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Research Article

Argeli bast fiber as wonder reinforcing agent for biodegradable polymer composites

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Abstract

This paper has aimed to concisely discuss the insight into the potential of the Argeli bast fibers as reinforcing agents in eco-friendly polymer composites. The Argeli fibers used in this work as biofiller were prepared by mechanical disintegration of the sun-dried Argeli bast fiber bundles followed by chemical treatments and hence incorporated into the matrix of the PLA/PBAT blend *via* melt compounding. The materials were characterized by advanced analytical tools such as tensile and Shore D hardness testing, and optical and electron microscopy. The Argeli fibers, originally comprising the compact bundles of microfibrils glued together, were found to exfoliate into constituent microfibers and distribute uniformly in the PLA/PBAT blends matrix upon melt processing. The addition of Argeli fiber into the PLA/PBAT mixture led to a reinforcement of the polymeric matrix with an increase in the tensile modulus as well as Shore D hardness, the properties being further enhanced with chemical treatments of the fibers. The latter enhancement in properties was attributed to the chemical treatment-induced formation of a highly crystalline pure cellulosic framework due to the dissolution of the amorphous parts as well as other impurities from the neat fibers. The Argeli fibers exhibited a potential reinforcing agent for the biodegradable polymer composite.

Keywords: Argeli fiber, morphology, PLA/PBAT blend, polymer composites, shore hardness

Introduction

Plastics are utilized extensively in many different industries due to their remarkable properties: which include being strong, resilient, resistant to light and chemicals, and good for a wide range of temperatures. Due to these properties and their affordability, plastics are now in high demand around the world, with 400 million metric tons being consumed annually (Devasahayam et al., 2019). It eventually results in a large amount of plastic waste being discarded in the environment (Chaiwutthinan et al., 2019; Hernández-López et al., 2019). Among commodity plastics, polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC), polyethylene terephthalate (PET) and polystyrene (PS) are the most commonly used conventional polymers (Andrady & Neal, 2009). Mostly these synthetic plastics are non-biodegradable, atmospheric resistant, and endure a long time in the natural environment; resulting in massive volumes of waste that lead to serious ecological, economic, and health problems (Weng et al., 2013). Thus, new biodegradable environmentally friendly, cost-effective, renewable resource-based alternative plastic materials have been searched.

As a result of increasing environmental consciousness, the industrial and academic sectors have supported the use of green, biodegradable, ecologically friendly, natural resource-based plastic and composite materials as an alternative to conventional plastics. Starch, polylactic acid (PLA), polyhydroxyalkanoate (PHA), polyhydroxy butylene succinate (PBS), and polycaprolactone (PCL) are a few examples of biodegradable plastics, whose chemical and physical properties deteriorate, degrade, and finally materialized into CO_2 and H_2O when exposed to the microorganisms (Tokiwa et al., 2009). These plastics undergo biodegradation owing to the presence of hydrolyzable ester bonds and play a major role over non-degradable plastics. Among biodegradable plastics, PLA is a linear, most promising, economically viable, biobased, biodegradable thermoplastic polyester that potentially substitutes for oil-based polymers in a variety of industrial applications (Madhavan Nampoothiri et al., 2010). It is obtained from plant resources such as starch, and rice by fermentation process. In contrast to other degradable polymers, PLA not only completely degrades under specific conditions but also offers several advantages including low cost, exceptional mechanical strength, excellent clarity, suitable barrier properties, and ease of processing (Wu

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et al., 2023). Commercially interesting these polymers have some unique properties like a glossy appearance; high rigidity and good transparency that make PLA the best polymer for various applications including packaging, textiles, films, and many more. However, low impact strength, low heat resistance, easily hydrolyzable, and high brittleness properties of PLA limit their applications in food packaging, biomedical devices, and automotive components (Chaiwutthinan et al., 2019; Weng et al., 2013).

