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Analysis and optimization of low-cost compression molding process for manufacture of particulate bone powder PLA composites

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Abstract

Poly Lactic Acid (PLA) is a bio-based polymer gaining attracted for the fabrication of composite materials. This research examines the mechanical characteristics of biodegradable PLA composites reinforced with animal bone particles through the compression molding technique. The reinforcement of animal bone powder was varied from 5% to 20% wt, and the variables of molding parameters of dwell time, die temperature, and pressure. The experiments are carried out using Design of Experiments (DOE). The hardness, tensile, and flexural properties have been evaluated. The finding reported that the incorporation of animal bone powder leads to an enhancement in mechanical properties. The tensile strength of PLA composite was improved by 19.26% to neat PLA samples under similar process parameters. The Hardness of the composite was improved by 24.39% when compared to neat PLA samples. The flexural strength of composites was improved by 15% to the neat PLA samples. Composites containing higher weight percentages of fillers demonstrated increased stiffness and strength, albeit with a reduction in ductility. The process parameters of compression molding were optimized using Taguchi analysis for higher tensile strength. The findings suggest that the inclusion of reinforcement plays a role in enhancing the mechanical performance of composites. In addition, SEM analysis confirmed the particle was well-dispersed, contributing properties enhancement.

Keywords PLA, DOE, Bone powder, Sugarcane bagasse powder, Sustainable materials

1 Introduction

Composite materials consist of two or more distinct materials combined to enhance properties beyond those of the individual components. The matrix can consist of either metal or polymer. The composite material produced demonstrates enhanced mechanical, thermal, and electrical characteristics, rendering it appropriate for a variety of applications, including aerospace, automotive, construction, and electronics. Composite materials possess a lower weight, durability, resistance to corrosion, and good strength, and are used in various applications [1–3]. Furthermore, polymer composite materials



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offer a high degree of customization, allowing their properties to be adjusted to meet particular application requirements. Knowledge of the composite process has heightened the worldwide demand for natural resources. Wide use of oil-derived products, along with greenhouse gas emissions, has led to a scarcity of recyclable and biodegradable materials.

PLA is a biodegradable polymer sourced from natural materials like corn starch, sugarcane, and potato starch, and is produced through the activity of microorganisms [4, 5]. In recent years, due to its low toxicity and biodegradability, PLA has grown in popularity as an eco-friendly substitute for traditional plastics. Packaging, textile fibres, and biological applications are among its uses [6, 7]. However, its properties, such as toughness, are relatively inadequate, which may hinder its commercialization. Recently, researchers studied hydroxyapatite and tried to incorporate it with PLA to enhance the strength of the composite. Nano-hydroxyapatite filler improves tensile strength, elastic modulus, and biodegradation rate. Furthermore, better compatibility between the matrix and the filler can lead to further enhancements in mechanical properties, which can be achieved through the surface modification of nano-hydroxyapatite [8–10]. There has also been significant interest among researchers in low-cost, biodegradable fillers such as bone powder and starch powder [11–14]. Additionally, natural fiber reinforced PLA composites are widely utilized, and techniques like fiber treatment can be employed to improve their properties [15–17].

The compression molding process can yield denser and stronger composites based on PLA [18, 19]. A variety of process parameters, including pressure, temperature, and dwell time, significantly affect the strength of the composite, necessitating stringent control over these factors. In this process, granules are heated within a mold with the required pressure to liquefy the material, allowing it to flow at elevated temperatures. Once the material cools to room temperature, it solidifies and takes the shape of the mold. The performance of this method was regulated by modifying the pressure, duration, and temperature. Additionally, compression molding incurs lower costs for mold fabrication compared to alternative ways, such as injection and extrusion molding [20–23].

Rubio-López et al. [24] conducted a study on how fabrication parameters affect the strength of bio-composites, utilizing film stacking and compression molding techniques with reinforcement fibers sourced from jute, flax, and cotton. The findings showed that preheating the material for two to three minutes produced the highest tensile strength of 100 MPa. Additionally, Roberto et al. [25] noted that the leaves of *Posidonia oceanica*, a prevalent seagrass in PLA-based bio-composites, result in enhanced flexural modulus and tensile strength. Numerous researchers have explored composites of fiber-reinforced polymer, resulting in enhanced mechanical properties; however, a significant limitation is the inherent hydrophilic nature of the fibers, which has undeniable effects. The lifespan is reduced due to their high moisture absorption capabilities.

Mohan et al. [26] investigated the performance of bamboo-based polymer composites and found tensile strength enhanced by 32% by adding sisal fiber with a maximum flexural strength of 46.81 MPa. Chatuvedi and Rakesh [27] developed hybrid PLA reinforced powders using walnut shell and pine needle ash powders, varying the composition from 2% to 8% by weight. A degradability test conducted in water over eight weeks revealed the formation of patches and noticeable material removal from the top surface. Nano

fillers have the potential to harm the environment. Li et al. [28] used eggshell powder-based composite. Their findings indicate that the composite films demonstrate strong interaction and adhesion, achieving a maximum compressive strength of 30%. Additionally, it was observed that these films decompose within one month when placed in soil.

Cow bone fragments were used as reinforcement in a polyester matrix material by Oladele et al. [29]. They looked at 75, 106, and 300 μm particle sizes at weight proportions of 2, 4, 6, and 8% and discovered that these additions improved the polyester matrix's mechanical qualities. In particular, toughness was enhanced by coarser particles, while strength was raised by smaller particles. In another study by Nagarjun et al. [30] utilized micro fillers derived from tamarind and date seeds. The tensile strength of the PLA matrix showed substantial improvement, and flexural strength improved nearly twofold for reinforcements from both palm and tamarind.

Animal bones, sourced from livestock waste, contribute to reducing environmental pollution. In India, affluent individuals often prefer goat and sheep meat as part of their non-vegetarian diet, while the bones are frequently regarded as waste. Recently, animal bone powder has gained traction for its potential application in the production of PLA composites. These waste bones are not only cost-effective but also biodegradable. However, there has been limited emphasis on bone particulates. Consequently, research has concentrated on the mechanical performance of PLA composites reinforced with animal bone particles, which were fabricated using the compression molding technique. A microstructural analysis was conducted to assess the impact of the filler on tensile strength, elongation percentage, and flexural strength. Additionally, the compression molding parameters were optimized through the Taguchi method to enhance strength and hardness. Using a hand layup process, Molla and Belay [31] created hybrid epoxy composites reinforced with bamboo and ox bone powder. These composites included a 40% weight fraction of 75-micron reinforcement and various orientations of bamboo. Bamboo fibre and powdered animal bones were used as reinforcements in the study. The findings indicated improvements in impact, flexural, and compressive strength.

