

Synthesis of Novel Hybrid Biocomposites and Experimental Investigation of Mechanical Properties

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Abstract

As industries increasingly adopt additive manufacturing, the demand for materials that offer better mechanical properties and functionality is on the rise. The purpose of present research work is to develop a novel hybrid biocomposites for 3D printing applications. The acacia bark, waste powder obtained from wet maharayan oil consisting of herbs and other bio filler was added in polylactic acid (PLA) for synthesis of hybrid biocomposites filament by twin screw extrusion-based process for FDM 3D printing. The tensile strength, impact strength, and hardness were investigated. The morphological study was conducted using SEM. The four different composition samples were obtained with varying range of PLA and other bio filler materials. The specimens for different tests were prepared as per ASTM D638 and ASTM D256 standard. The result indicates slight increase in tensile strength of one composition, decrease of impact strength and increase of hardness values for all compositions. The obtained results provide an understanding of preparation of a patented novel hybrid biocomposites with the aim to bring out the mechanical and morphological aspects of it.

Keywords: 3D printing; hybrid biocomposites; PLA; mechanical properties; morphology

INTRODUCTION

The additive manufacturing 3D printing sector has growing tremendously over last few years. The 3D printing technology is more competent to produce complex parts, customised parts, less printing time than conventional forming processes [1]. The 3D printing is best suitable for prototype as well commercial parts in the market. The maximum research has been done and still going on the process parameter optimization of different 3D printing technology but still there is need to focus on new 3D printing material inventions, material properties development research [2]. The commonly used 3D

printing materials are polymers, metal and alloys, composites, ceramics, concrete etc. used in aerospace, automotive, biomedical sectors [3]. The polymer has wide scope of applications in sports, medical, toy industries along with automotive sector [4]. The 3D printing polymers are thermoplastic type polymers like acrylonitrile butadiene styrene copolymers (ABS), polyimide (PA), polycarbonate (PC), Polylactic acid (PLA) and thermosetting types like polystyrene, polyamides, photopolymer resins [5]. The synthetic polymers are driven from petroleum products which are non-biodegradable, non-bio compactible nature [6]. According to global environmental aspects, there is demanded need for utilization of renewable sources, regulations and policies made to use sustainable material [7]. Focusing on this high impact requirement, researchers have been

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developing biodegradable, bio compactable materials [8,9]. The aim towards sustainable environment is easily available, low cost, bio compossible, biomass, bio waste and agricultural waste could be an alternate solution. Since last decade, biodegradable materials are going to use in automotive industry for various applications like sound efficiency, customized colour, recyclable and cost-effective parts and assemblies [10, 11].

For Fused deposition method (FDM) 3D printing, PLA, polyimide, and polyethylene are recyclable, biodegradable and bio compactible materials [12]. The PLA is extracted from renewable sources like starch, cane sugar etc. The biodegradability and bio compatibility of PLA with different types of biomasses or waste is higher. Hence PLA has been selected to form novel hybrid biocomposites [13]. The properties possessed by PLA biocomposites are most favourable, least cost for FDM 3D printing applications [14].

The number of researches has been carried out to develop PLA biocomposites by using bio filler materials like hemp, banana, sisal, acacia, neem powder, jute, kenaf, cotton, pineapple, bamboo, flax etc. The addition of this in PLA showed higher values of properties depends upon less bio filler weight % addition, fine particle size, homogenous formation of hybrid composite. Whereas the lower results occurred due to high weight % bio filler, untreated fillers, maximum moisture content filler etc [10,15]. The applications of bio composite are in wide range of fields like automotive, food packaging, mobile casing, window frames, railings, decorative parts, doors, electronics good packaging, agricultural etc [16]. Day by day the 3D printing product demand is increasing which leads to issues of non-degradability, recycling, environmental issues etc. The present research work tried to address the above problem by innovating novel hybrid bio composite material. Printable polymer composites indeed play a vital role in advancing the capabilities of 3D printing, addressing many of the limitations associated with traditional pure polymers. The incorporation of composites allows for the customization of geometries, leading to more design freedom, optimization for specific applications and minimizing material waste. [17-25].

