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Research Article

Characterization of Luffa-reinforced Polyaniline Films

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ABSTRACT

Herein, Polyaniline (PANI)/polyethylene oxide (PEO) - luffa cylindrica bio-composite films of various mass fractions (%) have been prepared via casting solution of emeraldine base polyaniline and cellulose extracted from luffa. The biopolymer films were structurally and thermally characterized using X-ray diffraction (XRD), Fournier Transform-InfraRed (FT-IR) spectroscopy, and differential scanning calorimetry analysis (DSC). Moreover, the electrical properties of conductive biopolymer solutions were measured by using the conductivity meter. According to the obtained results, treated luffa increases the conductivity of biopolymers. XRD results reveal that luffa increases the crystallinity of bio-composite films in comparison to PANI/PEO films. FTIR analysis proved the presence of functional groups of PANI, PEO, and luffa in the film structure. Also, an increase in the weight of luffa in the bio-composite film brings about an increase in the peak intensities of the O-H group. It is determined that luffa enhances the thermal stability of composites via the results of DSC analysis.

Keywords: Luffa, PANI, Bio-composite, film

Luffa Takviyeli Polianilin Filmlerin Karakterizasyonu

Öz

Burada, emeraldin bazlı polianilin ve lif kabağından ekstrakte edilen selülozun döküm çözeltisi kullanılarak çeşitli kütle fraksiyonlarına (%) polianilin (PANI)/polietilen oksit (PEO) - lif kabağı cylindrica biyo-kompozit filmler hazırlanmıştır. Biyopolimer filmler, X-ışını kırınımı (XRD), Fournier Dönüşüm-Kızılötesi (FT-IR) spektroskopisi ve diferansiyel taramalı kalorimetri analizi (DSC) kullanılarak yapısal ve termal olarak karakterize edilmiştir. Ayrıca, iletkenlik ölçer kullanılarak iletken biyopolimer çözeltilerinin elektriksel özellikleri ölçülmüştür. Elde edilen sonuçlara göre, işlenmiş lif kabağı biyopolimerlerin iletkenliğini artırmaktadır. XRD sonuçları, lif kabağının PANI/PEO filmlerine kıyasla biyo-kompozit filmlerin kristalliliğini artırdığını ortaya koymaktadır. FTIR analizi, film yapısında PANI, PEO ve lif kabağı fonksiyonel gruplarının varlığını kanıtlamıştır. Ayrıca, biyo-kompozit ince filmdeki lif kabağı ağırlığındaki artış, O-H grubunun pik yoğunluklarında artışa neden olur. Lif kabağının DSC analizinin sonuçlarına göre kompozitlerin termal kararlılığını artırdığı belirlenmiştir.

Anahtar Kelimeler: Luffa, PANI, Biyokompozit, İnce Film

I. INTRODUCTION

Due to environmental problems, the rate of use of biomaterials in industry is increasing considerably. Among them, fiber-reinforced composites (FRC) have gained great attention due to substantial mechanical properties such as flexibility, good strength, and low density, and environmental properties such as biocompatibility, biodegradability, and availability [1,2]. Owing to these properties, FRC has found a wide application in the automotive [3], marine [4], aerospace construction [5], and textile industries [6]. FRCs may contain synthetic fibers like carbon, glass, and Kevlar or natural fibers such as jute, kenaf, sisal, cotton and luffa. However, natural fibers are preferred over synthetic fibers due to easier recycling and lower environmental impact. Since it is difficult to dissociate non-natural fibers from composites, it brings about industrial waste.