A study on the applications of bone and bamboo fibre using epoxy as a binder to create a novel composite was carried out by Ikpoza et al. [32]. They evaluated elasticity, modulus of rupture, and tensile stress testing and reported excellent results.

According to the literature, no research has been done using bone particles as reinforcement in PLA composites. Animal bone particles made by ball milling and creating PLA composites using a compression moulding machine and ideal process conditions are thus the main focus of the study.

2 Materials and methods

Animal bone powder, derived from goat bones, is used. This study does not involve a clinical trial and, therefore, was not registered in a clinical trial registry. Bones serve as a significant source of calcium, phosphorus, and magnesium, utilized as polymer composites. The fine particle size of the bone powder (50–70 microns) enhances its surface area, facilitating improved adhesion with the polymer matrix. The bones undergo a cleaning process with water to remove any residual flesh, followed by a sun-drying period of 5–6 h every day for one month. Subsequently, the bones are crushed into smaller fragments and milled using a standard mill to create powder. The resulting powder is then processed in a ball milling machine for 36 h to achieve a particle size reduction to 20

microns. Figure 1 illustrates the macro view of PLA granules as obtained from suppliers. Figure 2 illustrates the macro view of the bone particulate powder utilized in the experiments, with Fig. 2 (a) depicting the bones as received and Fig. 2 (b) showing the dried fragments, and Fig. 2 (c) presenting the macro view of the micro powder post-ball milling.

The process of compression molding has been employed to manufacture PLA composites. This molding technique entails the introduction of a specified amount of materials into a preheated die, determined by the weight ratio of PLA to animal bone powder. The die is specifically engineered to create composite plates. Initially, the PLA and bone powder mixture is blended in a high-speed mixer to achieve an even distribution of the bone powder. The quality of the PLA composite depends on the duration of mixing and the temperature applied. Subsequently, the estimated volume of the semi-solid mixture is placed into the mold, where pressure is exerted using a hydraulic press[33]. The composites are produced according to the design specifications. The plates are extracted promptly after the melt solidifies within the mold and cools to ambient temperature.

The samples were sectioned for the assessment of mechanical properties. Specimens were prepared as per ASTM D638 standards, and tests were performed at a crosshead speed of 5 mm/min by a Universal Testing Machine (UTM), shown in Fig. 3. Flexural tests were carried out as per ASTM D790 standards with a UTM. For the Izod impact tests, specimens were created according to ASTM D256 standards and tested on an impact testing machine.

The microstructure of composite samples of both pure PLA and PLA composites was captured at various locations on the samples using optical microscopy with a magnification of 200X. The samples were coated with gold via sputter coating to avoid charging. SEM analysis was performed using an Oxford INCA 400 model at an acceleration voltage of 10 kV. The process parameters were determined through a review of existing literature and preliminary experimental trials. The critical variables include compression pressure, temperature, and holding time.

Table 1 presents levels of process parameters, which were determined through experimental trials and existing literature [34]. The impact of specific factors such as die temperature, pressure, dwell time, and the percentage of powder reinforcement was assessed using the Taguchi S/N ratio. The process that had the greatest effect on achieving



Fig. 1 Granules of PLA



(a) Bones



(b) Dried fragments



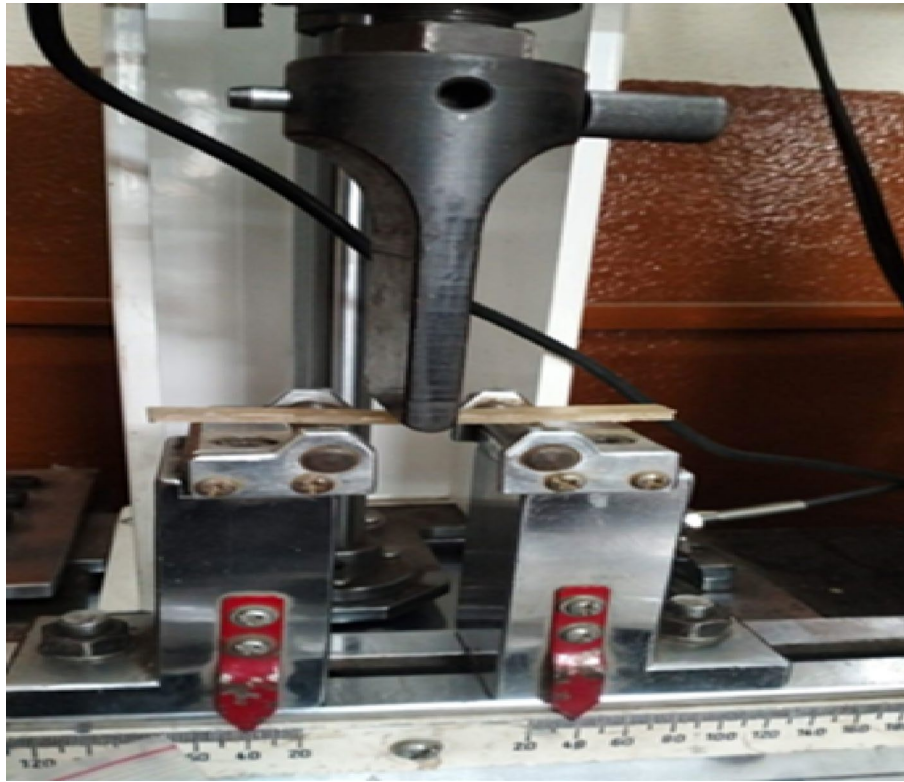
(c) Micro powder

Fig. 2 Steps in the preparation of bone powder

maximum tensile strength and hardness was identified through the signal-to-noise ratio, while the contribution percentage of each process parameter was analyzed using Analysis of Variance. The optimal parameters for the process were estimated and validated through final experiments. The low-cost experimental setup was developed in conjunction with a hydraulic press utilized as a Compression Molding set-up die, as illustrated in Fig. 4, and Fig. 5 is a close view of the compression molding die.

