Biologically Degummed and Chemically Treated Okra Bast Fibers-Reinforced Poly(Vinyl Alcohol) Composites

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ABSTRACT
This paper focuses on preparation of poly(vinyl alcohol) (PVA)-based composite films reinforced with okra bast fibers at different percentages of 5, 10, 20% via solution casting method. Fibers obtained from different sections of okra plants were biologically degummed and scoured with Na2CO3. Selected fibers were bleached, treated with maleic anhydride or grafted with vinyl acetate. Mechanical, physical and biodegradational properties of the composites were investigated. The tensile strength of the produced composites ranges between 33.8 and 55.1 MPa, elasticity modulus from 1.8 to 2.6 GPa, elongation rate at break varies in 2.8-10%. Chemical treatments led to improved mechanical performance whereas increased fiber content reduces tensile strength, stiffness and elongation, as well as water absorption. Fiber addition significantly affected biodegradation in a complicated way: by decelerating mass loss but accelerating deterioration of mechanical properties.

1. INTRODUCTION
Biopolymers and biocomposites have gained notable consideration over the last few decades. The main advantages of biopolymers are their biodegradability and the reduction of carbon dioxide emissions. Polyvinyl alcohol (PVA), is a kind of biodegradable and water-soluble polymer that can be utilized in an array of applications such as agricultural mulch films, biodegradable packaging, food coating, fuel cells, paper covering, textile sizing agents, etc. [1-3]. Due to its excellent processability and biocompatibility, it has found use in blends and composites with various natural polymers including starch, chitosan, cellulose, fibroin, or lignocellulosic fillers [4]. The hydrophilic PVA is compatible with lignocellulose fibers. –OH groups of natural fibers can link to the –OH groups of PVA via hydrogen bonds [5].

Lignocellulosic fibers are renewable and abundantly available materials on the globe composed mainly of cellulose, lignin, and hemicellulose. Those fibers can be obtained from traditional fiber crops as well as from agro-residual sources. Recently, agro-residual fiber sources get a priority to scientists because those crops serve two purposes simultaneously: food production and fiber production. Some agro-residual fibers obtained from agricultural residues such as okra stem [6], coir [7], banana fruit and bunch stems [8], corn husk [9], cotton straw [10], pineapple leaf [11], enset [12] etc. have proven their potential as a raw material in textiles and composites industries. Due to bearing high cellulose content, high tensile strength, stiffness, and aspect ratio; okra bast fiber has been used to make a composite with a variety of polymers viz. polypropylene [13], epoxy [14], light density polyethylene (LDPE) [15], etc. The composites of PVA and okra fiber may also have the prospective to produce materials with excellent mechanical properties and promising performance.

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The performance of PVA/lignocellulose fibers composites depends on processing methods, quality of filler, filler distribution, compatibility of filler and matrix, etc. Though both PVA and cellulose contain hydroxyl groups, their composites provide poor moisture resistance which reduces mechanical properties by destroying the bond between filler and matrix. Again, direct incorporation of virgin lignocellulose fibers into the PVA matrix may not always give good mechanical properties. In most cases, the –OH groups of cellulose involve either intra- or intermolecular hydrogen bonds with each other. Therefore, those cellulose molecules are not willing to react with matrix molecules, which is necessary for the mechanical strength of the composites. The strength of the composite can be improved in two ways: (i) by using cross-linking agents which increase their compatibility and (ii) by chemical treatments of fiber. For long-term durability of these composites, researchers have proposed many chemical modifications such as alkali treatment [16], plasma treatment [17], and surface modifications using silanes [18], maleic anhydride [19], vinyl monomers [20], etc. to decrease their moisture affinity.

Cinelli et al. [21] produced composites of PVA reinforced with other agro-residual materials that are obtained from corn-based ethanol production, i.e. corn fiber and corn starch, and some plasticizers including glycerol and pentaerythritol via injection molding. The corn fibers were from pericarp sections of the corn kernels. They reported tensile strength around 8 MPa, elongation rate between 400-600%, and Young’s modulus in the range of 36-100 MPa. They found that mechanical properties were sustained upon soaking for 30 minutes in water and in a medium at at 50% relative humidity and 23 °C for 1 year.