To overcome those commercial limitations and to enhance its native characteristics, PLA has been blended with different polymers such as PBAT, PBS, PHA, and PHB which is also a completely biodegradable polymer (Dhakal et al., 2022a; Dhakal et al., 2022b). There are several reports concerning the biodegradation behaviour of the PBAT (Pokhrel et al., 2021; Giri et al., 2019) and well as the PLA (Fukushima et al., 2009) when used both as a single and one of the components of the composites or blends. However, the mixing of PLA with another biodegradable polymer (such as PBAT) leads to a decrease in the ductility. To compensate for the loss in ductility while keeping the biodegradability of the polymers, the blending of polymers with natural fibers such as jute, hemp, kenaf, sisal, bamboo, coir, pineapple, etc. could provide a reliable option (Adhikari et al., 2012; Al-Itry et al., 2012; Giri et al., 2019). Among the various natural fibers, Argeli *(Edgeworthia gardneri)* bast fiber is one of the potential fibers for the development of green polymer composites. In this context, we have reported the detailed structure-properties relationship of Nepal's one of themost important natural fibers, the Argeli particularly focusing on their physicochemical properties and their response towards thermal and mechanical stresses in a recent study (Gautam et al., 2024). The present work aims to report the first study on the mechanical reinforcing ability of the Argeli bast fibers in completely biodegradable polymer blends.

Materials and Methods

Materials

Argeli Fibers: The raw materials for natural fibers preparation, the Argeli bast fibers, were collected from the Bhumika Handmade Paper Udyog, Gokarneshwor - 8, Kathmandu, Nepal. The bast fibers are traditionally prepared from the bush stem, separating the fiber bundles mechanically using a metal strip, washed with water, and sun-dried and stored as bundles before their use for paper-making purposes (see Fig. 1).

The dried neat Argeli bast strip was chopped into about 5 cm long pieces and finely powdered using a laboratory grinder, filtered through sieves with mesh size 250 μm, dried in a hot air oven at 80 °C and stored in a dry place in polyethylene bags. The powder was further subjected to caustic soda and hydrogen peroxide treatment to expose the crystalline cellulosic fibers with the removal of amorphous parts such as lignin, hemicellulose, wax, etc. (Gautam et al., 2024).

PBAT: Poly(butylene adipate-co-terephthalate) (PBAT), a completely biodegradable polymer, with the trade name *Ecoflex® F blend C1200* and having a density, melting point, and average molecular weight of 1.25-1.27 g/cm^3, ca. 120 °C and 52.1 kg/mole, respectively was provided by BASF Ludwigshafen, Germany.

PLA: The biobased and biodegradable polylactic acid (PLA), a commercial product with the trade name *Luminy® LX175* and having a density, melting point, and average molecular weight of 1.24 g/cm^3, 155 °C, and 245 kg/mole, respectively was supplied by Total Energies Corbion, Gorinchem, The Netherlands.

Chemicals: The laboratory-grade chemicals such as acetic acid, sodium chlorite, sulfuric acid, acetone, and sodium hydroxide pellets were purchased from Hi-Media Laboratories Pvt. Ltd. A-516, Mumbai, India, and used without further processing.

Sample preparation

The fibers, and polymer granules were dried separately using a special dryer operated at 80 °C for 12 h. The dry pellets of PLA and PBAT were mixed in the weight ratio of 45:55 and melt-mixed with different proportions of Argeli fibers (such as 10, 20, 30, and 40 by wt.-% of total polymer, named as POLY) using a Twin-screw extruder (LTE20-44/00, Lab Tech Engineering Company, Ltd, Germany) operated at 170 °C with the screw speed of 120 rpm. The mixtures were pelletized, air-cooled, and subjected to compression molding at 180 °C and 15 bar pressure for 6 minutes.