All samples were found macroscopically good and free from voids. Figure 6 shows the fabricated PLA composite sample 8.

In this study, the L16 orthogonal array designed using the Taguchi method was employed, focusing on four process parameters: compression pressure, temperature, holding time, and percentage reinforcement. The Taguchi method was selected due to its capability for optimization with a limited number of experiments. The results pertain to the composites produced through compression molding with various variables. The

**Fig. 3** Universal Testing Machine**Table 1** Process parameters and levels

Process parameters and level	Level 1	Level 2	Level 3	Level 4
Die Temperature (°C)	160	170	180	190
Pressure (MPa)	1	2	3	4
Dwell time (min)	20	25	30	35
Reinforcement (%)	5	10	15	20

Taguchi approach provides a strong experimental design that requires fewer trials, making it particularly suitable for this analysis.

The Taguchi method employs the Signal-to-Noise (S/N) ratio as the objective function for optimizing process parameters. It categorizes the computation of the S/N ratio into three types: Nominal, Larger, and Smaller. This study utilizes the larger-the-better criterion to determine the S/N ratio, which emphasizes maximizing the desired output to enhance process performance. S/N ratio formula for calculating Eq. (1).

$$\frac{S}{N} \text{ Ratio} = -10 \log \left(\frac{1}{n \sum \frac{1}{y^2}} \right) \quad (1)$$

Where y is the observed output response and n represents the number of observations.

3 Results and discussions

3.1 S/N ratio evaluation

The average ultimate tensile strength of the PLA composite is considered the output characteristic. The signal-to-noise (S/N) ratio is utilized to assess the process's



Fig. 4 Compression molding set up with a hydraulic press

robustness, with higher values signifying improved performance in relation to noise levels. The S/N ratio was derived from the ultimate tensile strengths of the composites, and calculations were performed using MINITAB software. Table 2 presents the S/N ratios for ultimate tensile strength, hardness, and flexural strength of the composites, while Table 3 details the response means and ratios for tensile strength. Figures 7 and 8 show the Response plot comparison of S/N ratio and mean for tensile strength, respectively. Table 3 shows the response means and S/N ratios for flexural strength. Larger S/N ratio values suggest that the process parameters are closely aligned with the output response, thereby enhancing the quality of the composites. The significance of each of the process parameters was determined based on delta statistics. Larger delta values indicate a greater influence on the output response. The rank of the process parameters in descending order, highlighting the critical role of reinforcement particles as the most influential parameter. Table 3 shows the mean response table of the S/N ratio for tensile strength, which indicates that reinforcement particles are the most influential parameter. The optimal values of the process are at 15% reinforcement, die temperature of 190 °C, Compression pressure of 3 MPa, and dwell time of 35 min.



Fig. 5 Compression molding die

3.2 Analysis of variance (ANOVA)

ANOVA is a statistical method employed to assess the influence of input variables and their interactions with output features, serving as an essential tool for optimizing parameters. Additionally, ANOVA quantifies the percentage contribution of each process parameter, aiding in the identification of the most significant parameter for attaining optimal values. Additionally, the Fisher F-test serves as a tool to assess which factors significantly influence R%. A high F value typically indicates that alterations in the process parameters have a substantial effect on the quality characteristics. Table 4 presents the ANOVA results regarding the mean tensile strength. It is found that the reinforcement has a 97.64% contribution, which is the highest value from the analysis in achieving the strength of composites. The higher the F Value of 137.52, and the lower the P value of 0.001 was obtained for reinforcement and can be considered as an influential parameter. The confirmation test is performed with optimal process parameters, and the experimental values are within the 5% variations, and found that the model is validated within the levels of process parameters.

**Fig. 6** PLA Composite sample 8**Table 2** S/N ratio of ultimate tensile, flexural strength, and hardness of composites

Run	Reinforce- ment (% wt)	Die tem- perature (°C)	Pres- sure (MPa)	Dwell time (min)	UTS (MPa)	Elon- gation (%)	Shore Hardness	Flexural Strength (MPa)	S/N ratio for tensile strength
1	5	160	1	20	43.17	2.51	91.28	90.46	32.7036
2	5	170	2	25	42.28	2.43	91.25	98.17	32.5227
3	5	180	3	30	44.36	2.32	93.74	108.14	32.9398
4	5	190	4	35	44.96	2.30	92.50	104.80	33.0565
5	10	160	2	30	48.15	2.67	94.47	113.29	33.6519
6	10	170	1	35	49.73	1.99	95.94	112.41	33.932
7	10	180	4	20	50.26	2.81	92.80	109.17	34.0244
8	10	190	3	25	54.82	2.94	93.39	113.28	34.7788
9	15	160	3	35	67.42	3.19	97.43	119.23	36.575
10	15	170	4	30	68.16	3.17	98.12	120.16	36.6706
11	15	180	1	25	69.25	3.22	93.49	114.87	36.8084
12	15	190	2	20	70.12	3.56	93	108.71	36.9168
13	20	160	4	25	64.25	3.41	94.15	110.71	36.1575
14	20	170	3	20	65.15	3.25	91.97	108.71	36.2783
15	20	180	2	35	66.70	3.40	94.93	110.80	36.4825
16	20	190	1	30	63.11	3.06	96.68	113.78	36.0020

3.3 Tensile strength of composites

Figure 9 illustrates the tensile properties of pure PLA and PLA composites, and found that PLA composites enhance the tensile strength of the composites. The highest tensile strength is observed for the PLA composite with 15%, while the lowest is found for the PLA composite with 5% composite. Additionally, the compression pressure significantly contributes to the improved strength of the composites. This phenomenon recognized the effective scattering of particles within PLA and the robust interfacial interactions

Table 3 Mean response table for signal to noise ratio and experimental data for means