To the best of authors knowledge, no title found for this hybrid biocomposites or combination of materials selected here, hence got the idea about this research work with use of asparagus racemosus powder, industry herbal waste powder along with acacia bark powder to form PLA biocomposites. In this paper an endeavour was made to focus this research area, to work over it and investigate novel hybrid biocomposites PLA and their mechanical characteristics. The current research study gives synthesis and characterization of novel patent filed and published on Hybrid biocomposites 3D printing material and process. [Patent no.202421036685]

METHODOLOGY

Materials and Methods of Preparation

The PLA 3D850 grade granules were provided by Ingeo NaturTec India Ltd, (Chennai, India) used as a base material for hybrid biocomposites preparation. The acacia bark, asparagus racemosus powder 80 mesh size each was provided by SG Phyto Pharma Pvt Ltd. (Kolhapur, India). The non-hazardous solid waste obtained by processing maharayan oil (herbal product) which was used as a bio filler material. Waste powder was obtained by processing the waste as shown in Figure 1 a, b, c & d respectively.

Sample Preparation

The virgin PLA granules and bio filler materials acacia powder, asparagus racemosus powder and prepared herbal dry waste powder from industry was mixed with certain ratio to prepare four samples. These samples were prepared for 70:15:15 and 80:10:10 ratios on trial-and-error basic which had given less formability and higher brittleness. Hence, the suitable ratio was selected and formed as shown in Table no.1.



Figure 1. (a) Industry waste; (b) process of crushing; (c) & (d) powder.

Table 1. Compositions of samples.

	P1	P2	P3	P4
PLA	96%	96%	96%	98%
Asparagus racemosus	3.0 %	1.0 %	0.50%	--
Acacia bark powder	1.0 %	3.0 %	0.50%	0.50%
Waste powder by processing waste of Mahanarayan oil	--	--	3.0%	1.50%

Sample Pallet Preparation

Each sample was transformed through batch mixing, melt blending, and extrusion in pallet formation process. The blending was done using twin screw extruder machine as shown in Figure 2 [15]. The eight heating zones with set temperature for each sample is mentioned in Table 2 and table 3. The pallets were prepared from this extrude wires for each sample.

Fabrication of Hybrid Composite Filament

The prepared pallets were transferred to single screw filament extruder machine. Preheating at 200°C for 2 hours and then melting at 280°C was carried out. This melt mixture was extruded through die to give continuous filament of 1.75 mm diameter. The process was repeated for remaining samples. The samples were fabricated by FDM 3D printer Creality Ender 3. (Orient 3D printing, Kolhapur, India). First models of testing sample as per ASTM were prepared using Autodesk Fusion software. The optimal printing parameter conditions were set at 0.2 mm layer height, 0.8 mm nozzle, 1.75 mm filament

diameter, 0.8 mm line width, temperature 65°C, nozzle temperature 210°C, speed 50 mm/s and 100 % infill density. It is exported as G code file used in 3D printer. The material is prepared as per standard procedures and received national biodiversity authority certification.

