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Design and Characterisation of PLA-Based Biocomposite Filaments Reinforced with Pecan Shell for 3D Printing

Gliden Jaroslav Dubois-Makarova and Alejandro Antonio Lara-Ruiz
Bachelor's Degree in Industrial Engineering
Carrera de Ingeniería Industrial, Universidad de Lima, Perú
20170500@aloe.ulima.edu.pe, 20173522@aloe.ulima.edu.pe

Juan Carlos Quiroz-Flores and Marcos Fernando Ruiz-Ruiz
Research Professors
Carrera de Ingeniería Industrial, Universidad de Lima, Perú
jcquiroz@ulima,edu.pe, mruiz@ulima.edu.pe

Abstract

This study designed, produced, and validated 3D printing filaments based on polylactic acid (PLA) reinforced with pecan shell powder, with the aim of assessing their technical and functional feasibility as a sustainable biocomposite suitable for extrusion and 3D printing processes. Formulations containing 5 % and 10 % of agricultural residue were developed, controlling the particle size of the additive to prevent clogging during extrusion and ensure a homogeneous dispersion within the polymer matrix. The characterisation of the filaments included tensile and impact mechanical tests, as well as thermogravimetric analysis (TGA). Standardised specimens were fabricated via 3D printing to evaluate the material's processability and the dimensional stability of the printed parts. The formulation containing 5 % pecan shell exhibited the best overall performance, showing increases of 18.2 % in Stress Peak, 8.7 % in Young's Modulus, and 5.9 % in Impact Strength compared to pure PLA, as well as a slight improvement in thermal stability for the biocomposite. These findings confirm the technical feasibility of using pecan shell, an agricultural by-product native to the Peruvian context, as a natural additive in the manufacturing of biocomposite filaments. The research contributes to the revaluation of agro-industrial waste and to the development of more sustainable and cost-efficient additive manufacturing, proposing a functional biocomposite material with potential applications in the 3D printing of lightweight components, biodegradable packaging, and decorative parts with low to moderate mechanical requirements.

Keywords
3D printing filament, pecan shell powder, polylactic acid (PLA), biocomposite, circular economy.

1. Introduction
The growing dependence on petroleum-derived synthetic polymers has generated a global environmental challenge, as the degradation of such materials can take between 10 and 450 years (Mikula et al. 2020). Morales et al. (2023) report that, in the case of Colombia, the plastics sector reached a production of 1.36 million metric tons in 2020, with a recycling rate of only 8.7 %. This figure is considerably lower when compared to the European Union, where Mikula et al. (2020) indicate that 75.1 % of plastic waste was processed (32.5 % through recycling and 42.6 % through energy recovery), while 24.9 % was sent to landfills. In the Peruvian context, APIPLAST (2024) states that annual production amounts to approximately 1.2 million metric tons, of which only 10 % is properly recycled, demonstrating a substantially lower waste-management performance relative to international standards.

Considering this scenario, biopolymers have emerged as a strategic alternative to mitigate plastic pollution. Among them, polylactic acid (PLA) has become a reference material due to its bio-based origin, biodegradability, and suitable

mechanical properties for multiple applications (Kristiawan et al. 2021). This polymer is obtained from renewable sources such as corn or sugar cane and is widely used in the packaging industry, the biomedical sector and, as the main input, in additive manufacturing. Nevertheless, PLA exhibits inherent limitations, such as brittleness, low tensile strength, and limited thermal stability, which restrict its use in demanding structural applications. With the aim of overcoming these limitations, several studies have investigated the incorporation of natural reinforcements and lignocellulosic residues to enhance its performance (Paternina et al. 2023; Morales et al. 2021; Barreto et al. 2024; Aliotta et al. 2024).

In Peru, one of the agro-industrial residues with the greatest revaluation potential is pecan shell. The cultivation of this nut has experienced sustained growth over the past decade, exceeding 3700 hectares of cultivated surface, primarily concentrated in the regions of Ica, Lima, and Arequipa (MIDAGRI, 2025). This production process generates a considerable volume of shell waste which, despite its high cellulose and lignin content, is often discarded or used solely as low-value biomass. These characteristics position pecan shell as a potentially suitable reinforcement for the development of polymeric materials; however, its application in the production of 3D printing filaments has not yet been reported to date.

