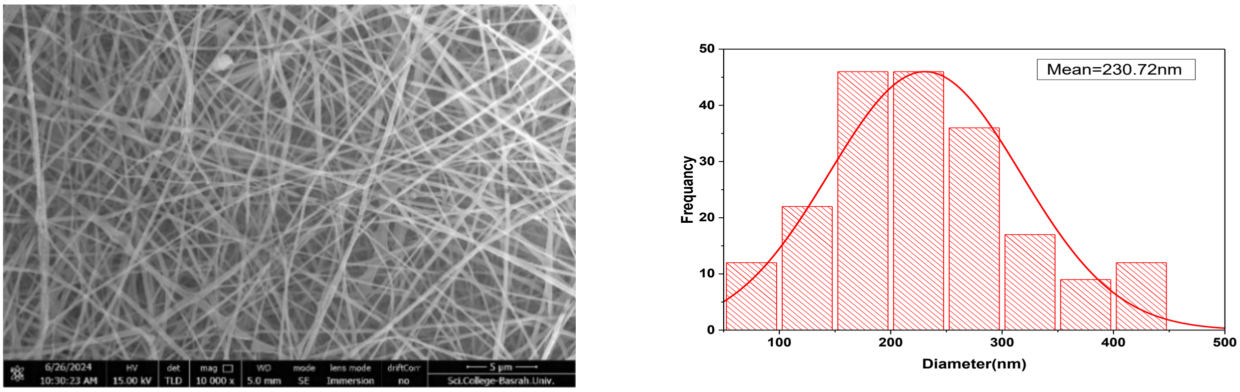


Figure (7): SEM image of PVA/CS NF blend.

4.4. Mechanical tests

Figures (8) show the stress-strain relationship of (PVA/CS NF) the Young Modulus was about 2.94MPa, the tensile strength was 5.69N, while the maximum elongation at brake was nearly 88.7%.

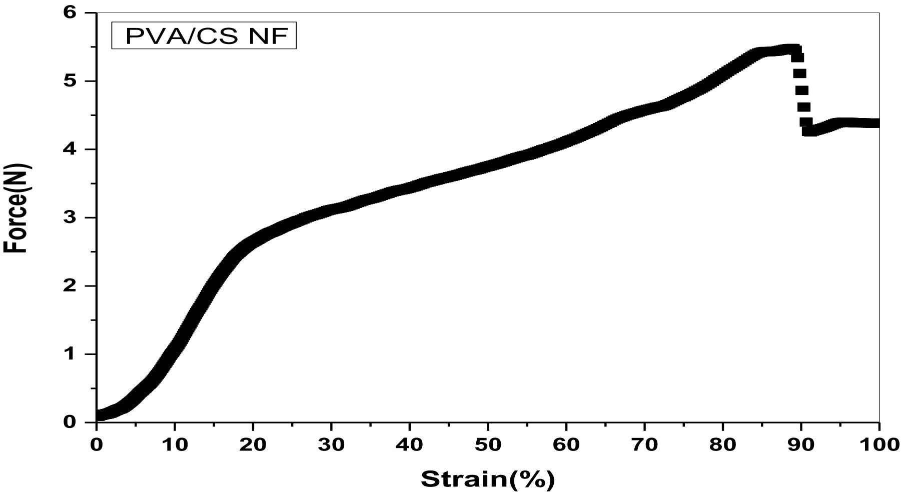


Figure (8): Tensile strength of PVA/CS NF

4.5 Hydrophilicity

Wettability is an important property of a solid surface for both research and practical applications.[35] Surfaces with special wettability play an important role in our daily life as well as in industrial applications. Surface roughness and surface energy are the dominant factors in determining the wettability of a surface. The measured contact angle of PVA/CS nanofiber blend is found to be about 66.72°as shown in Figure (9), indicating their hydrophilic nature. This result agreements with another study [36].