Level	S/N ratio				Experimental data for the mean			
	R (%)	DT(0 C)	P(MPa)	Dwell time(S)	R (%)	DT(0 C)	P(MPa)	Dwell time(S)
1	43.69	55.75	56.31	57.18	32.81	34.77	34.86	34.98
2	50.74	56.33	56.81	57.65	34.10	34.85	34.89	35.07
3	68.74	57.64	57.94	55.94	36.74	35.06	35.14	34.82
4	64.80	58.25	56.91	57.20	36.23	35.19	34.98	35.01
Max- Min(Delta)	25.04	2.50	1.62	1.71	3.94	0.42	0.28	0.25
Rank	1	2	4	3	1	2	3	4

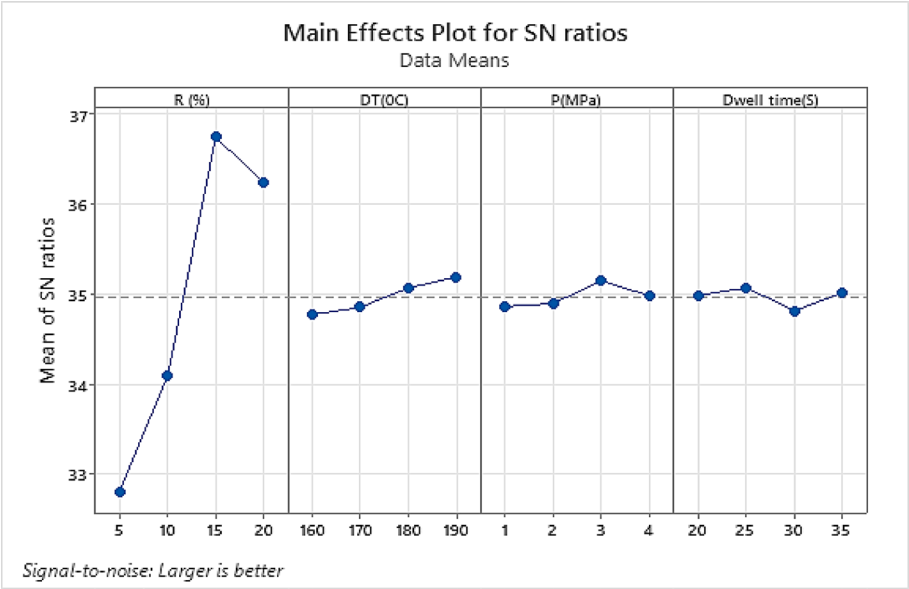


Fig. 7 Response plot comparison of S/N ratio

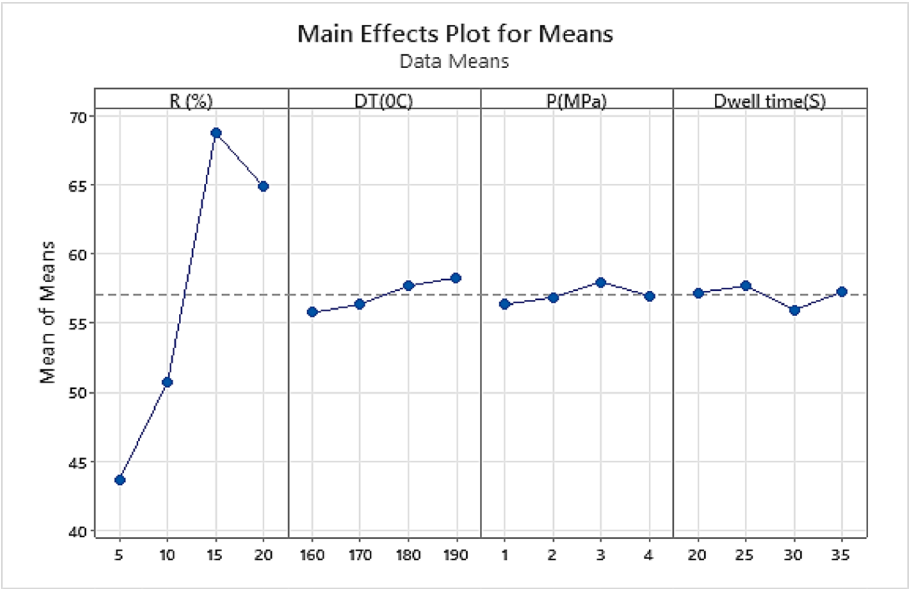
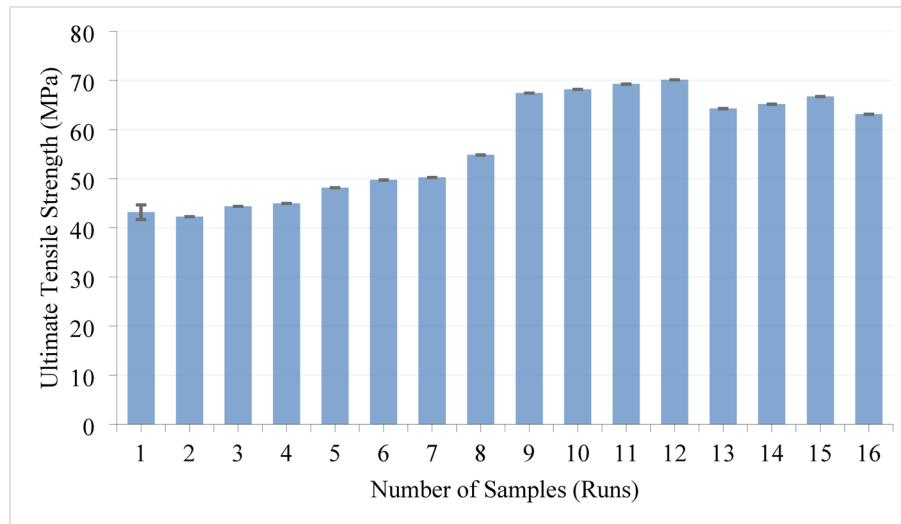


Fig. 8 Response plot for mean tensile strength

Table 4 ANOVA for mean tensile strength

		. Mean				
Source	DF	Adj SS	Adj MS	F-Value	P-Value	% age of contribution
R (%)	3	1659.70	553.233	137.52	0.001	97.64
DT(0 C)	3	16.00	5.332	1.33	0.411	0.94
P(MPa)	3	5.57	1.856	0.46	0.729	0.33
Dwell time(S)	3	6.43	2.143	0.53	0.691	0.38
Residual Error	3	12.07	4.023			0.71
Total	15	1699.76				100.00

**Fig. 9** Tensile strength of composites

between bone particles and PLA. The overall performance of the composites is influenced by the adhesion and compatibility between the polymer and the particles, as well as the crystalline nature of both PLA and bone powder.