Imam et al. [22] developed cast films of PVA-based composites including cornstarch, orange fibers; and hexamethoxymethylmelamine (HMMM) as a crosslinking agent, as well as urea and glycerol as plasticizers. Orange fibers were lignocellulosic fibers obtained from remnants of orange juice production. They studied the thermal behavior, biodegradation, water permeability chemical structures of the composites.

Ching et al [23] produced PVA composites reinforced with empty fruit bunch fibers at 0.5-3 % loading percentages via casting evaporation. Empty fruit bunch fibers were obtained from oil palms. The researchers reported increased tensile strength and elasticity modulus at 1% fiber addition. The mechanical properties further increased with thermal treatment at 50 °C for 6 h. They reported a tensile strength range from 8.25 to around 15 MPa, elasticity modulus between around 10 MPa and around 35 MPa. They reported enhanced mechanical properties with reduced fiber size.

Ali et al. [24] produced kenaf fiber-reinforced PVA composites via solution casting at fiber loading percentages 2.5, 10, 15, and 20%. They applied chemical treatments on the fiber which resulted in better tensile properties. Fiber addition as well as chemical treatments led to better flexural performance. The attained tensile strength values range between app. 9-18 MPa, elasticity modulus app. 36-65 MPa, and elongation rate app. 50-280%.

Parvin et al. [25] developed fibrillated cellulose fiber-reinforced PVA composites. They reported increment in tensile strength and elasticity modulus, together with decrease in elongation rate and maximum yield force in the composites upon addition of fibrillated cellulose fibers in PVA. The paper also ascribed the impact of p-phenlenediamine treatments of fibrillated cellulose fibers.

In the present investigation, fibers were obtained from different sections of okra stem via biological degumming and and scoured with Na2CO3 to prepare finer fibers by removing non-cellulosic matters. Selected okra bast fibers were then bleached, treated with maleic anhydride or vinyl acetate prior to composite preparation. The prepared composites were characterized in terms of their mechanical, physical and biodegradational properties. The effects of plant location where the fibers were extracted, and the effects of chemical treatments of okra fiber on the properties of their PVA film composites have been thoroughly investigated.

2. MATERIAL AND METHOD

2.1 Material

Okra stems were collected from a local agricultural farm in Denizli providence, Turkey. Maleic anhydride and vinyl acetate monomers were purchased from Sigma-Aldrich. Polyvinyl alcohol 72000 powder (98% hydrolyzed) was supplied from Merck, Germany. Solvents and reagents used in the investigation were analytical reagent grade.

2.2 Method

Okra bast fiber extraction and chemical treatments have been conducted as explained in the previous study [9] and is summarily described as follows:

Okra stems were divided into three different parts: upper, middle and bottom portions and kept separately in plastic bottles for water retting for 2 weeks. Then, the retted fibers were washed by running water and dried in ambient air. Following this, the fibers were scoured in 3.5g/L Na2CO3 and 6.5g/L soap solution at 70 °C for 30 min at a fiber to liquor ratio of 1:50 (w/v).

Some of the scoured bast fibers were alkalinized in a 10 g/L NaOH solution at a fiber to liquor ratio of 1:50 (w/v) for 3 h at room temperature. Then the fibers were washed thoroughly and neutralized in a dilute acidic solution.

Another portion of the scoured fibers were bleached with 0.7 wt% sodium chloride (NaClO2) solution for 90 min at 85-95°C at a fiber to liquor ratio of 1:50 (w/v). Throughout the process pH was maintained at 4 by using a buffer...
mixture of pH 4 (acetic acid-sodium acetate) at a buffer solution to chlorite solution proportion of 1:10. After completion of bleaching, the fibers were washed thoroughly with distilled water and then treated with 0.2% (w/v) sodium meta-bi-sulfite (Na₂S₂O₅) solution for 20 min at a fiber to liquor ratio of (w/v). Finally, the fibers were filtered and washed thoroughly with distilled water, and dried in ambient air.