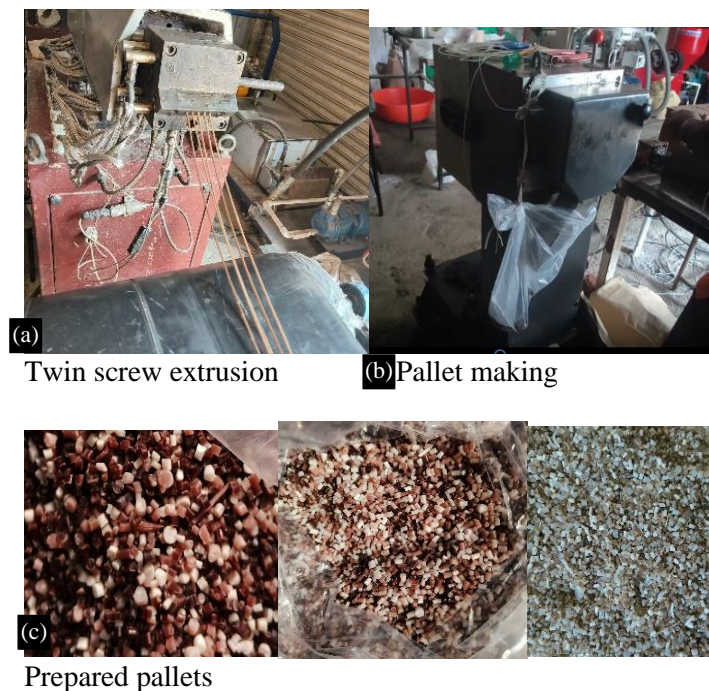


Figure 2. a) Twin screw extrusion, b) Pallet making and c) Prepared pallets for P1,P2,P3 and P4 samples.

Table 2. Heating zone temperatures for samples.

	Barrel 1 temperature (°C)	Barrel 2 temperature (°C)	Barrel3 temperature (°C)	Barrel4 temperature (°C)	Barrel5 temperature (°C)	Barrel6 temperature (°C)	Barrel7 temperature (°C)	Barrel8 temperature (°C)
Sample 1	61	80	100	130	150	160	150	149
Sample 2	63	80	99	130	152	160	152	155
Sample 3	64	80	100	130	156	160	151	152
Sample 4	54	80	100	131	152	160	152	153

Table 3. Extruder control parameters for samples.

	Melt temp(°C)	Melt pressure	Volumetric torque	Main motor torque
Sample1	153	18	87	17
Sample 2	185	5	87	9
Sample 3	187	5	87	17
Sample 4	160	4	87	9

Characterization

The dog bone shape ASTM D638 [26] specimens (1, 2, 3, and 4) as shown in Figure 3. a and b respectively. These were 3D printed for each respective sample composition in flat to bed and on edge orientation as shown in Figure 3. c and d respectively. The tensile test on each specimen sample was conducted by using Universal Testing Machine (UTM). [Source: ELCA laboratory, Pune].

The notched impact testing specimens were prepared as per ASTM D256 [27]. The test was performed on Impact testing machine with pendulum capacity load applied was 21.68 Joules. The five specimens were prepared, tested and the average results were calculated for specimens 1,2,3 and 4. The hardness tests were conducted for each specimen by using Durometer -Shore D. The five different readings on each specimen were taken and average was calculated to find the hardness value for each specimen. Virgin PLA and all prepared specimens were scanned under SEM to study the morphology using SEM-EDX at an acceleration voltage 15.0 KV at different magnifications.



Figure 3. Tensile test specimens as per ASTM D638 a) and b) Tensile specimens as per ASTM D638; c) flat to bed and d) on edge orientation.

RESULTS AND DISCUSSION

Tensile Strength

For determining the tensile strength and percentage of elongation, the force was gradually applied until the breakdown [28]. The results of the flat to bed and on edge orientations are presented in Table No.4. It shows the average values of the five specimens that were taken for the tensile test for each sample and orientation. This is as the printing orientation greatly influences the specimen tensile load, on edge offers more layers than flat to bed and they are parallel to tensile force. The results obtained confirms that orientation of 3D printing has significant impact on tensile test properties [10,18]. The stress vs strain graphs of specimens are represented in fig.no. 4 for both orientations. The specimen 1,2 shows higher results than virgin PLA for both the orientations as asparagus racemosus has fibrous structure. While specimen 3,4 shows lower than virgin PLA except percentage elongation at on edge.

The tensile strength of all bio composites with on edge orientation showed higher results than flat to bed orientation with 1%, 12.05%, 29.72% and 18.70% respectively. Also, on-edge orientation showed higher results than flat to bed orientation in percentage elongation. The Figure no.4 represents comparative between P1, P2, P3 and P4 with Tensile Strength, % Elongation and Yield Strength.

Table 4. Tensile test results.