However, an increase in the amount of bone powder in PLA results in a reduction of tensile strength due to its brittle characteristics. The agglomeration of CS particles at low TS leads to stress concentration within the matrix, resulting in a reduction of tensile strength. This assertion is corroborated by SEM and optical microstructural analyses, which reveal congestion and agglomeration of CS particles within the polymer matrix. Additionally, a weak interface around the aggregated CS particles may have contributed to the decline in tensile strength. Moreover, it was observed that the percentage elongation of the composites decreases as the reinforcement increases. The incorporation of fillers in PLA composites was observed to enhance the elongation at break. Likewise, the inclusion of bone powder microfillers in PLA composites also contributed to the improvement of mechanical properties, particularly elongation at break, with the optimal particle content reaching up to 20%.

3.4 Hardness of composites

Shore hardness measurements (also called durometer) are performed using the Shore A or Shore D scale as per ASTM D2240 standards. The Shore A scale is performed for softer materials, whereas the Shore D scale is used for rubbers or elastomers. The pointer of the durometer is between 0 and 100 when load is applied onto the pointer. The higher

number indicates the hardness of the material. The method involves measuring the penetration depth of an indenter with a conical tip into the surface of a sample with 4500 g. Five readings are taken for each sample at different locations on the surface of the flat orientation, and the average is taken as the hardness of the sample. The Fig. 10 illustrates the variations in Shore D hardness of PLA composites and shows that PLA composite samples are better than the neat PLA samples. The fluctuations in hardness within the composites are primarily attributed to agglomerations caused by the reinforcement particles, which are more susceptible to porosity. Additionally, refinement might enhance interfacial bonding and robust network structure. Conversely, hardness diminished due to insufficient dispersion of the reinforced particles, resulting from agglomeration and the formation of voids.

3.5 Flexural strength of composites

Figure 11. Shows the Flexural strength of composites. It is reported from the graph that flexural strength is improved with the addition of reinforcement particles. The inclusion of bone powder in PLA enhances energy absorption under load during testing. This reinforcement may function as an impact modifier, improving the toughness and ductility of the composites. A strong interfacial energy between PLA and the reinforcement facilitates better dispersion of the reinforcement. A similar type of trend has been reported by other researchers. However, excessive reinforcement can lead to decreased energy absorption due to particle agglomeration and stacking, which can be mitigated by optimizing selection parameters. The enhancement in toughness may be attributed to the heterogeneous NU Latino effect of bone powder combined with PLA, as well as the mechanical interlocking between the reinforcement and PLA, which improves the crystallinity of the composites. Figure 12 shows that the surface plot of UTS is improved with the increase of dwell time and reinforcement. The prolonged dwell time could reduce the defects and form the bond. Figure 13 shows the surface plots of UTS that interact between reinforcement particles and found an improvement of UTS with an increase in both parameters. The proper bonding between reinforcement and matrix material could be the reason for higher UTS. A similar trend is also observed with the surface plot of