Luffa cylindrica (Luffa) is one of the natural plant fibers composed of cellulose, hemicellulose, lignin, and waxy materials. It is classified in the cucurbitaceous family. It is common in Asia, Africa, and South America. Luffa, also known as a vegetable sponge, used in this study, was obtained in the southern part of Turkey. Luffa cylindrica (LC) has been used in a variety of investigations. LC has used printed circuit board (PCB) instead of glass fibers to eliminate recycling problems [7]. Sahli, M. et al. did a photocatalytic study with luffa cylindrical to investigate the structural properties of new composite [8]. Moreover, diverse bio-composites reinforced by luffa cylindrica have been fabricated in order to improve the physical, chemical, and electrical properties of materials [9-11]. For sensor applications, luffa cylindrica has been utilized as a sensing layer to investigate the sensing properties of devices [12]. Polyaniline (PANI) is one of the applicable conductive materials in the literature, such as gas sensors [13], humidity sensors [14], and biomedical devices [15] due to its easy synthesis, low cost, and easy transition from the conductor (emeraldine-salt) type to the insulator (emeraldine-based) type [16]. Generally, PANI is used with polyvinyl alcohol (PVA), polyethylene oxide (PEO), Poly (Vinyl Chloride) (PVC), etc. [17,18] since it has been difficult to obtain PANI as a self-contained film. For film or hydrogel film synthesis, the desired properties are solubility and bio-compatibility, and when compared to the others, PEO has been preferred more due to its excellent solubility in water, bio-compatibility, and non-toxicity [19]. For developing composite film, several methods are used to obtain smooth and homogeneous film, such as spin coating [20], dip coating [21], and solvent casting [22]. The most used method is the solvent casting method, which does not require any special apparatus to conduct an experiment [23]. To synthesize conductive polymers, several methods are used, such as solution mixing and impregnation method, in situ polymerization method, melt mixing method, and so on. Each method is applicable according to the study's purposes. For instance, to investigate the mechanical properties of the material, the in-situ polymerization method can be used [24], or to modify the surface properties of the composite, the solution mixing method is more convenient [25]. Also, for thermoplastic materials, the melt mixing method may be used [26].

In this work, PANI/PEO/LC bio-composite films were fabricated in different weight ratios of luffa in PANI/PEO copolymer by using the melt mixing method. This study aims to investigate the effect of luffa on conductive copolymer PANI/PEO composites on their structural, chemical, and thermal characteristics, which have not been extensively studied before by adding different weight ratios of it. Initially, cellulose of luffa cylindrica was extracted by means of an alkali treatment method. Then, PANI was polymerized with PEO, which was added to the solution to obtain a film. Finally, the treated luffa was added and stirred with PANI/PEO copolymer with different weight ratios in order to investigate the structural effects on the biopolymer film. To investigate the structural, chemical, and thermal properties of films, FTIR, XRD, and DSC analyses were conducted, respectively.

II. MATERIALS AND METHODS

A.1 Materials

The Luffa Cylindrica utilized in this work are from Hatay, Turkey. Ethanol, xylene, sodium hydroxide (NaOH), hydrochloric acid (HCl) were used for luffa treatment process. PANI-emeraldin base ($M_w = 50,000$), PEO ($M_v = 900,000$), camphor sulfonic acid (CSA) were used for PANI/PEO solution. PANI, PEO, CSA were purchased from Sigma-Aldrich and other chemicals were acquired as guaranteed-grade reagents from Merck and other commercial suppliers and utilized without additional purification.

A. 2. Methods

A.2.1. Luffa Treatment

The alkali treatment methods were utilized in the luffa cylindrica to eliminate hemicellulose, lignin and other waxy and gummy substances[27]. Before this procedure, luffa cylindrica slices were dried in a vacuum oven at 50 C for 8 hours. As illustrated in Figure 1, luffa cylindrica were cut into small pieces to place in the vibratory disc mill and powdered at 1000 rpm for a minute. At each chemical treatment steps, luffa was rinsed with distilled water and ethanol and then filtered with a vacuum motor. Firstly, 5g grounded luffa in a flask was stirred in xylene/ethanol (2:1, v/v) for 6 hours. After that, 2 M hydrochloric acid (HCL) was added into flask at 85 C for 8 h. After filtration, luffa was treated with NaOH for 2 h at 85 C and as a final step, hydrogen peroxide was put into flask for 6 h to remove all constituents and impurities from Luffa cylindrica. Then, after filtering treated luffa powder with distilled water at least twice to ensure the elimination of chemical substances and dried, the brownish treated luffa was attained as shown in Figure 1.