Some of the bleached fibers were treated 1% maleic anhydride (MA) in toluene at 65°C for 2h. After completing reaction, fibers were refluxed 3 times by acetone and finally dried in an electric oven.

The grafting reaction of vinyl acetate (VA) on a portion of bleached okra bast fibers was carried in N, N Dimethylformamide (DMF) at 95°C, 50 wt% VA monomer and 5 wt% pyridine were taken on the basis of fibers weight. The reaction was continued for 3 h in water bath and then refluxed by acetone. After washing several times by acetone, fibers were dried in an oven.

As reported in the previous study [9], alkalization led to decrease in linear density, tensile modulus and tenacity and increase in moisture content and breaking elongation of fibers. Effect of alkalization was more pronounced in comparison to other fiber treatments. The properties of water retted and scoured fibers can be given as linear density (10.24, 9.97, 6.97 tex), moisture content (6.79, 5.38, 6.25%), initial modulus (1557, 1823, 1876 cN/tex), breaking tenacity (28.78, 32.70, 31.22 cN/tex) and breaking elongation of(6.25%), initial modulus (1557, 1823, 1876 cN/tex), breaking tenacity (28.78, 32.70, 31.22 cN/tex) and breaking elongation (1.99, 2.02, 2.03%) in the order top, middle and bottom, respectively. Those of alkalized, bleached, maleic anhydride-treated and vinyl acetate-grafted fibers are listed as linear density (8.26, 9.53, 9.04 and 10.24 tex), moisture content (7.11, 3.82, 5.44 and 3.62%), initial modulus (1044, 1862, 1784 and 1677 cN/tex), breaking tenacity (29.14, 40.69, 24.85 and 32.65 cN/tex) and breaking elongation (3.86, 2.23, 1.54 and 2.20 %), respectively [9].

Preparation of okra bast reinforced PVA composites can be explained as follows: Okra bast fibers were cut into the possible smallest size by scissors and then smashed in a ceramic mortar. PVA was dissolved in distilled water with continuous stirring using a magnetic stirrer for 1-1.5 h at a temperature around 80°C to prepare a PVA aqueous solution. The PVA and water ratio was maintained as 1:20. Pre-calculated amounts (5, 10, 20 wt% fiber with respect to PVA) of fibers were then added to the PVA solution (Table 1). The stirring was performed at the same temperature for a further 30 min. The final mixtures were then cast on a wooden mold. The films were kept to dry at room temperature for 1-2 days and then taken off from the mold. The films were dried at room temperature for a few weeks to evaporate the remaining water. Neat PVA film was also prepared in the same fashion but without the addition of fiber as the reference sample. Before characterization, composites were sized and dried in an oven at 80 °C for 2.5h.

Before characterization (in terms of density, mechanical properties and moisture content properties), the samples were conditioned at 21°C and 65% relative humidity for at least 24 h.

Densities of the composites have been determined by measuring the dimensions of the samples using a mechanical caliper and obtaining their mass by using a mechanical caliper and obtaining their mass by using a mechanical caliper and obtaining their mass by using a mechanical caliper and obtaining their mass by using a mechanical caliper and obtaining their mass by using a mechanical caliper and obtaining their mass by using a mechanical caliper and obtaining their mass by using a mechanical caliper and obtaining their mass by using a mechanical caliper. For density measurement, the formula: 

\[
\rho = m/V,
\]

where \(\rho\) is the density in (g/cm³), \(m\) represents mass (g) and \(V\) stands for volume in cm³.