	Tensile Strength (N/mm ²)		% Elongation		Yield strength(N/mm ²)	
	Flat to bed	On edge	Flat to bed	On edge	Flat to bed	On edge
PLA	40.93	50.70	4.72	2.0	40.93	47.09
P1	49.75	50.29	5.59	5.61	45.40	45.80
P2	44.42	50.12	2.12	2.44	39.50	45.21
P3	33.43	45.18	1.56	2.7	30.25	37.47
P4	37.51	45.25	2.6	2.16	37.32	40.99

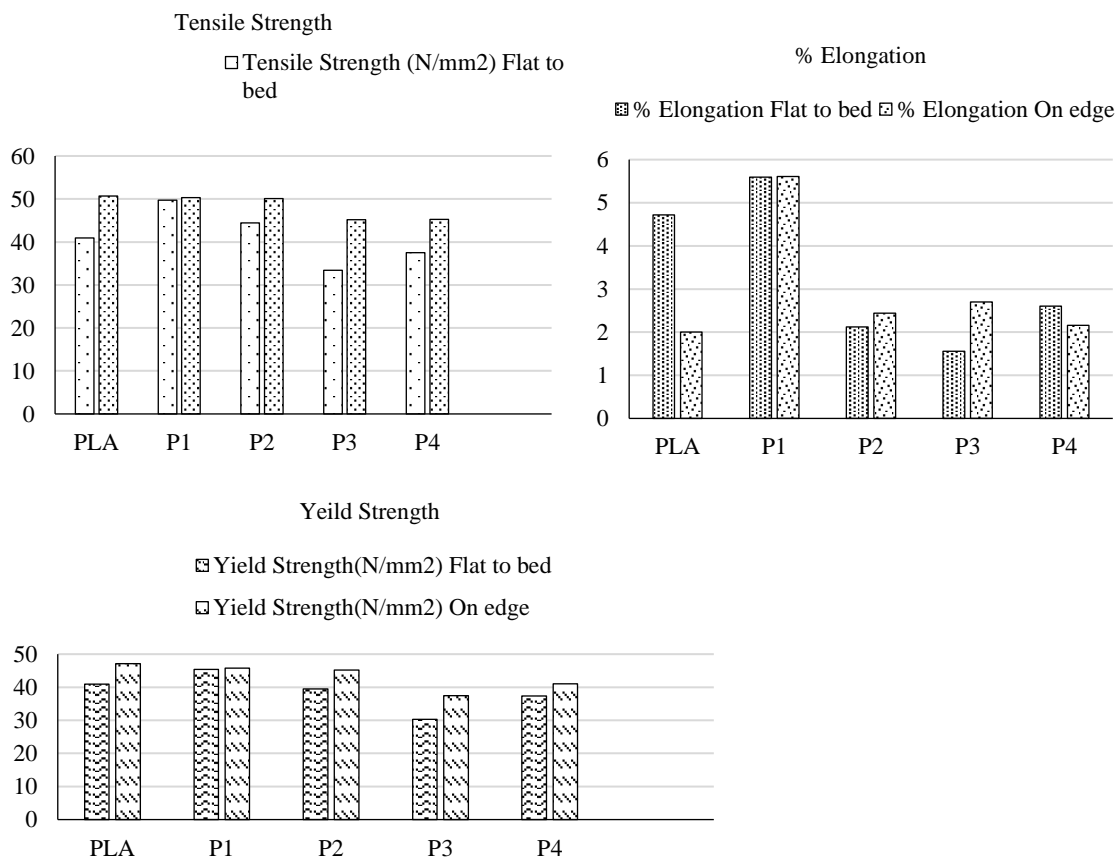


Figure 4. Comparison between P1, P2, P3 and P4 a) Tensile Strength b) % Elongation c) yield strength.