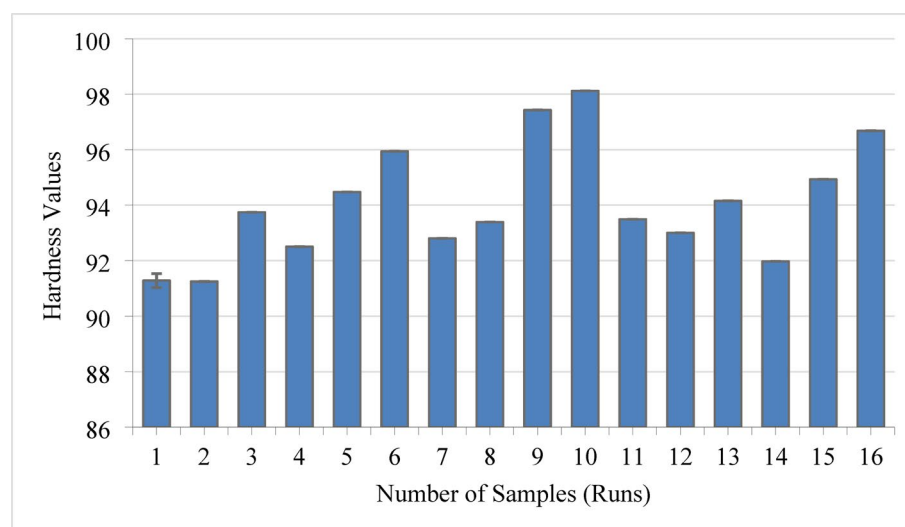


Fig. 10 Results of Hardness of composites

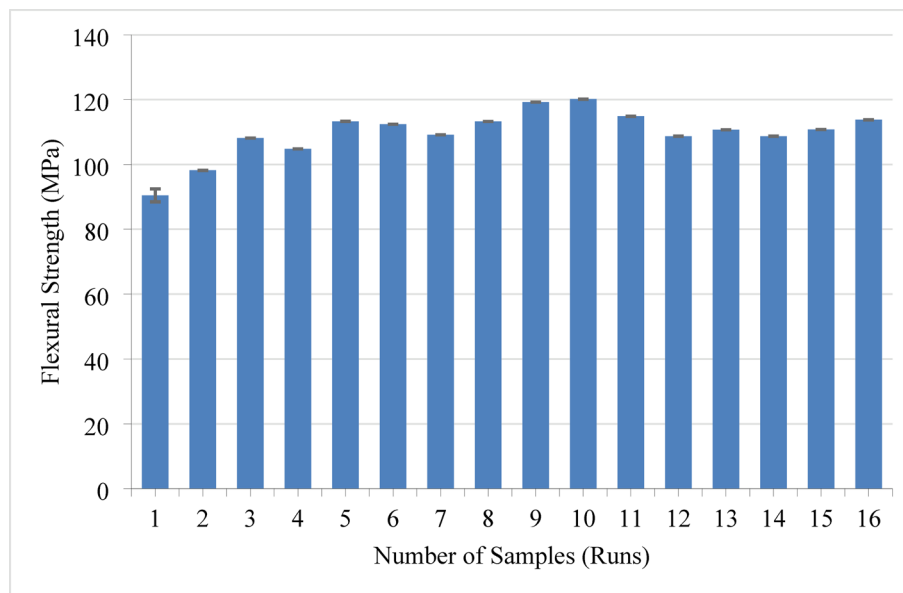


Fig. 11 Flexural strength of composites

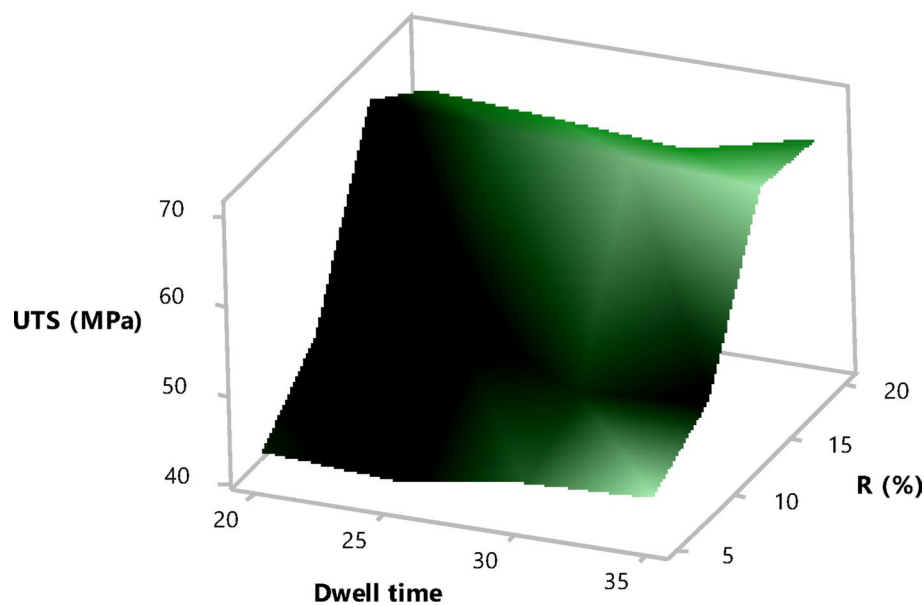


Fig. 12 Surface plot of UTS(MPa) Vs R%, Dwell time(s)

UTS vs. pressure (MPa), Reinforcement in Fig. 14. The UTS is found with low compression and high die temperature as per the interaction of Fig. 15.

3.6 Microstructure analysis

The microstructure of the composite materials was examined through scanning electron microscopy. The particles are dispersed in the matrix material due to the mixing of the matrix material and reinforcement particles at higher temperatures. It is observed that a heterogeneous microstructure was formed due to Incorporation of bone powder filler reinforcement into the PLA matrix. Distribution of filler particles within the matrix is uneven, resulting in fluctuations in mechanical properties. Increasing the weight