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<th>Treatment#2</th>
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<th>PVA content (wt%)</th>
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Table 1. Processing parameters for okra fiber-reinforced PVA composites.
The tensile properties of neat PVA and composite samples were evaluated by tensile tests, performed, as prescribed by ASTM D 638, on rectangular samples 100 mm × 10 mm. The test was carried out by using Tinius Olsen H10KT(R) Tester, US with QMat for Textiles(R) software, and the initial grip separation was 50 mm long. The load was measured by using a 500 N cell, and applied in displacement control with a crosshead speed of 5 mm/min. The measurements were performed at room temperature and at least 7 samples for each formulation were tested.

The water absorption behavior was measured as per ASTM D570-98 (2010). Specimens for each sample composite were in dimensions of 100 mm × 10 mm × (0.24 - 0.48) mm. The specimens were dried in an oven at 80°C for 2.5 h prior to testing. The specimens were weighed before being soaked in water. The specimens were immersed in water at room temperature (approximately 25°C). After 24 h, 48 h, and 72 h, specimens were taken out of the water and weighed once moisture on the surface moisture was dried with tissue paper. The moisture absorption was determined according to Eq (2):

\[
\text{Water absorption} \% = \left( \frac{W - W_0}{W_0} \right) \times 100, \quad (2)
\]

where \(W\) and \(W_0\) is the initial weight and the weight after water absorption, respectively.

Soil degradation test has been carried out according to soil burial degradation experiment explained in [26]. Accordingly, the composite samples were cut into 100 mm × 10 mm rectangular specimens. Degradability test was carried out in a plastic container (44×30×23 cm³) containing farmland soil having pH 7 by maintaining high relative humidity (90-95%) via sprinkling water at room temperature (25±5°C) daily. The specimens were dug out from the farmland soil after 14 days, and then washed and dried in air for 3 days and thereafter at 80°C for 2.5h in an oven. Biodegradability was measured by determining weight loss and deterioration in tensile properties of composite samples. Three specimens were tested for tensile properties of each biodegradation sample. Weight loss was calculated as follows:

\[
\text{Weight loss} \% = \left( \frac{W_0 - W}{W_0} \right) \times 100, \quad (3)
\]

where \(W_0\) and \(W\) is the initial weight and the weight after soil degradation, respectively.

The findings were statistically interpreted by applying analysis of variance (ANOVA) test with \(\alpha\) at 0.05 significance level.

Scanning electron microscopy (SEM) of fracture surfaces of okra bast fiber-reinforced PVA composites were obtained via use of a Zeiss EVO LS 10 scanning electron microscope, at 20 kV voltage and ×100 and ×300 magnification conditions. Composites were gold coated at a thickness of 5 nm via use of a Cressington auto 108 model sputter coater.

3. RESULTS AND DISCUSSION

3.1 Density

Density of neat PVA was measured as 0.85 g/cm³. Density of poly(vinyl alcohol) is reported to be 1.19 - 1.31 g/cm³ in the literature [27], whereas the bulk density of PVA is reported as low as 0.40-0.67 g/cm³ [28]. The measured density falls between the density and bulk density of PVA. The bulk density depends on the crystallinity and packing density of the polymer and is lower than that of a single polymer chain [29]. In the composites, it is seen that addition of fibers led to slight decrease in composite density, as shown in Figure 1. Significant effect of fiber loading and fiber treatments on composite density has been detected (p values 1.27 × 10⁻¹³ and 0.01, respectively). Additionally, the location in the okra plant where the fibers were extracted also had significant effect on density (p value 0.0001).

3.2 Mechanical properties

Mechanical properties of composites are of prime importance and determine in which applications they can be utilized [30]. Increase in tensile strength was observed upon 5% loading of (wt) maleic anhydrite- and vinyl acetate-treated okra bast fibers (Fig 2). Findings of modulus of elasticity also agree with tensile strength data (Fig 3). On the other hand, elongation at break substantially decreased with fiber addition. The tensile strength and elasticity modulus values of the current study are greater than those of cellulosic fiber-reinforced PVA composites reported in the literaure [21], [22], [24].