The 'Flat to Bed' orientation showed lower tensile strength due to weaker interlayer bonding caused by the layer-by-layer deposition method in 3D printing. Whereas the 'On Edge' orientation the tensile force is applied parallel to the layers which leading to higher tensile strength. For the requirement of high tensile strength, the On-Edge orientation is more suitable [30]. The virgin PLA having higher tensile strength "On Edge" than "Flat to Bed," reflecting inherent anisotropic behaviour in additively manufactured materials [31]. Composition P1 and P2 demonstrate more isotropic behaviour, likely due to better adhesion between layers or uniform filler dispersion. Composition P3 and P4 show higher anisotropy, which could result from poor interlayer adhesion or uneven distribution of reinforcements [32]. The graph compares the percentage elongation (a measure of how much a material stretches before breaking) for samples printed in the Flat to Bed and On Edge orientations. This could be due to specific material or structural properties of the sample, where the interlayer bonding may still influence elongation. The graph emphasizes the importance of orientation in mechanical testing for applications requiring specific load-bearing characteristics. The differences in yield strength between orientations explores the influence of material anisotropy, microstructure, or filler alignment within the matrix.

Due to have better filler dispersion or more uniform microstructure, gives better yield strength. The graph focuses the importance of orientation in mechanical testing for applications requiring specific load-bearing characteristics [33].

Impact Test

The impact strength of the biocomposites for various orientations is displayed in Table no. 5. This strength was found to be much higher specimen 2 and slightly lower for specimen 1,3,4 than that of pure PLA [18]. From an orientation perspective, the results of the Izod impact test seems to be consistent with tensile test. This is also because of additional layers, which run parallel to the impact force and creates increased resistance to impact load [29].

Due to brittle nature of PLA, gives lower impact strength than prepared bio composite [33,34]. The impact strength of composition P2 showed higher results (peaking at approximately 54 J/m,) than P1, P3 and P4. Composition P2 better interlayer adhesion, or optimized layer orientation to resist impact forces. The addition of fibres like acacia helps to increase the toughness of material [35]. The SEM shows fibers embedded in the matrix with minimal gaps, corresponding to higher impact values. The study shows that, PLA and flax fiber gives more impact strength than prepared biocomposite due to a strong interfacial bond between the flax fibers and PLA matrix whereas PLA and hemp fibers, wood fibers gives lower impact strength. The hemp fibers has rigid and brittle causes less energy absorption [33,35].

Hardness Test

The hardness test was conducted for each specimen by using Durometer-Shore D. The five different readings on each specimen were taken and average was calculated to find the hardness value for each specimen mentioned in Table 6. It displays that the average hardness value of all composite specimens was greater than virgin PLA. The hardness value of composition specimens 2, 3 showed greater value than composition specimens 1 and 4 as shown in Figure 5. The most likely explanation of this result is that the addition of 3% acacia bark powder which is harder than other bio filler materials showed increase in hardness by 7.8% than virgin PLA and 5.2% than other compositions. The composition specimen 3 consisting equal amount of acacia bark powder, asparagus racemosus powder along with industrial waste obtained the greater hardness value.

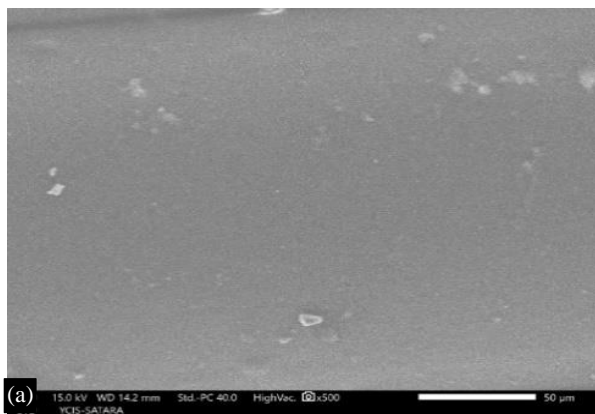
Due to uniform dispersion of fibers, no voids present and uniform fiber distribution gives higher hardness values. PLA has low resistance to surface deformation contributes to the lower Shore D value [36]. The Flax or stiff fibers like hemp and wood improve hardness. Which showed similar results with addition of acacia bark powder and waste powder [32].