Additive manufacturing, particularly 3D printing using the Fused Deposition Modelling (FDM) technique, has revolutionised the fabrication of prototypes and functional components due to its scalability, cost-effectiveness, and versatility in material processing (Daminabo et al. 2020). Nonetheless, most of the filaments used in this process are manufactured from virgin polymers, thereby perpetuating dependence on non-renewable resources. In this regard, the development of biocomposite filaments formulated with PLA and natural reinforcements represents a promising alternative to reduce environmental impact, optimise raw-material costs, and strengthen circular-economy principles within the field of additive manufacturing.

Within this framework, the present research proposes the utilisation of pecan shell, an agro-industrial residue characteristic of the Peruvian context, as a reinforcing additive to produce PLA-based 3D printing filaments. The study aims to develop a biocomposite material capable of enhancing the mechanical and thermal properties of PLA without compromising its processability or dimensional stability during printing. In this manner, it seeks to provide experimental evidence regarding the incorporation of local agricultural residues into polymeric matrices and their influence on filament performance throughout the additive manufacturing process. Overall, this research contributes to the advancement of sustainable technological alternatives focused on the revaluation of Peruvian agro-industrial waste, thereby promoting a more sustainable, efficient production model aligned with circular economy principles.

1.1 Objectives

The present investigation aims to design and manufacture 3D printing filaments derived from sustainable, low-environmental-impact materials, directed toward optimising functional performance and reducing the costs associated with the use of virgin polymers in additive manufacturing processes. The study focuses on evaluating the influence of pecan shell incorporation on the mechanical and thermal properties of the filament, establishing direct comparisons with the behaviour of pure polylactic acid (PLA) as the reference base material. Moreover, the optimal formulation and processing parameters are determined to maximise the resistance, thermal stability, and processability of the biocomposite, without compromising extrusion or print quality. To this end, the effects of different additive concentrations (5 % and 10 %) are analysed with respect to variables such as Stress Peak, Young's Modulus, Impact Strength, and thermal stability. The aim of this analysis is to establish the functional viability of the developed biocomposite and its potential application as a sustainable, technically feasible, and lower-cost alternative within the field of additive manufacturing.

2. Literature Review

Additive manufacturing has established itself as one of the most transformative technologies in modern engineering due to its capacity to produce customised components with complex geometries, high dimensional accuracy, and more efficient material utilisation. As noted by Mohanavel et al. (2021), this technology is reshaping production systems by enabling the fabrication of lighter components with significantly shorter manufacturing times compared to conventional methods, thereby underscoring its strategic role in industrial and technological development. Among the diverse techniques encompassed within additive manufacturing, Fused Deposition Modelling (FDM) stands out for its accessibility, versatility, and low operational cost, as it employs thermoplastic filaments that are melted and deposited sequentially layer by layer through a controlled extrusion head (Dey et al. 2021). Owing to this flexibility,

3D printing exhibits substantial application potential across sectors such as aerospace, automotive, biomedical, construction, electronics, food, architecture, and design (Shahrubudin et al. 2019).

Among the polymers most widely used in FDM are acrylonitrile butadiene styrene (ABS) and polypropylene (PP); however, biopolymers such as polylactic acid (PLA) have emerged as more sustainable alternatives. Its ease of processing, low melting temperature, and compatibility with most 3D printers position PLA as the predominant matrix for filament production. Nevertheless, its intrinsic limitations in mechanical and thermal resistance have encouraged the incorporation of natural reinforcements with the aim of enhancing its performance and functionality.

In this regard, the use of agricultural residues as natural reinforcements in polymer matrices has demonstrated significant benefits, both in terms of material performance and sustainability indicators within the production process. Numerous studies have examined the incorporation of additives such as rice husk (Barreto et al. 2024), wood flour (Tokdemir and Altun, 2022; Petchwattana et al. 2019), cashew nutshell (Paternina et al. 2023), kenaf fibre (Acevedo et al. 2020), among others. The literature suggests that, depending on the type, particle size, and concentration of the additive, it is possible to increase stiffness, thermal stability, and impact resistance, while simultaneously preserving material biodegradability and reducing production costs (Mazzanti et al. 2019). Furthermore, the partial substitution of virgin polymers with agro-industrial residues contributes to reducing the consumption of non-renewable resources and, according to Oladapo et al. (2023), can decrease CO_2-equivalent emissions by up to 30 % in comparison with materials manufactured exclusively from conventional polymers.