![Figure 1. Measured density of 100% PVA and the okra bast fiber-reinforced PVA composites](image-url)
Fiber loading and fiber treatments were found to have significant effect on tensile strength. While the part of plant where the fibers were extracted, was not (p values 1.09×10^-6, 7.8×10^-4, and 0.19, respectively). For all batches tensile strength is in descending order for 5%>10%>20% fiber loading. Fiber loading at rates lower than 5% can be tried for future research. Rahman et al [31] reported increased mechanical strength and stiffness of PVA-based composites until a loading of 9% crystalline cellulose of jute and reduction in mechanical performance for higher loading percentages due to possible agglomeration. Ali et al [24] reported decreased tensile strength of PVA composites with increase in reinforcing kenaf fiber percentage. In the current study chemically treated okra bast fibers tend to result in higher strength in comparison to scoured okra bast fibers. Similarly, Ali et al. [24] reported improved strength from chemically treated kenaf fiber-reinforced PVA composites. The section of the plant where the fibers were extracted was not found to have significant effect on tensile strength. The reason for this may be the fact the differences in mechanical properties among fibers obtained from different parts of the plant are lower than those among differently treated fibers. In the previous study [9], increased breaking was reported increased tenacity upon bleaching and reduced breaking tenacity for alkalization and maleic anhydride treatment for okra bast fibers. Maleic abhydride-treated fibers showed the lowest tenacity and breaking elongation among differently treated fibers, whereas maleic anhydride-treated okra fiber-reinforced composite films show the greatest strength among studied composite films. Tensile findings of the composites and fibers can be interpreted as the surface chemistry of the fibers, and in turn compatibility with the matrix, are reflected more in the strength of the produced composites than differences in fiber tenacity.

In elongation at break testing, three factors were found to have significant effects (p values 3.19×10^-5, 1.08×10^-4, and 1.73×10^-6, respectively). Breaking elongation rate has been recorded as 97% for neat PVA. In general, increasing fiber loading results in decrease in elongation as seen in Fig 3. This is due to the fact that okra bast fibers show much more brittle nature with lower elongation rates in comparison to PVA. Similarly, Santi et al [32] reported decreased elongation break based on increased cellulose fiber load, Ching et al [23] on increased empty bunch fiber percentage, Ali et al. [24] on higher kenaf fiber content, and Cinelli et al [21] upon addition of corn fibers in PVA composites. The cellulosic fibers restrict mobility of PVA macromolecules as reflected in elongation reduction. Even though alkalized fiber showed nearly twice the elongation of other fibers, this phenomenon cannot be observed in the composite films.

![Figure 2. Maximum tensile strength (MPa) values of 100% PVA and the okra bast fiber-reinforced PVA composites](image)

![Figure 3. Modulus of elasticity (MPa) of 100% PVA and the okra bast fiber-reinforced PVA composites](image)
3.3 Water absorption

When water absorption behavior of okra bast fiber-reinforced PVA composites are investigated, it is seen that addition of okra bast fiber systemically reduces water absorption with increased fiber loading (Fig 5). This is due to the fact that water absorption of okra bast fibers are lower than that of PVA. No significant effect of immersion time could be obtained in the test duration range (p value 0.78). Water absorption rate was significantly affected by fiber loading, whereas no significant effect of fiber treatments or plant part of fiber extraction was detected (p values 0.0001-0.002, 0.39-0.58, 0.16-0.29, respectively). Most of the time, water absorption decreases with increase in fiber loading. This hints water absorption can be controlled via addition of different percentages of fibers. Similarly, Ali et al. [24] also reported decreased moisture uptake with increased kenaf fiber load in PVA composites in humid environment. In the durations the test concluded, no mass loss due to the water solubility of PVA was observed. Cinelli et al. [21] reported 19% water uptake upon 30 minute water soaking. They did not provide data for longer durations. For further research, water absorption of okra bast fiber-reinforced PVA composites can be investigated at shorter and longer durations to monitor water absorption behavior.
3.4 Biodegradation