Table 5. Impact test results.

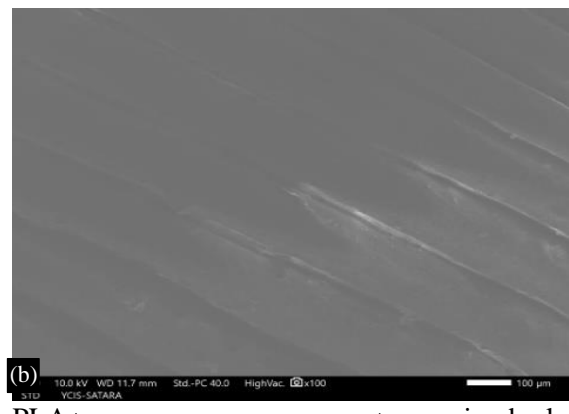
	PLA	P1	P2	P3	P4
Izod Impact strength (J/m)	36.26	38.13	54.00	45.60	43.25

Table 6. Hardness test readings.

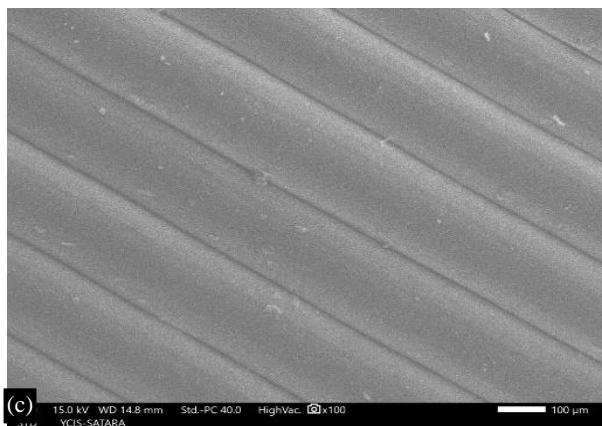
	PLA	P1	P2	P3	P4
Hardness (Shore D)	74	77	79	79	78
	73	78	81	80	78
	73	77	80	81	79
	76	79	82	79	77
	74	79	78	81	78
Average (Shore D)	74	78	80	80	78



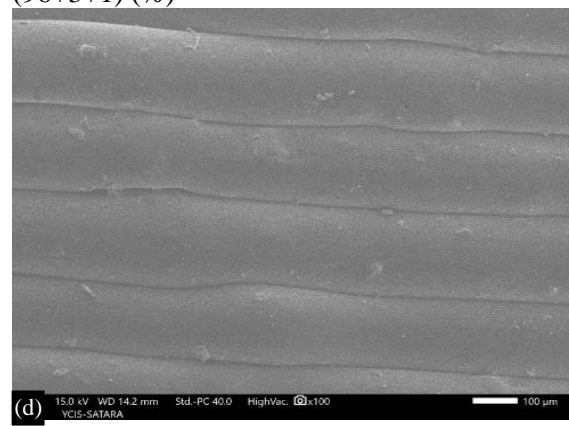
Pure PLA



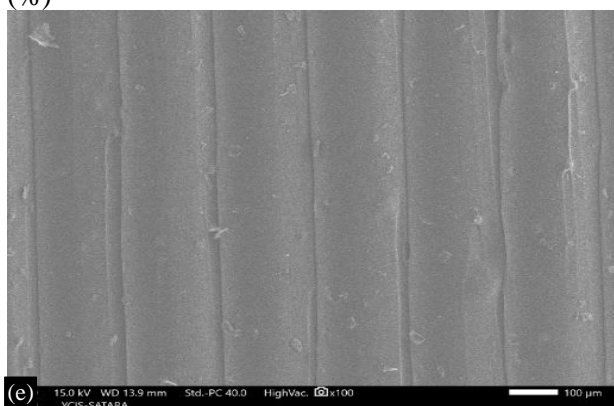
PLA+ asparagus racemosus+ acacia bark (96+3+1) (%)



PLA+ asparagus racemosus+ acacia bark (96+1+3) (%)



PLA+ asparagus racemosus +acacia bark+waste (96+0.5+0.5+3) (%)



PLA+ acacia bark+ waste (98+0+0.5+0.5) (%)

Figure 6. SEM micrographs for specimen samples.

