

Investigating the Impact of Polyvinyl Alcohol Addition on the Thermal Behavior and Chemical Structure of Electrospun Cellulose Nanofibers

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Abstract. The study aimed to investigate the effect of adding polyvinyl alcohol (PVA) on the chemical and thermal properties of cellulose nanofibers (CNFs). The CNF was prepared using electrospinning with varying amounts of PVA, and the resulting samples were analyzed using Fourier-transform infrared (FTIR) spectroscopy and Thermogravimetric analysis (TGA). The FTIR results showed that adding PVA did not significantly affect the chemical structure of CNF but led to the appearance of new peaks attributed to the characteristic peaks of both CNF and PVA. Moreover, the TGA results revealed that all samples underwent weight degradation after being heated from 350°C. The addition and removal of PVA did not significantly alter the thermal behavior of the CNF. Overall, the results suggested that PVA can be used as a compatible additive to CNF without significant changes to their properties, which could have potential applications in various fields, including biodegradable packaging and drug delivery systems.

1 Introduction

Cellulose nanofibers are a promising material with a wide range of potential applications, including in biomedicine, packaging, textiles, and energy storage [1]. Electrospinning is a popular technique for producing cellulose nanofibers, which involves the use of an electric field to generate a charged jet of polymer solution that is then collected on a grounded substrate [2]. However, cellulose nanofibers produced by electrospinning often have poor mechanical properties, such as low tensile strength and Young's modulus, which can limit their use in some applications [3]. To overcome these limitations, various additives have been studied to improve the mechanical properties of electrospun cellulose nanofibers. Polyvinyl alcohol (PVA) is one such additive that has been shown to enhance the mechanical properties of cellulose nanofibers [4]. PVA is a water-soluble synthetic polymer that has good compatibility with cellulose and forms strong hydrogen bonds with it. When added to the polymer solution before electrospinning, PVA can help to increase the viscosity and surface tension of the solution, leading to improved fiber alignment and tensile strength of the resulting nanofibers [5].

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Fourier-transform infrared spectroscopy (FTIR) and thermogravimetric analysis (TGA) are two common analytical techniques used to investigate the chemical and thermal properties of materials [6]. FTIR is used to analyze the functional groups and chemical bonds present in a sample, while TGA measures changes in the weight of a sample as it is heated or cooled, providing information on its thermal stability and behavior. These techniques are widely used in the field of materials science to understand the properties of new materials and optimize their design for various applications. The aim of this study is to investigate the impact of adding PVA to electrospun cellulose nanofibers on their chemical structure and thermal behavior, using FTIR and TGA as analytical techniques. The study is likely to involve preparing different samples of electrospun cellulose nanofibers with varying amounts of PVA, as well as a control sample of cellulose nanofibers without any PVA.

The samples will then be analyzed using FTIR and TGA to investigate changes in their chemical structure and thermal behavior. FTIR may be used to investigate changes in the functional groups and chemical bonds present in the samples due to the addition of PVA, while TGA may be used to investigate changes in their thermal stability and behavior. The results of the study may shed light on the mechanisms by which PVA modifies the properties of electrospun cellulose nanofibers, as well as provide insights into how to optimize the design of cellulose-based materials for various applications [7]. The study may also contribute to the development of new materials with improved mechanical properties for use in areas such as tissue engineering, packaging, and energy storage.

The novelty of this study lies in its investigation of the impact of PVA addition on the chemical structure and thermal behavior of electrospun cellulose nanofibers using FTIR and TGA. While previous studies have investigated the effects of various additives on the mechanical properties of cellulose nanofibers, there has been relatively little research on how these additives affect the chemical and thermal properties of the resulting nanofibers [8]–[10]. By using FTIR and TGA to analyze the samples, this study will provide a detailed understanding of the chemical changes that occur in the cellulose nanofibers as a result of PVA addition, as well as the thermal behavior of these nanofibers. This information will be valuable for designing cellulose-based materials with improved mechanical and thermal properties, which could have a wide range of practical applications.

In addition, this study will contribute to the growing body of knowledge on the use of electrospinning as a technique for producing cellulose nanofibers with enhanced properties. As electrospinning is a relatively new and promising technique for producing nanofibers [11]–[13], there is still much to be learned about the factors that affect their properties and how to optimize their design for specific applications. Overall, the novelty of this study lies in its use of advanced analytical techniques to investigate the impact of PVA addition on the chemical structure and thermal behavior of electrospun cellulose nanofibers, which has the potential to contribute to the development of new materials with improved properties for a variety of applications.