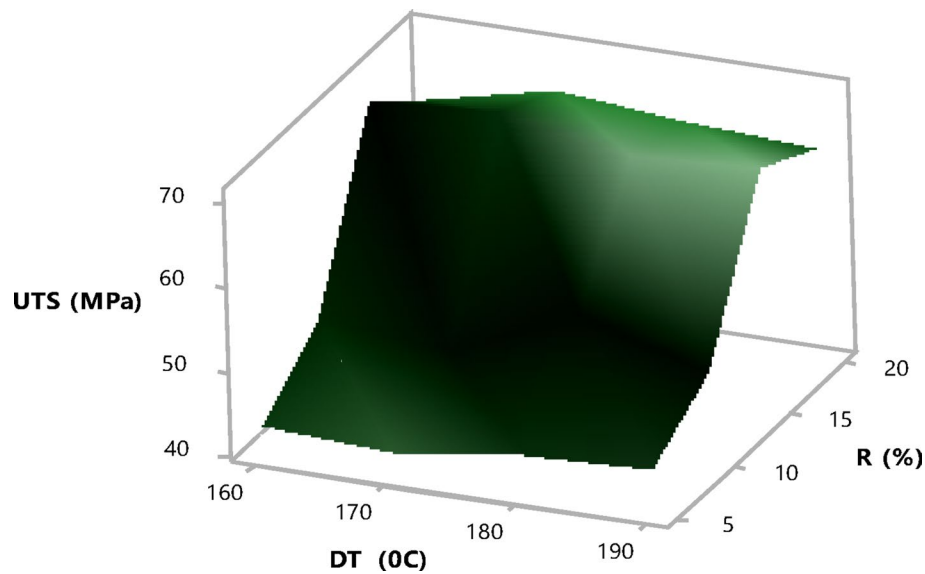


Fig. 13 Surface plot of UTS Vs DT(°C), R%

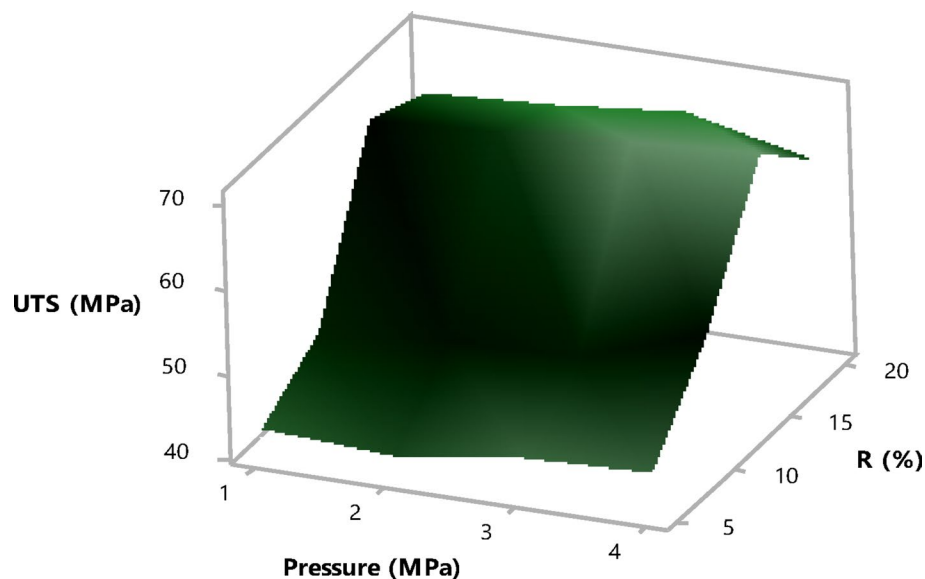


Fig. 14 Surface plot of UTS Vs Pressure (MPa), Reinforcement

percentages of fillers enhances the heterogeneity of the microstructure. Figure 16 illustrates the SEM image of bone powder, which has different size of different sizes ranging from 100 microns and 540 microns. The porosity and blow holes are found due to a weaker bond between the matrix materials and reinforcement particles. Figure 17 shows a 15% reinforcement PLA composite, which has higher tensile strength. The uniform distribution of particles is attributed to higher strength due to interfacial adhesion between particles and matrix material. Figure 18 shows the SEM image lower strength of the PLA composite for PLA + 10% animal bone powder. This adversely affected the mechanical properties of the PLA composite, potentially due to inadequate bonding between the PLA matrix and the bone powder particles and the barrier to dislocation movement of the composites.

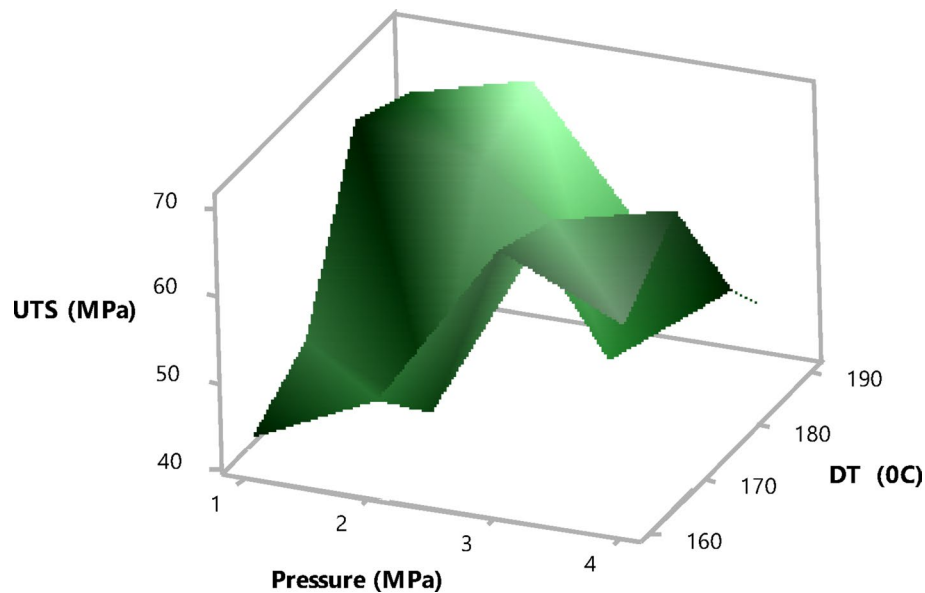


Fig. 15 Surface plot of UTS Vs Pressure (MPa), DT(°C)

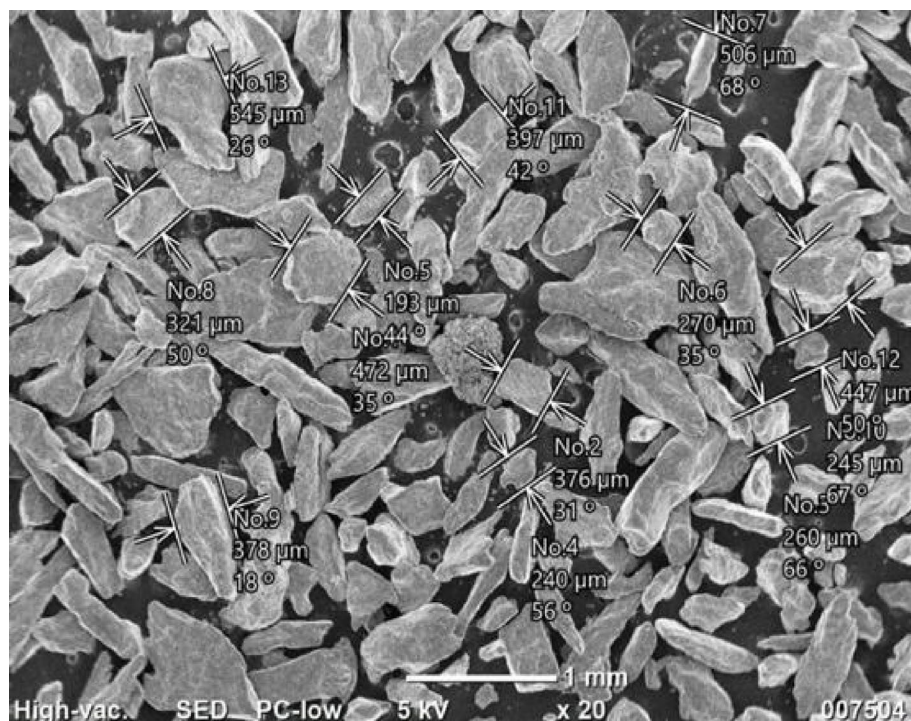


Fig. 16 SEM Image of animal bone powder

4 Conclusions

The PLA bone powder reinforced composites are successfully fabricated using a low-cost compression molding process. Incorporating animal bone powder can greatly enhance the mechanical characteristics of PLA-based composite materials. The composites created with these fillers demonstrated increased stiffness and strength, although this was accompanied by a reduction in ductility. The highest values of hardness (98.12) were obtained for 15% reinforcement, which is more than neat PLA. The tensile strength