A.2.2. Preparation of PANI/PEO/Luffa Films

The PANI/PEO composite solution was synthesized via the melt mixing method. The polyaniline were dispersed in an aqueous solution mixture with CSA (1:1.5) in chloroform/DMF (1:1) and stirred for 24h in the flask. Then, 2% PEO was added to the PANI/Luffa solutions as a carrier polymer, and they were stirred 24 hours at room temperature. As a final step, the luffa cellulose powder (treated luffa powders) was added and mixed in PANI/PEO solution for 24 h to obtain the bio-composite solution. All the stirring process has done in room temperature and humidity (25 °C, % 35). For the bio-composite film structure; 4 samples were prepared as PANI/PEO (here in after referred to as PANI or P), 1PANI:1Luffa (1P:1L), 1PANI:2Luffa (1P:2L), 2PANI:1Luffa (2P:1L) with casting solution method. The Table 1 contains the ratios used in the samples.

Table 1. Weight ratio of luffa and PANI/PEO bio-composites.

Sample	PANI/PEO (%)	Luffa (%)	Chloroform/DMF (ml)
PANI/PEO	3	-	12
1P:1L	3	3	12
1P:2L	3	6	12
2P:1L	6	3	12

A. 2.3. Instrumentation

Endress + Hauser conductivity meter was used to measure electrical properties of biopolymer films. Fourier transform infrared (FT-IR) and X-ray diffraction (XRD) spectroscopies were performed to

analyse vibrational and structural properties of PANI/PEO/LF bio-composite films. Perkin Elmer Spectrum 100 series FT-IR spectrometer was used to obtain functional groups of films. For XRD analyses, Bruker AXS D8 Advance system was utilized in the 2θ range of 5° – 50° at room temperature. DSC measurements were conducted on a Perkin Elmer Jade DSC.

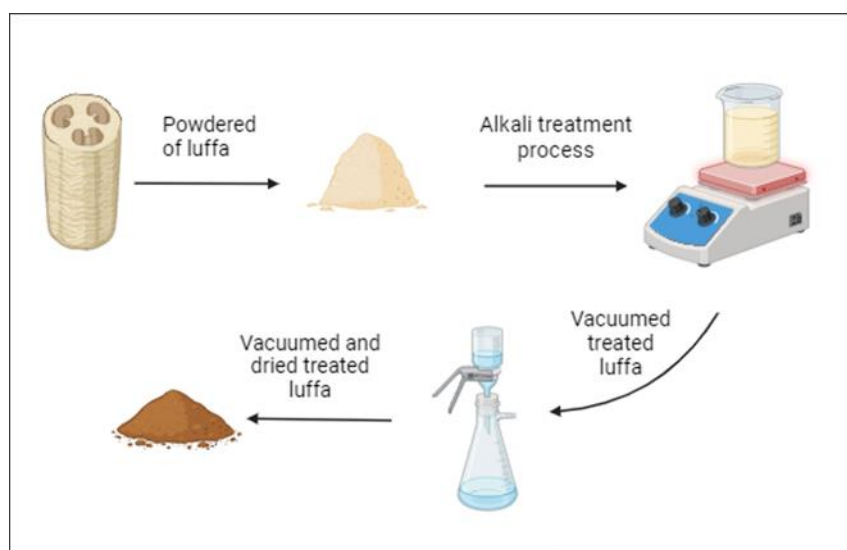


Figure 1. Schematic of extracting cellulose from luffa cylindrica.

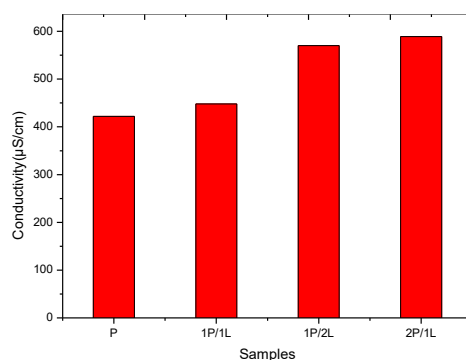


Figure 2. The conductivity value of bio-composite films with different luffa concentration

III. RESULTS AND DISCUSSIONS

The conductivity of the prepared bio-composite films was shown in Figure 2. According to the results, the conductivity values increase with the increasing ratio of PANI in the bio-composite as expected. However, the luffa ratio also improves the conductivity of bio-composite films. As shown in Figure 2, the conductivity value of 1P/2L is bigger than the conductivity of 1P/1L. This ascent may occur due to the plasticizing effect of blending polymers with macro molecules, bringing about an increase in conductivity at room temperature [9]. This increment may also be because of the chemicals that are used in the alkali treatment of luffa cylindrica.