2 Experimental Section

2.1 Procedure

A mixture of 40 ml cellulose and 1 gr polyvinyl alcohol (PVA) was heated to 90°C with stirring at 350 rpm for 2 hours. The resulting PVA solution was then fed into the spinneret, and electrospinning was performed at input voltages of 30 volts for 30 minutes, with a spinneret-collector distance of 8 cm, following the method used in a recent study [14] to produce cellulose nanofibers (CNF). PVA was added in varying amounts of 1 gram and designated as C1. A control sample was also prepared without PVA, according to the same

procedure and labeled as C0. The sample preparation process was illustrated in Figure 1. TGA and FTIR analysis were performed to investigate the effect of PVA addition on the thermal properties and chemical structure of the samples. Additionally, DSC analysis was conducted to determine the degree of crystallinity of the electrospun CNF using the following equation (1):

$$X_c [\%] = \left[(\Delta H_m / \Phi_{PVA}) / \Delta H_m^o \right] \times 100 \quad (1)$$

where ΔH_m (J/g) is the enthalpy of fusion of the CNF/PVA, ΔH_m^o is the enthalpy of fusion of a PVA crystal of infinite size (assumed to be 93.6 J/g), and Φ_{PVA} is the fraction of PVA in the composite.

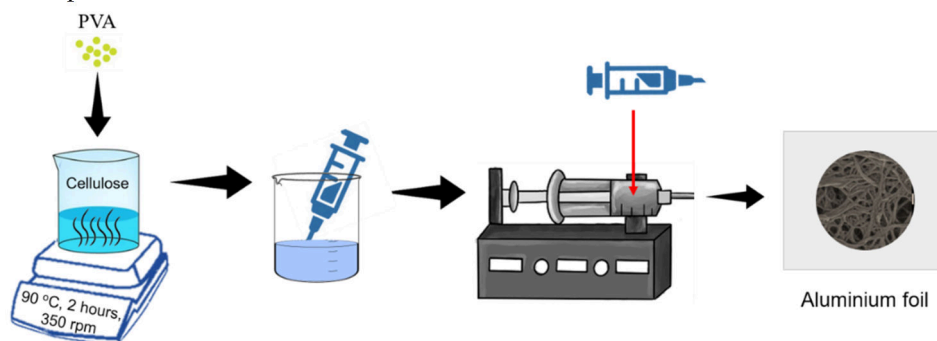


Fig. 1. The illustration of sample preparation

3 Results and Discussion

3.1 Thermal Properties

Thermal properties were investigated to analyze the thermal behavior of CNF before and after being added with PVA to different temperature treatments. In addition, the thermal behavior of the sample after removal of the PVA was also observed to confirm the effect of its existence. Figure 3a shows the TGA curve of samples of C0, C1, PVA removed C1, and PVA samples at a temperature of 30-850°C with a heating rate of 10°C/min and a flow rate of 30 mL/min. In the temperature range of 50-100 °C, evaporation of moisture CNF and PVA occurs [15], the next stage of the amorphous region pyrolysis process in the temperature range of 100-300 °C [16], and in the temperature range of 350°C – 600 °C occurs stages of degradation of amorphous PVA, glycosidic linkage and crystalline region of cellulose [17]. All samples underwent weight degradation after being heated from 350 °C. at that temperature, samples C0 and C1 lost weight by 7% and 5%, respectively.

When the temperature was increased again to 518 °C, the weight of the sample C0 decreased drastically until at a temperature of 560 °C the remaining weight was only 2% of the total weight. The increase in temperature above it, only experienced a small and slow decrease. Sample C1 experienced only 25% weight loss up to 560 °C. At this temperature, cellulose undergoes degradation in the crystalline region [18], but PVA supports temperature resistance in the crystalline structure of cellulose by increasing the bond between PVA and the cellulose fiber network [17]. Increasing the degree of crystallinity is the most likely reason for cellulosic compactness [19], thus limiting the degradation process so as to show better thermal stability than the other samples [20]. This is necessary for the stability of the mass reduction to heat applied to the electrospun CNF sample. On the one hand, the

heterogeneous molecular structure of PVA causes it to behave in a multistep gradient pyrolysis.

The stability of the skeleton structure of PVA against thermal decomposition is the main reason that PVA only loses weight by 44% after 800 °C heat treatment [21]. So the dominant residue in sample C1 is basically PVA thermal decomposition products when heating reaches 700 °C. In the case of CNF from which PVA was removed using a distilled water washing process, the aromatic skeleton peak of PVA disappeared, and the spectrum was almost identical to that of neat C0. This means that a simple washing process can effectively remove PVA from the dried and redispersed CNF/PVA system (see black and blue curve line on Figure. 2a).