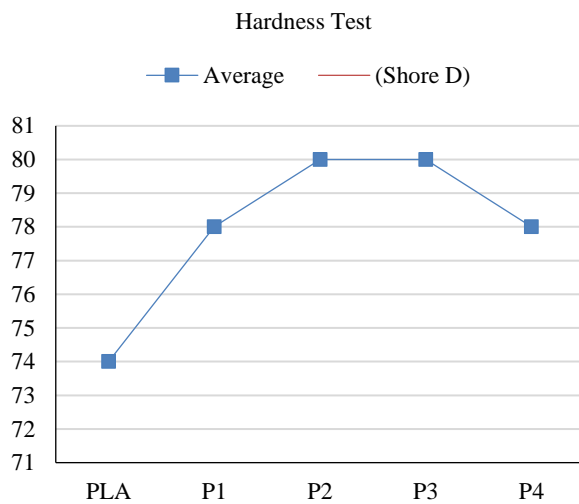


Figure 5. Hardness for specimen samples.

SEM Results

Figure 6 shows SEM micrographs of virgin PLA and specimens. Figure 6. a represents SEM image of virgin PLA which has a smooth surface and does not show any agglomerations indicating homogeneous morphology. As can be seen from Figure 6. b and c addition of bio fillers asparagus racemosus and acacia shows fibrous structure. Also, it does not show any agglomerations showing filler materials were dispersed homogeneously. The surface texture on fiber can be clearly visible [34]. Thus, the results at this stage approves that these bio fillers compositions are compatible with PLA. Figure 6. d and Figure 6. e corresponds to the micrographs of bio fillers composition indicates fibrous structure with some agglomerations due to hydrophilic nature of PLA [36].

CONCLUSIONS AND FUTURE SCOPE

The novel hybrid biocomposites lead to a variety of challenges in the field of 3D printing engineering. The addition of these materials to virgin 3D printing material may ensure overcoming the future environmental, economic and performance issues and provide possible market for an industrial application. In this research work tensile, impact and hardness properties of hybrid biocomposites for all four specimens were investigated. The on-edge orientation consistently outperformed the flat-to-bed orientation in tensile strength and elongation, emphasizing the significance of layer alignment in load-bearing applications. Among the compositions, Sample 2 exhibited the highest impact strength, likely due to improved resistance to crack formation. Hardness improvements were observed across all samples compared to virgin PLA, with Samples 2 and 3 showing the most significant increases, highlighting the role of uniform filler dispersion and optimized material design in enhancing surface resistance. These findings underline the potential of bio-composite materials for applications requiring tailored mechanical performance. The SEM studies showed fibrous structure with smooth and homogeneous surface and some agglomerations for different specimens suggesting the concentrations of bio fillers with PLA are compatible. The new insights of research contribute to novel biocomposites material formed with natural wood colours which reduces the costing of recolouring process and time to virgin PLA. Hence this would be benefited to consumers, filament manufactures as well the waste producing companies. However, the limitations of the present study offer some future guidelines. First, studies can be performed with different bio filler materials with different weight percentage, second is surface roughness properties of 3D printed specimens need to be addressed. The comparative study of prepared biocomposite gives satisfactory results. These are suitable for automotive parts, consumer electronics, construction materials etc. The future of hybrid biocomposites is promising leading to the development of processing techniques by examination of various 3D printing methods for thermal considerations and post-processing treatments which can enhance mechanical properties.

List of Abbreviations:

Abbreviations	Term
PLA	Poly Lactic Acid
FDM	Fused Deposition Method
UTM	Universal Testing Machine
3D	Three Dimensional
SEM	Scanning Electron Microscope
SEM-EDX	Scanning Electron Microscopy with Energy Dispersive X-ray

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