From an operational standpoint, the final quality of the filament depends on both the adequate conditioning of raw materials and the rigorous control of extrusion parameters. PLA and the corresponding additives must be previously grinded, dried, and sieved, maintaining a recommended particle size between 250 and 425 µm, as reported by Morales et al. (2021), to ensure homogeneous dispersion of the reinforcement within the polymer matrix prior to extrusion. Hachimi et al. (2021) describe that the extrusion process begins with the feeding of the mixture, which is conveyed by a screw into a thermally resisted heated barrel. The molten mass is subsequently compressed and forced through a nozzle, yielding a filament with standard diameters of 1.75 mm or 2.85 mm. Kristiawan et al. (2022) emphasise that extrusion temperature and feed rate constitute critical parameters whose optimal combination determines filament performance and quality, varying according to the type of polymer and reinforcement employed. These factors directly influence filament processability and the quality of the printed parts, making their optimisation essential for obtaining consistent and functional materials.

Subsequently, filament validation is conducted through standardised tests that allow the characterisation of mechanical and thermal properties in accordance with international standards. The most frequently employed include the tensile test (ASTM D638), the Izod impact test (ASTM D256), and thermogravimetric analysis (ISO 11358-1), which provide essential information regarding material strength, toughness, and thermal stability. The results obtained enable correlating biocomposite composition with functional performance and, consequently, determining its suitability for diverse applications in additive manufacturing. Several studies report notable improvements in these indicators, such as a 22.8 % increase in Young's Modulus for ABS filaments reinforced with 7 % oil palm fibre (Ahmad et al. 2023); an increase in degradation temperature up to 336 °C when incorporating 10 % coconut fibre (Trivedi et al. 2023); and a 22.2 % increase in impact resistance for PLA filaments with 1 % rice husk (Barreto et al. 2024).

The reviewed literature strongly supports the use of natural fibres and agricultural residues as reinforcements in polymer matrices, evidencing their capacity to improve mechanical and thermal properties as well as the sustainability of filaments. Nevertheless, no studies have been identified that evaluate the use of pecan shell as an additive in PLA matrices, nor that examine its behaviour during extrusion and 3D printing processes. Considering its high cellulose and lignin content, its availability in the Peruvian context, and its low cost, pecan shell represents a resource with high potential for the development of functional biocomposites with reduced environmental impact. In this context, the present research seeks to demonstrate the technical and functional feasibility of employing this agro-industrial residue to produce sustainable filaments for additive manufacturing, thereby contributing to the exploration of new biocomposites applicable to 3D printing.

3. Methods

The present study adopted a quantitative, experimental approach aimed at evaluating the technical and functional viability of 3D printing filaments fabricated from polylactic acid (PLA) and pecan shell. The experimental design allowed for the controlled manipulation of independent variables, namely additive percentage, extrusion temperature,

and winding speed, with the purpose of assessing their effect on the dependent variable, filament diameter, and indirectly on the material's mechanical and thermal properties under laboratory conditions. The methodology was structured in five phases: raw-material conditioning, filament extrusion, process optimisation via Taguchi design, 3D printing of standardised specimens, and functional validation through mechanical and thermal testing. An overview of the methodological sequence is presented in Figure 1.