The results of the thermal analysis conducted in this study demonstrate the significant influence of PVA on the thermal behavior of CNF. The weight loss of the sample containing PVA (C1) was less than that of the sample without PVA (C0) due to the thermal stability of the PVA skeleton structure. This behavior is consistent with previous research on the thermal behavior of PVA [22]. Additionally, the CNF/PVA system increased the bond between PVA and cellulose fibers, thereby increasing the degree of crystallinity and improving the thermal stability of the sample. These findings suggest that the electrospun CNF samples have good thermal stability, which is crucial for their use in practical applications.

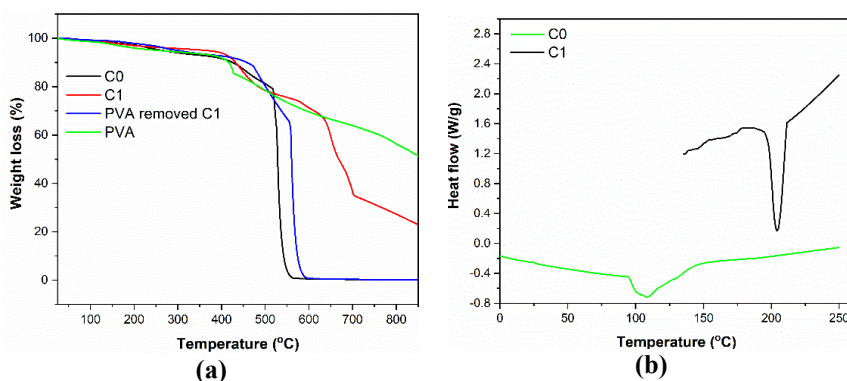


Fig. 2. TGA curve (a), and DSC curve (b) of C0, C1, PVA removed C1, and PVA samples

Moreover, the study also revealed that the removal of PVA using a simple washing process did not significantly alter the thermal properties of CNF. The FTIR spectra of the PVA-removed C1 samples showed no detectable aromatic skeletal peaks of PVA, which indicates the effective removal of PVA. This result is consistent with previous studies, which reported that a simple washing process using distilled water can effectively remove PVA from CNF/PVA systems [23]. The ability to remove PVA without significantly altering the thermal properties of CNF is important for the practical application of electrospun CNF samples, as it enables the customization of the properties of the sample to suit the intended application. Overall, the results of this study provide insights into the thermal behavior of CNF/PVA systems and demonstrate the effectiveness of a simple washing process for PVA removal.

In Figure 3b and Table 1, the DSC curve and data for C1 and C0 sample are provided, respectively. The glass transition temperature (T_g) of pure CNF is 61°C, whereas C1 has a slightly higher T_g of 79°C. T_g refers to the temperature at which a polymer changes from a hard to a soft state, and higher T_g values imply increased polymer rigidity. As C1 has a higher T_g value than C0, it suggests that C1 is better at maintaining its rigidity than C0 [24]. However, C1's crystallization temperature (T_c) of 103.9°C is higher than C0, indicating that cellulose can act as a nucleating agent in electrospun CNFs. Moreover, C0 has a large

enthalpy melting, which leads to a melting temperature (T_m) of 181.2°C. Endothermic peaks in C0 may be related to changes in the heat capacity of lignin when the polymer reaches its T_m value and the release of water and other volatiles. C1 has a degree of crystallinity (X_c) of 15.49%, while C0 has a lower X_c value of 14.74%. C0's higher X_c value indicates that its strength and stiffness are higher than C1, and it also reduces solubility and permeability [25].

Table 1. DSC data of C0 and C1 sample

Samples	T_g (°C)	T_c (°C)	T_m (°C)	ΔH_m (J/g)	X_c (%)
C0	61	119	181.2	13.8	14.74
C1	79	103.9	140.3	14.5	15.49

The thermal properties analysis of electrospun CNF with and without PVA addition provides valuable information on the behavior of the material at different temperature treatments [26]. The TGA results demonstrate that the addition of PVA has a significant impact on the weight loss of the sample at high temperatures. PVA exhibits a multistep gradient pyrolysis due to its heterogeneous molecular structure and high thermal stability [27]. This, in turn, contributes to the PVA thermal decomposition products as the dominant residue in the CNF/PVA electrospun CNF. The removal of PVA from the CNF/PVA system through a simple washing process is effective in restoring the CNF's original characteristics. The DSC results suggest that the addition of CNF to PVA increases the glass transition temperature and crystallinity, indicating an improvement in the stiffness and strength of the material. This is attributed to CNF's ability to maintain its rigidity at higher temperatures and PVA's role as a nucleating agent in the electrospun CNF. Overall, these results provide crucial insights into the thermal behavior and characteristics of CNF/PVA electrospun CNF and highlight its potential for various industrial applications.