Characterization techniques

The surface and fracture surface morphologies of fibers and composite samples were investigated using a JEOL JSM-6360 SEM operated at an accelerating voltage of 15 kV. The specimen surface was sputter-coated with a thin film of gold or carbon before the SEM imaging.

Tensile Testing: The tensile tests of the composites were performed using a Universal Testing Machine (Zwick Roell GmbH & Co. KG, Ulm, Germany) according to ISO 527 at 23 °C to determine the mechanical parameters including tensile strength, elongation at break, and tensile modulus. Each composite was tested 5 times and the average value of the mechanical parameter was calculated. The Young's modulus and tensile strength are determined by slopes of the initial part of the corresponding stress-strain curves and the maximum stress level attained by the materials, respectively (see Fig. 2).

Figure 1 Photographs showing the Argeli bush growing in the village of Sankhuwasabha district, Nepal (a) and a sun-dried bundle of mechanically isolated Argeli bast fibers (b)

Shore D Hardness: The hardness of the composites was assessed following the ISO 868 standard using a Shore D Hardness Tester (Zwick Durometer, Germany). The thickness of the test specimen was 6 mm. In this test, an indenter fitted with a calibrated spring is penetrated into the material surface (see the scheme in Fig. 2) and the hardness of the material is determined by comparing the measured values with the standard ones according to ISO 868.

The overall experimental design for the proposed research work is presented in Fig. 2. The required amount of the dry mixture of the polymers and the fiber is fed into the extruder and subjected to melt mixing at 170 °C and the resulting pellets are subjected to compression molding. The specimens for different tests are then prepared from the molded plaques.