Regarding structural analysis, firstly XRD was carried out with 35 kV power and a 30-mA current at room temperature in the angle range between 5° to 50° . The XRD spectroscopy results of PANI/PEO, 1P/1L, 2P/1L, 1P/2L and luffa are illustrated in Figure 3(a) and Figure (3b), respectively. For XRD pattern of PANI/PEO, the intense peaks were observed in $2\theta \cong 15.34^\circ$ (PANI), $2\theta \cong 20.32^\circ$ (PANI) and $2\theta \cong 26.66^\circ$ (PEO) as also reported in literature [28-30]. When adding luffa cellulose at different ratios

to the PANI/PEO copolymer, the peak intensities increase, and the peak widths decrease with the increasing the amount of luffa as shown in Figure 3(b). This behaviour means that luffa enhance the crystalline structure of PANI/PEO film [31]. As reported by Prabowo et al, treated ijuk fibers also increase the crystallinity of polypropylene composite[32]. The XRD results are also consistent with the conductivity of the bio-composite films, as seen in Table 1, the increasing of conductivity by adding luffa also increased the crystallinity of the films[33]. Our XRD results are also compatible with the study conducted by Awatef Dali et al. who fabricated PANI-coated luffa thin film by in-situ polymerization of anilin and obtained more ordered structure than PANI [34].

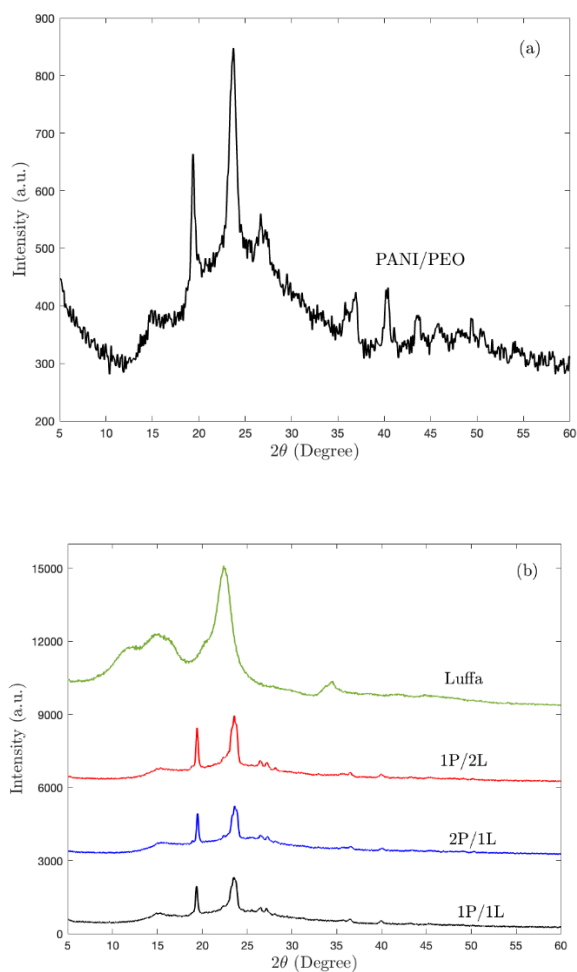


Figure 3. X-ray diffraction patterns of (a) PANI/PEO, (b) Luffa ,1P/2L , 2P/1L and 1P/1L films.