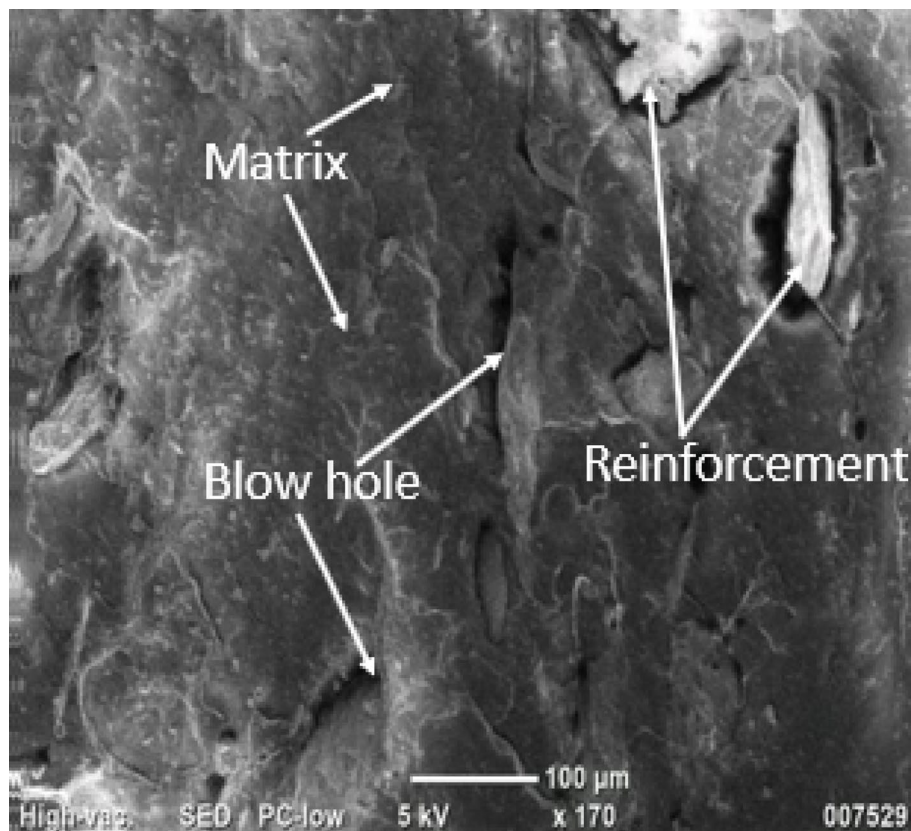


Fig. 17 SEM Image of PLA + 15% animal bone powder

of 98 MPa for 15% reinforcement, which is more than neat PLA. It is found that flexural strength is improved with the addition of reinforcement. It is found that hardness and tensile strength were improved by 24.38% and 19.26% respectively. The flexural strength of composites was improved by 15% to the neat PLA samples. The process parameters are optimized for better tensile strength. The optimal values of the process are at 15% reinforcement, die temperature of 190 °C, Compression pressure of 3 MPa, and dwell time of 35 min. The ANOVA shows that reinforcement has a 97.4% contribution to achieving better strength of composites. The confirmation test has been carried out with optimal values and a validated model. The enhanced performance may be attributed to the reduced particle size and the improved distribution of micro-bone powder particles within the PLA matrix.

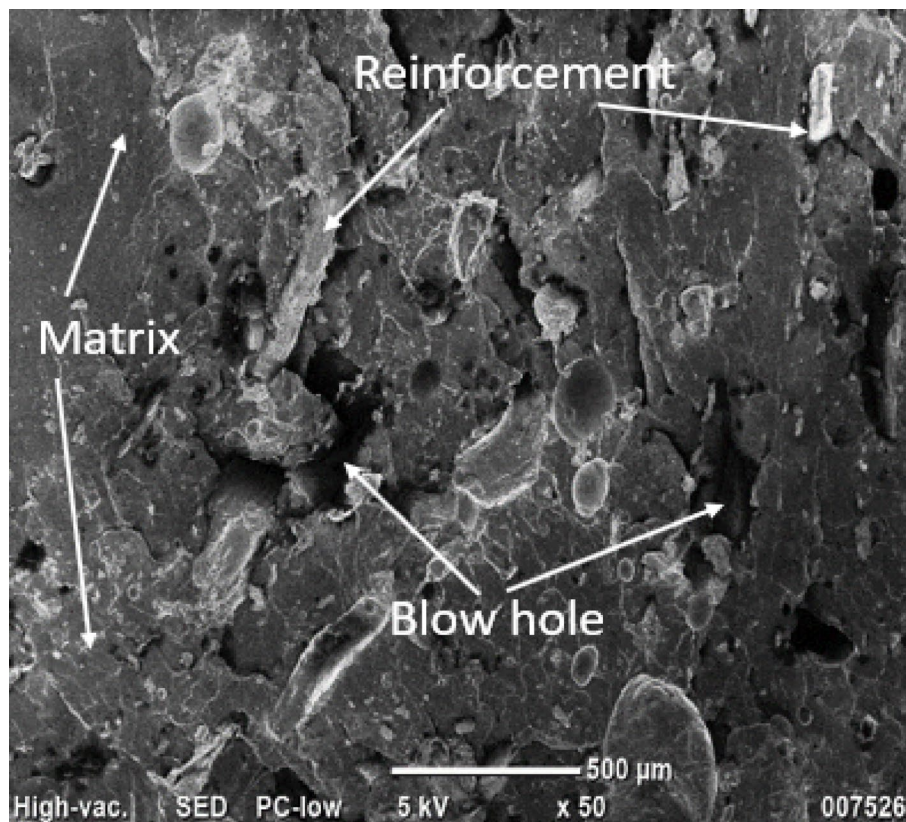


Fig. 18 SEM Image of PLA + 10% animal bone powder

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Author contributions

Santha Rao Dakarapu: Writing-original draft preparation, Investigation, Methodology, Visualization. Praveena Chakravarthy Maddili: Data curation, Methodology, Visualization, Reviewing and Editing, Javvadi Eswara Manikanta: Investigation, Data curation, Reviewing and Editing. Nitin Ambhore: Investigation, Data curation, Reviewing and Editing, Supervision. Attila Szántó: Data curation, Visualization, Reviewing, and Editing, Funding acquisition. Timotei István Erdei: Visualization, Reviewing and Editing, Supervision, and Funding acquisition.

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Data availability

All data generated or analysed during this study are included in this published article.

Declarations

Ethics approval

Not applicable.

Competing interests

The authors declare no competing interests.

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