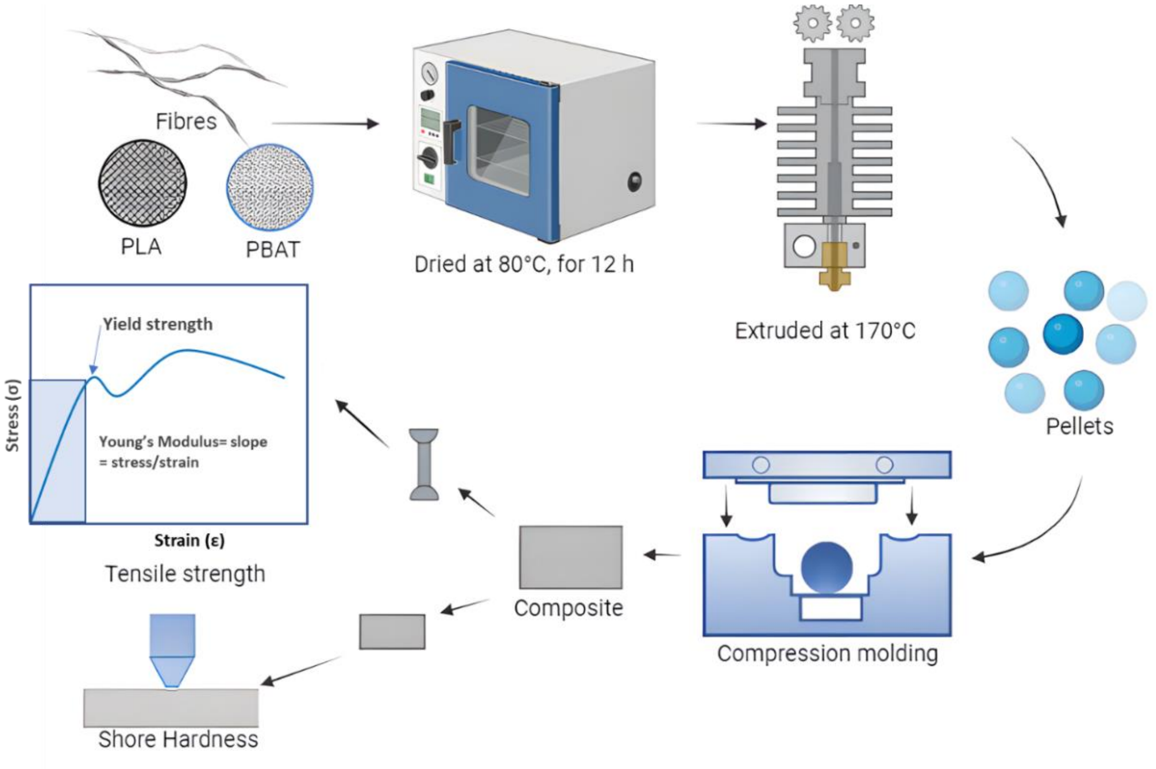

Figure 2 Schematic illustration showing the experimental design for preparation of polymer composite and their analyses (prepared partly using the BioRender software)

Results and Discussion

Fig. 3 presents SEM micrographs of the Argeli bast fibers in different magnifications. It can be observed that the fibers do constitute not individual entities but comprise an integrated bundle of a large number of fibrils, several tens of μm thick and with a length extending to a larger length scale, that is not possible to measure in the micrograph. The fibrils seem to have adhered to each other in the bundle, the adhesion being provided by the amorphous part of the natural fibers (Peterlin & Ingram, 1970; Heredia-Guereero et al., 2014). The thickness of the individual fibril can be estimated in the micrographs as being in the range of 5-10 μm.

The higher magnification image of the Argeli bast fibers shows (Fig. 3b) that the individual fibers are further composed of much thinner fibrillated structures, with a thickness in the range of 100 nm, which run parallel along the fibril's directions. Such structures are considered nanofibrils organized in the hierarchical structures of the lignocellulosic frameworks (Inoué & Leblond, 1986).

It can be thus concluded that the Argeli fibers in the neat state manifest the hierarchical structural details ranging from macroscale (in bast fiber bundle) to microscale (as in microfibers as in Fig. 3a) to the nanoscale, which ultimately is used to generate fabricate the high-quality handmade papers that the local people used to fabricate for hundreds of years in Nepal's hills.

It should be noted that the natural fibers subjected to different physical, chemical, and mechanical treatments may undergo several structural changes in different length scales. The treatment disintegrates the fibers into smaller fibrils and fragments and converts them to microcrystalline and nanocrystalline cellulose. In particular, the caustic alkali treatment and bleaching with various chemicals lead to the structural disintegration of lignocellulosic substances dissolving the loosely bound amorphous constituents and exposing the crystalline structure of the cellulosic framework (Husnil et al., 2019).

Interestingly, no intact bundled Argeli bast fibrils have been disintegrated into individual fibrillar morphologies that are little uniformly distributed into the polymer matrix when melt processed via extrusion followed by compression molding at elevated temperature. This deviation in fiber morphology in the blends of the polymers implies that the Argeli fibers get detached from the bundles due to thermal and mechanical stress exerted during the melt processing of the composites. One typical observation is exemplified by the fracture surface morphology of a melt-processed composite comprising 80 parts by weight of PLA/PBAT blend (POLY) and 20 wt.-% of the neat Argeli fibers (Fig. 4).