The surface composition of bio-composite films was investigated via FT-IR spectroscopy. Figure 4(a) illustrates the changing of the chemical structure of treated luffa (TL) with untreated luffa (UTL). The strongest intensity peaks were observed in the region 3340 cm^{-1} in relation to the -OH group for cellulose and hemicellulose and 2900 cm^{-1} in relation to C-H groups due to the aliphatic fragments of the cellulose and hemicellulose components[35]. The absorption at 1429 cm^{-1} indicates the presence of C=C double bonds in aromatic stretching in lignin. A band at 1029 cm^{-1} corresponds to the vibration of the C-O stretching bond carbon of cellulose and hemicellulose [36]. After treatment of luffa, decreasing peak intensities occur, meaning the reduction of the corresponding functional groups. For instance, the intensities of 3340 cm^{-1} and 2900 cm^{-1} peaks decrease fairly, as shown in Figure 4(a). Hence, Lignin and hemicellulose were successfully eliminated from UTL as also studied by Yong et al. [37].

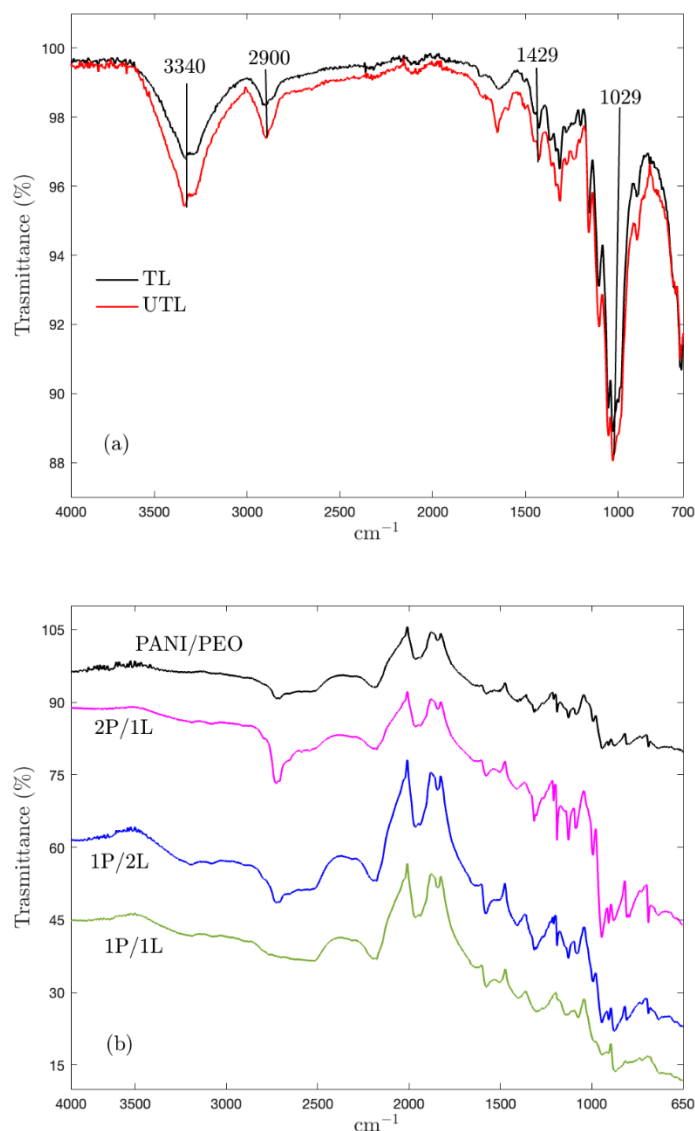


Figure 4. FT-IR spectroscopies of treated luffa (TL) and untreated luffa (UTL) (a) PANI/PEO copolymer, 2P/1L, 1P/2L, and 1P/1L bio-composite(b) films.

The comparison of functional groups of PANI/PEO, 2P/1L, 1P/1L, and 1P/2L biocomposite films was given in Figure 4(b). The composite film spectra are similar to the PANI/PEO film spectra. As shown in spectra (Figure 4b), the intensity of peaks increases when adding luffa to the PANI/PEO film. In addition, the peaks were shifted also a little from 2890 cm^{-1} to 2920 cm^{-1} and from 3382 cm^{-1} to 3347 cm^{-1} due to the chemical interaction of PANI/PEO with the luffa cellulose in Figure 3(b). Around 1700 cm^{-1} and 800 cm^{-1} regions, the transfer peaks occur on 1P/2L, 1P/1L, and 2P/1L owing to forming strong hydrogen bond between the luffa cellulose and the PANI/PEO co-polymer [38].