The study's findings are significant for designing sustainable electrospun CNF materials with improved mechanical and thermal properties. The CNF/PVA electrospun CNF can offer several advantages, such as lightweight, high strength, and thermal stability, making it suitable for diverse applications, including packaging, coatings, and biomedical devices. The ability to remove PVA from the CNF/PVA system through a simple washing process is a significant advantage that can contribute to reducing the environmental impact and cost of the electrospun CNF. Furthermore, the results demonstrate the role of PVA as a nucleating agent in the crystallization of PVA, which can be leveraged to improve the performance of other electrospun CNF systems. The study's limitations include the use of only one type of CNF source, and further research is needed to investigate the effect of different CNF sources on the electrospun CNF's properties. Nonetheless, the study's findings provide a useful framework for the development of sustainable electrospun CNF materials with enhanced mechanical and thermal properties.

3.2 Fourier Transform Infra-Red (FTIR)

Thermal properties were investigated to analyze the thermal behavior of CNF before and after being added with PVA to different temperature treatments. In addition, the thermal behavior of the sample after removal of the PVA was also observed to confirm the effect of its existence. Figure 3a shows the TGA curve of samples of C0, C1, PVA removed C1, and PVA samples at a temperature of 30-850°C with a heating rate of 10°C/min and a flow rate of 30 mL/min. In the temperature range of 50-100 °C, evaporation of moisture CNF and PVA occurs [28], the next stage of the amorphous region pyrolysis process in the temperature range of 100-300 °C [29], and in the temperature range of 350°C – 600 °C occurs stages of degradation of amorphous PVA, glycosidic linkage and crystalline region of cellulose [16].

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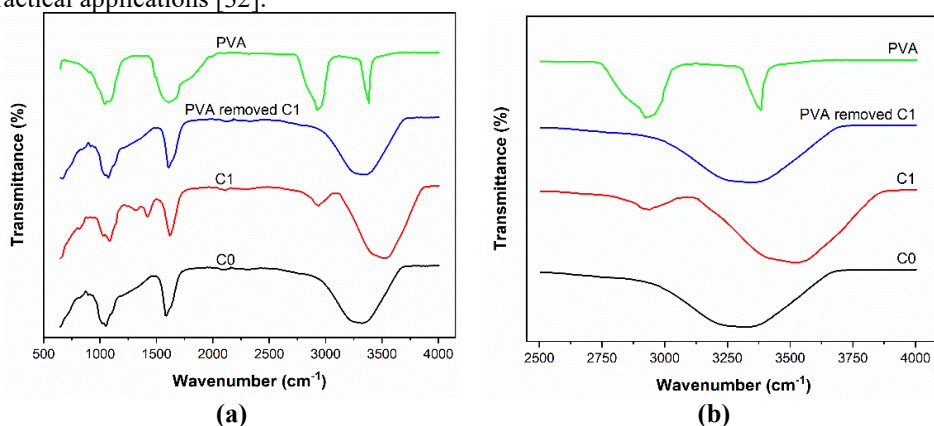


Fig. 3. FTIR spectra (a), and fingerprint region (b) of C0, C1, PVA removed C1, and PVA samples

4 Conclusion

In conclusion, the addition of PVA to electrospun CNFs showed significant effects on the thermal properties of the samples. The TGA and DSC analyses revealed that PVA can improve the thermal stability and crystallization behavior of electrospun CNFs, which is

important for maintaining their structural and mechanical properties at high temperatures. The removal of PVA from the electrospun CNFs was also found to be easily achievable through a simple washing process using distilled water. These findings provide insights into the design and optimization of electrospun CNFs for various applications where thermal stability is crucial. In terms of future perspectives, further studies can explore the effects of varying PVA concentrations on the mechanical performance of electrospun CNFs. Moreover, the use of other polymers or additives can be investigated to further enhance the properties of these electrospun CNFs. The development of cost-effective for the production of electrospun CNFs can facilitate their widespread use in various industries with broad applications in areas such as packaging, construction, and biomedicine.

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