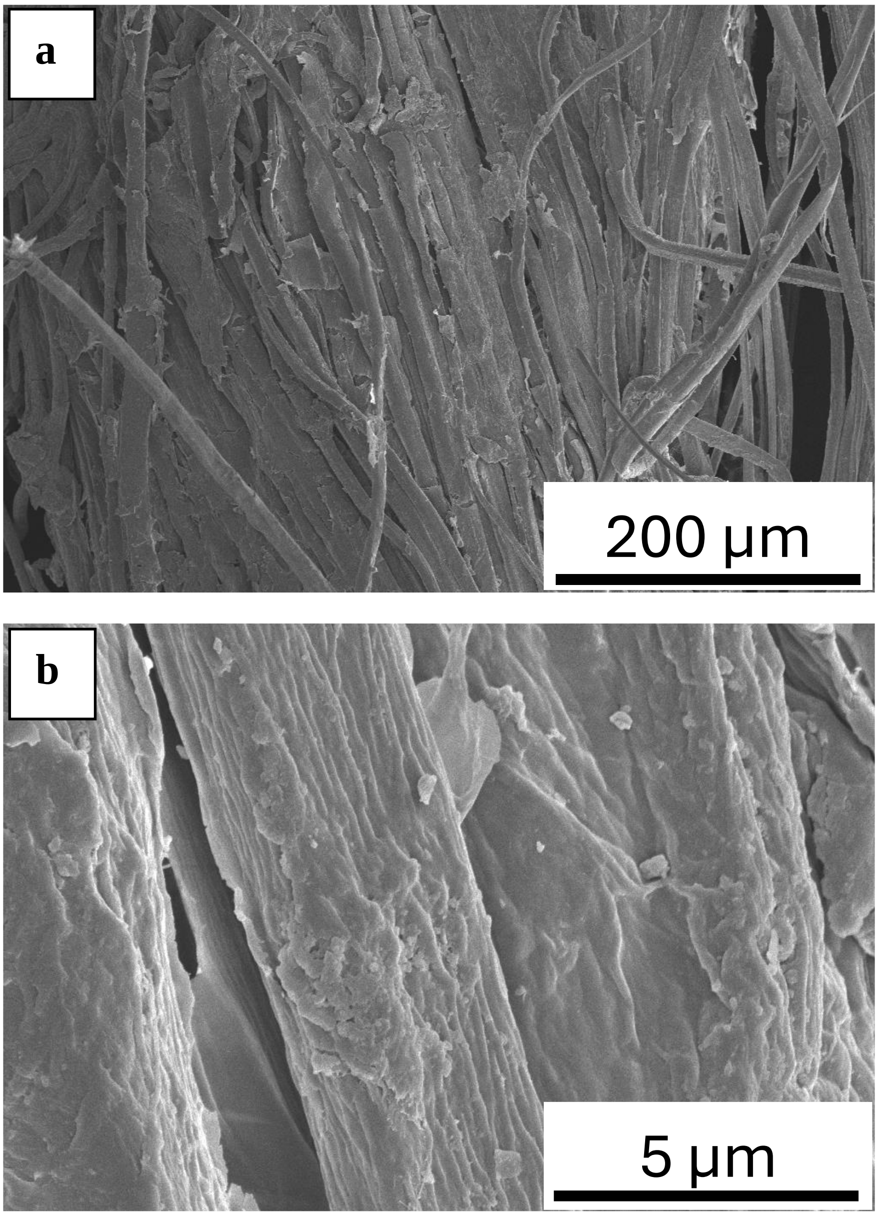

Figure 3 Lower (top) and higher (bottom) magnifications of the scanning electron micrographs showing the detailed morphology of Argeli fiber

The long, dark strips pointing outwards from the fracture surface appearing like dry wood branches are the well-separated Argeli fibers dispersed in the matrix of the polymer, which appear as rough surfaces in the background of the images presented in Fig. 4. The fibrillated rough surface of the polymer on the background of the SEM micrograph also hints at the notion of large plastic deformation of the polymer matrix.

The ease of the dispersion of the fibers, which appear as individual cylindrical hairs throughout the matrix with quite uniform distribution suggests that there is an affinity between the polymer matrix at hand and the fiber ensuring the mechanical reinforcement of the polymer blends. However, the pulling out of the fibers from the surface during the fracture of the polymer composite sample implies the high degree of incompatibility between them and the need for compatibilization at the polymer/fiber interface (Sobczak et al., 2013). It can indeed be expected that, besides the entropic constraints of high molecular mass polymer and fiber, the hydrophilic fibers are immiscible with a hydrophobic polymer blend, i.e. POLY.