The thermal behavior of bio-composite samples was investigated with DSC measurements to determine the glass transition temperature (T_g) of film composites. Figure 5 illustrates the heating DSC curves with the glass transition of 2P/1L, 1P/1L, and 1P/2L films. In this figure, the highest T_g ($202,31\text{ }^\circ\text{C}$) belongs to 1P/2L film, and when compared to the other films, the T_g value decreases with the decreasing luffa ratio in films. In other words, it can easily be said that luffa increases the crystallinity of the film, as reported by P.V. Joseph et al.[39] and decreases the free volume of the composites, which means the elongation of copolymer chains and, at the same time, strengthens the intermolecular force between the

polymer chains[40,41]. Another important result of glass transition temperature is that composites with higher glass transition temperature (T_g) values typically exhibit higher melting temperatures[42]. Hence, the 1P/2L bio-composite film has greater thermal stability due to higher T_g (202.31 °C) and is the most applicable specimen, especially for sensor technologies. Moreover, the first large endothermic peaks occur (around 60-70 °C) in the DSC analyses of each composite because of the evaporation of water/solvent molecules during the heat process, and the peak temperatures are compatible when compared to the literature that is about 50-80 °C [43].

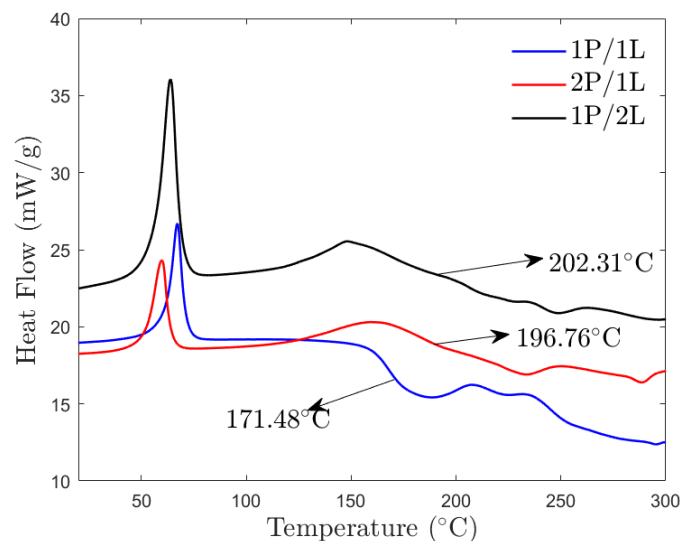


Figure 5. DSC thermograms of 2P/1L, 1P/2L, and 1P/1L bio-composite films.

IV. CONCLUSION

In brief, the structural, vibrational and thermal properties of bio-composites consisting of PANI, PEO, and Luffa cellulose with different weight ratios was investigated. It was confirmed by XRD spectroscopy that luffa-based cellulose can enhance the crystalline structure of PANI/PEO co-polymer by observing more intense peaks on 1P/2L bio-composite film. FTIR analysis proves the presence of functional groups of PANI, PEO, and cellulose in film structures. The alkali treatment successfully eliminated hemicellulose and lignin from the raw luffa. The DSC results show that cellulose extracted from luffa cylindrical increases the structure order of PANI/PEO film copolymer and makes films more crystallized, obtaining coherent outcomes with the XRD. Among synthesized bio-composite films, the 1P/2L is a good candidate that might potentially be used, especially in sensor technologies and other innovative functional materials, due to expressing better electrical properties as shown in Table 1 and thermal properties that has higher glass transition temperature ($T_g=202.31^\circ\text{C}$). The next step in this work will be to fabricate the PANI/PEO/Luffa bio-composite thin film using the spin coating method, characterize this new bio-composite in terms of its uniformity, homogeneity, and morphological properties using profilometry, SEM, or AFM, and test it for mechanical properties using mechanical test techniques.

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