Neat PVA and selected composite samples with promising mechanical properties were subjected to biodegradation in soil. A universal effect of okra bast fiber addition on biodegradation of composites as reflected in weight loss and deterioration of tensile properties, has not been detected. When weight loss due to biodegradation is investigated, it is seen that fiber loading has significant effect, whereas fiber treatment does not (p values 0.02, and 0.11, respectively). It is seen that, other than bleached fibers, all okra bast fibers resulted in decrease in weight loss of PVA composites (Fig 6). This result hints that okra bast fibers can be utilized to control PVA degradation. Okra bast fibers may form backbones which maintain integrity of the composite films. Among differently treated fibers, alkalized fiber-reinforced composites show the least weight loss. Alkalization separates extracellulosic materials from the fibers, which may in turn act as diminishing nutrition of biodegrading organisms and slowing weight loss. 14-day soil degradation resulted in 5.00-9.65% weight loss in the studied composites. Imam et al. [22] reported 41-57% weight loss for 120-day soil burial testing which is a much longer duration compared to the current study.

It is seen that biodegradation process has significant effect on tensile strength (Fig 7) and modulus of elasticity (Fig 8) of composites, whereas not on elongation at break (p values 1.82×10⁶, 7.25×10⁻¹⁷ and 0.80, respectively). On the other hand, soil degradation substantially decreases ductility of neat PVA with a 70% reduction in breaking elongation, while generally increasing elongation of fiber-reinforced composites (Fig 9). Fiber-reinforcement led to increased strength and modulus deterioration, where the effect is dramatic in case of strength loss. The results may be linked to the interaction between PVA and the okra bast fibers. The okra bast fibers may form channels for biodegrading effects to take action. Together with maintaining integrity, the cellulose chains may face scissions which negatively affect mechanical properties. Imam et al. [22] also reported none to negative effect of lignocellulosic fiber loading on PVA composite weight loss upon soil burial. On the other hand, they found positive effect of CO₂ production during soil burial which reflects the rate of biodegradation. Tan et al. [33] proposes that presence of lignocellulosic fibers forms nutrition to PVA-biodegrading microorganisms (reported to be 55 species), hence, accelerating biodegradation process. The findings show that the studied composite films exhibit biodegradable structure. Longer-duration biodegradation tests with measurements at selected intervals may be conducted for future research to obtain better understanding of the biodegradation behavior of okra bast fiber-reinforced PVA composite films.
3.5 Morphology

In Figure 10, fracture surfaces of okra bast fiber-reinforced PVA composites are presented. In the images, it is seen that fibers tend to lie parallel or perpendicular to the fracture plane. Fiber-rich and fiber-poor regions can be detected. Fiber pull-out observed in fracture surface reflects the potential to increase mechanical properties via improving fiber-matrix interface bonding.
4. CONCLUSION

In this study, poly(vinyl alcohol) (PVA)-based composite films were obtained via solution casting and reinforced with different percentages of okra bast fibers subjected to different chemical treatments. Mechanical, physical, and biodegradation properties of the composites were investigated. The tensile strength, elasticity modulus and elongation at break values of obtained composites range between 33.8 - 55.1 MPa, 1.8 - 2.6 GPa, and 2.8-10%, respectively. Chemical treatments led to improved mechanical performance whereas increased fiber content reduces tensile strength, stiffness and elongation, as well as water absorption. Among different treatments, maleic anhydride- and vinyl acetate- treated fiber-reinforced composites reported the highest tensile strength and modulus. Surface chemistry of fibers affect the tensile properties of the composite films more than the tensile properties of the fibers. Okra bast fiber addition significantly affected biodegradation in a complicated way: by decelerating mass loss but accelerating deterioration of mechanical properties. Okra bast fiber-reinforced PVA composite films show biodegradable nature for potential use in packaging and agricultural applications.

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