After analysis of the primary morphology of the fibers and the composite materials, also with variations in the chemical treatment methods of the fibers and the filler content in the polymer composite, the mechanical properties were evaluated comprehensively.

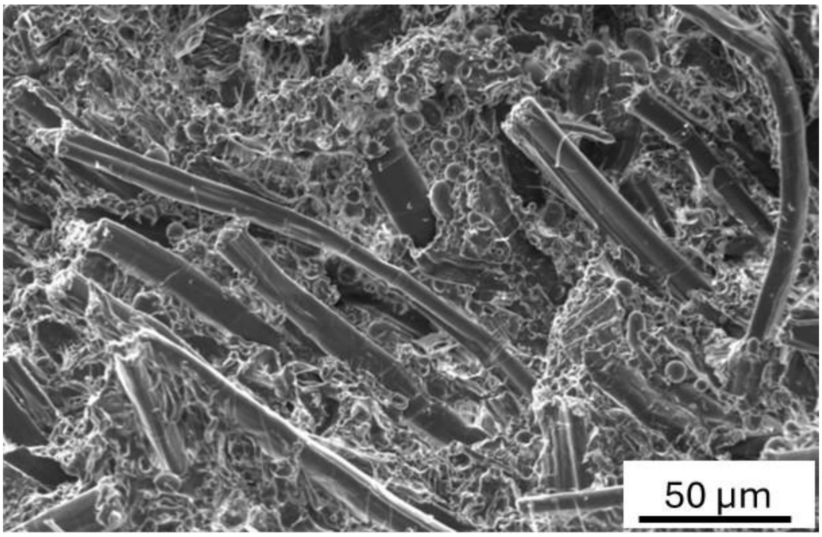

Figure 4 Representative SEM image showing the fracture surface morphology of a composite comprising 20 wt.-% of Argeli fiber and 80 wt.-% biodegradable polymer blend (POLY)