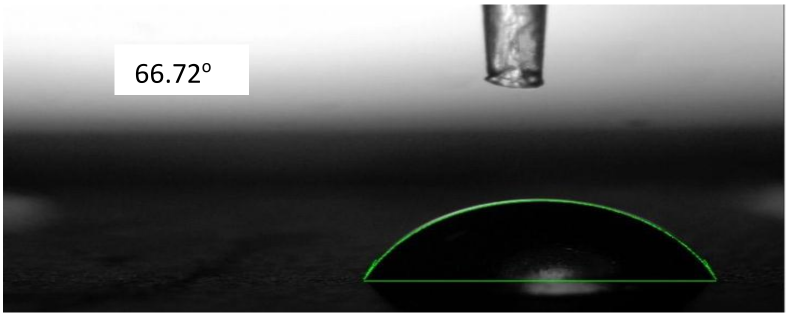


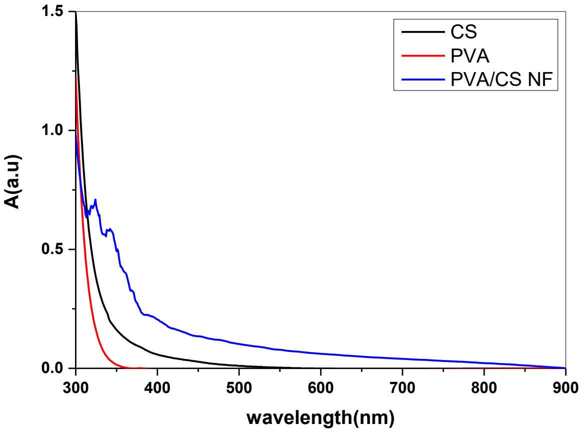
Figure (9): contact angle of PVA/CS nanofiber

4.6 optical characteristics

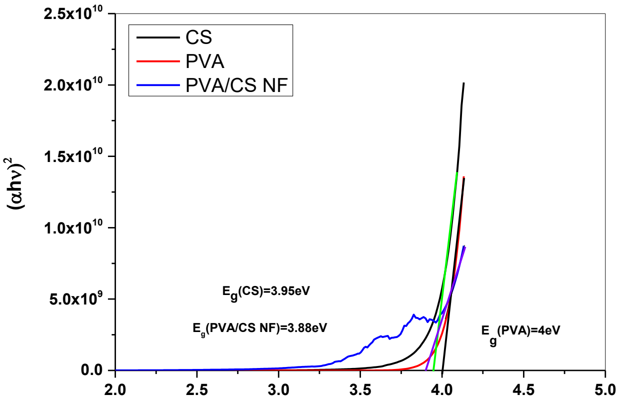
The energy gap (or band gap) of polyvinyl alcohol/chitosan (PVA/CS) nanofibers is a critical parameter often investigated in materials science, particularly for their applications in optoelectronics, sensors, and biomedical fields. The energy gap refers to the energy difference between the valence band and the conduction band in a material, which influences its electrical and optical properties. Figure (10) show the absorption spectrum of PVA/CS NF at wavelength range(300-900nm) . From figure we notice the peak at 323nm and 341nm is assigned to the $\pi \rightarrow \pi^*$ transition in the PVA/CS NF blend and it arises from the presence of carbonyl group containing segments in the blend [37]. The optical bandgap energy (E_g) is the amount of energy necessary to excite from HOMO (conduction band) to LUMO (valance band). The direct optical bandgap energy values of PVA/CS blend and blend composites are calculated using the Tauc's equation given below [38].

$$(\alpha h\nu)^2 = B(h\nu - E_g)$$

where α, $h\nu$ and B denote the absorption coefficient (the ratio of absorbance to route length), photon energy, and probability transition constant, respectively. Figure (11) shows the Tauc plots of PVA/CS NF blend. Extrapolating the straight-line part of curves to $\alpha = 0$ yields the E_g value of the sample. The E_g value of pure PVA is 4 eV while the CS of 3.95 eV and the nanofiber blend about 3.88eV respectively. The reduction in bandgap energy of nanofiber blend relative to pure blend is attributed to the formation of new intermediate states of energy between the valance band and conduction band [37]. Thus, the electrons can be easily excited through this intermediate energy level. The E_g decreases with blend is due to the homogeneous fibers in low beads.



Figure(10):Absorpation spectrum of PVA,CS and PVA/CS NF



Figure(11): Tauc plots of PVA, CS and PVA/CS NF

5. CONCLUSIONS

In this study, nanofibers composed of chitosan and polyvinyl alcohol (PVA) were successfully prepared using electrospinning. Fourier-transform infrared (FTIR) spectroscopy confirmed the formation of intermolecular interactions between chitosan and PVA, indicating successful blending of the polymers. Surface morphology analysis revealed uniform fiber distribution with minimal defects, supporting the efficiency of the electrospinning process. Mechanical property evaluation demonstrated enhanced tensile strength and elasticity, making the nanofibers suitable for various applications. Contact angle measurements indicated improved hydrophilicity, which is beneficial for biomedical and filtration applications. X-ray diffraction (XRD) analysis suggested a semi-crystalline structure, highlighting the compatibility of chitosan and PVA within the nanofiber matrix. Optical property analysis showed favorable transparency and light absorption characteristics, expanding their potential in optical and sensor technologies. A tunable energy gap makes PVA/CS nanofibers attractive for photocatalysis, UV shielding, and sensor technologies. Overall, the synthesized nanofibers exhibit excellent structural, mechanical, and optical properties, making them promising candidates for advanced material applications.

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