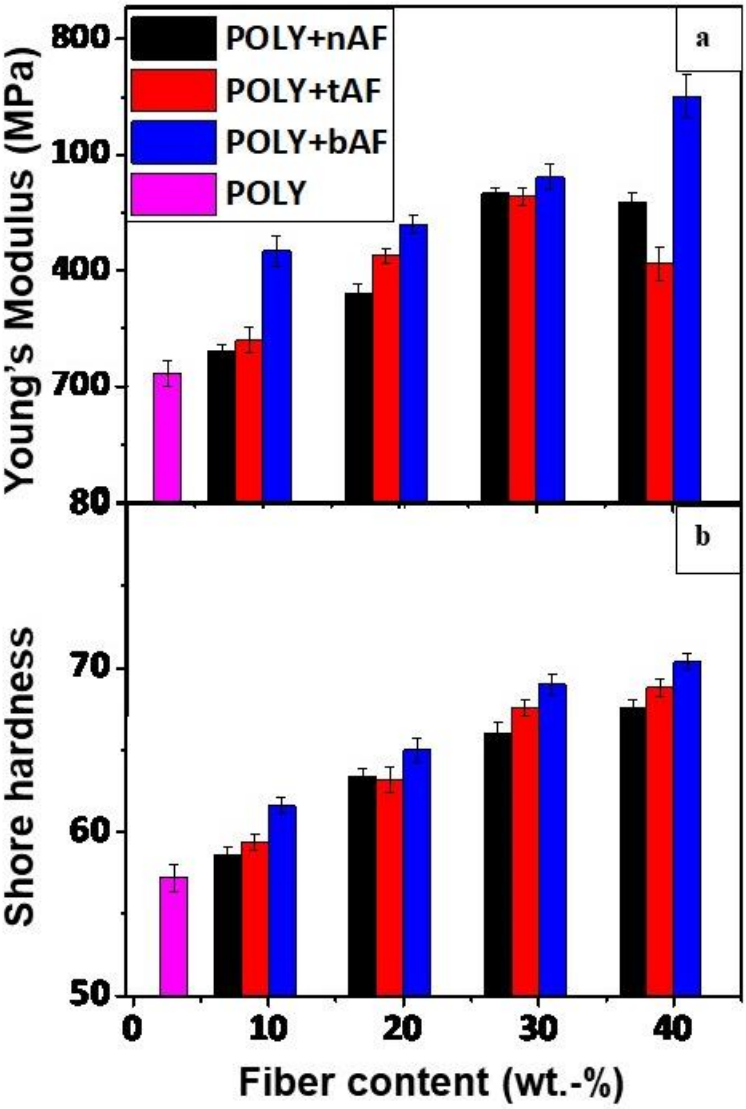

Figure 5 Variation of tensile Young's modulus (a) and Shore D hardness (b) of the polymer blend as a function of Argeli fiber content as filler; POLY: polymer blend matrix; nAF: neat fibers prepared by mechanical processing; tAF: alkali-treated fibers; and bAF: bleached fibers

The results obtained as tensile modulus, which signifies the stiffness of the composite material, and the Shore D hardness, signifying the resistance of the material against the deformation caused by penetration of an indenter (Correia & Valera, 2019), are presented in Fig. 5. The variation of Young's modulus with the content of fibers obtained by different treatments is shown in Fig. 5a.

The polymer blend (POLY) has the lowest modulus of 780 MPa, which increases with an increase in fiber content and reaches a value as high as 2450 MPa (more than 3 times the value of neat POLY) on the addition of 40 wt.-% of bAF, a huge improvement in the stiffness of the composites by the Argeli fibers.

A closer look at Fig. 5a reveals that with an increase in the fiber content, the value of Young's modulus increases almost linearly which attests to the reinforcing effect of the uniformly distributed fibers. Further, there is a trend in the increase in the reinforcing effect for the fibers in the following order: nAF<tAF<bAF. The highest value of the modulus for the composite consisting of the bAF can be attributed to the maximum amount of crystalline cellulose (Ku et al., 2011), which is the stiffest of all constituents of lignocellulosic fibers, present in the bAF.

The Shore D hardness measurement of the sample provides another testimony of the reinforcing effect of the natural fibers in the polymer blend. Fig. 5b shows that the value of hardness increases with an increase in fiber concentration. The hardness value of 55 MPa increases to 68 MPa for the composite comprising 40 wt.-% of bAF. Also, in this case, the effect of chemical modification on the modulus of elasticity can be observed.

Conclusions

The Argeli fibers in their pristine stage were found to consist of microfibrils, several microns in diameter, in the form of compact bundles glued together by amorphous components that dissolved upon chemical treatments. The microfibrils were found to exfoliate into constituent microfibers and distribute uniformly in the PLA/PBAT blend matrix upon melt processing. The addition of Argeli fiber into the PLA/PBAT mixture led to a reinforcement of the matrix with an increase in the tensile modulus as well as Shore D hardness, the properties being further augmented by chemically treated fibers. The observed enhancement in properties of the biocomposites was attributed to the formation of a highly crystalline and pure cellulosic framework due to the dissolution of the amorphous parts as well as other impurities from the neat fibers during their chemical treatment. Overall, the Argeli fibers exhibited a quality of highly potential reinforcing agent for the development of environment-friendly biodegradable polymer composite.

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Author Contributions: PG: Performing the experiments, analysis of data, editing the manuscript; LG: Laboratory arrangements, analysis of data, and supervision; OPB: Sample preparation, and data analysis; SP: Supervision of the work, manuscript editing; NLB: Supervision of the work, manuscript editing; MN: Funding acquisition, conceptualization, data analysis, and supervision; RA: Funding acquisition, conceptualization, supervision, manuscript drafting, and editing.

Conflicts of Interest: The author declares no conflicts of interest.

Data Availability Statement: The data that support the finding of this study are available from the corresponding author, upon reasonable request.

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