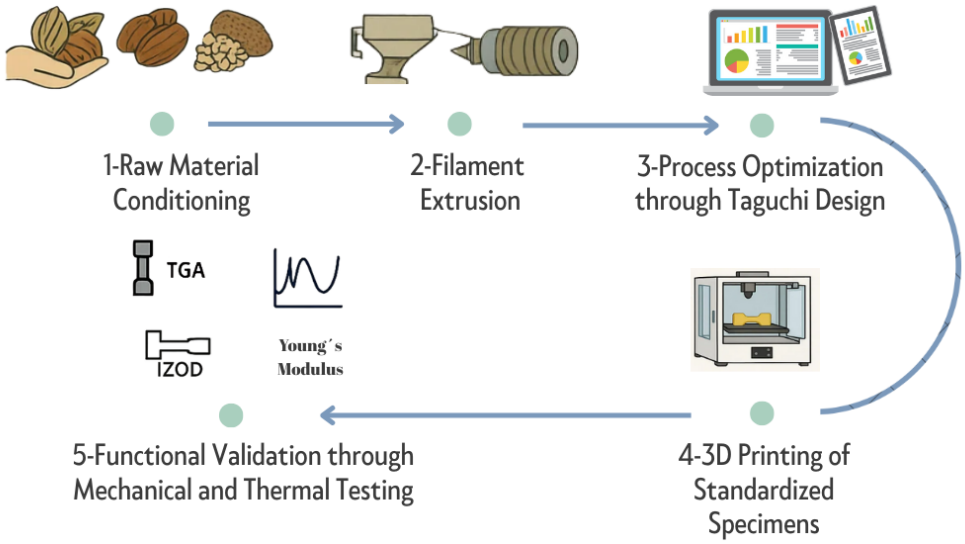

Figure 1. General methodological sequence of the process

The shells of pecans sourced from agro-industrial residues in the southern region of Peru and the polylactic acid (PLA) from the brand NatureWorks Ingeo Biopolymer 2003D (imported from Germany) were subjected to a grinding process using a Grondoy GR-PV30B knife mill and subsequently sieved until obtaining particles sized between 250 µm and 420 µm, in accordance with ASTM E11. The particulate material was thermally dried at 70 °C for 30 minutes in an industrial oven to eliminate surface moisture. This step is critical since elevated residual moisture can lead to defects such as bubbles, porosities or thermal degradation during processing (Morales et al. 2021). Moisture control of the PLA was performed following ASTM D6980, and for the lignocellulosic residue ASTM D4442 was applied, ensuring moisture levels below 0.5 % and 10 %, respectively. Such pre-conditioning proved essential in reducing residual moisture, promoting compatibility between components and ensuring homogeneous dispersion of the reinforcement within the polymer matrix, an indispensable condition for achieving extrusion stability and obtaining filaments of constant diameter. The raw-material conditioning process is depicted in Figure 2.

Figure 2. Conditioning of pecan shell and PLA

Extrusion was carried out in a single-screw extruder model SJ15 (China), equipped with dual-zone thermal control. Three formulations were processed according to the weight concentration of the additive: F0 (100 % PLA), F5 (95 %

PLA + 5 % pecan shell) and F10 (90 % PLA + 10 % pecan shell). The thermal operation range spanned from 140 °C to 160 °C, with winding speeds of 9, 11 and 13 rpm, yielding filaments with a nominal diameter of 1.75 ± 0.05 mm. The extruded material was cooled by forced air and subsequently wound on spools using an automatic winding system, thereby ensuring a continuous surface and adequate circularity of the filament. The stages of the described process are illustrated in Figure 3.

Figure 3. Extrusion and winding of PLA- pecan shell filaments

The definition of factors and levels for the experimental design was based on the prior application of the Quality Function Deployment (QFD) methodology, which enabled the translation of user requirements — filament cost, printed-part quality and sustainability — into controllable technical variables of the extrusion process. For optimisation, a Taguchi L9 (3^3) design was employed, considering three factors (temperature, winding speed and additive percentage) each at three levels. Statistical analysis was conducted using Minitab 19, employing the signal-to-noise ratio type "nominal is the best" to determine the optimal combination of parameters that maximises filament dimensional stability. Additionally, the statistical power of the experimental design was calculated, yielding a value of 0.979, which exceeds the minimum recommended threshold of 0.80 according to Montgomery (2021), thereby assuring result reliability. Mean values, standard deviations and signal-to-noise ratios for the nine runs of the L9 design are presented in Table 1.

Table 1. Experimental results and signal-to-noise ratio values obtained from the Taguchi L9 design

Run	Factor A	Factor B	Factor C	Mean	Std.Dev.	Coef. Var.	S/N
1	0 %	140	9	1.851	0.056	0.031	30.31
2	0 %	150	11	1751	0.011	0.006	44.19
3	0 %	160	13	1.610	0.068	0.042	27.54
4	5 %	140	11	1.753	0.011	0.006	43.95
5	5 %	150	13	1.634	0.037	0.022	32.99
6	5 %	160	9	1.891	0.048	0.025	31.99
7	10 %	140	13	1.581	0.044	0.028	31.03
8	10 %	150	9	1.754	0.012	0.007	43.15
9	10 %	160	11	1.633	0.034	0.021	33.67

Complementarily, Statistical Process Control (SPC) was applied to the critical variable of filament diameter, with the aim of identifying the most stable and capable extrusion conditions. For each parameter combination, 30 consecutive diameter measurements were recorded in accordance with ASTM E122, ensuring the statistical representativeness of the sample. The collected data were analysed using I–MR control charts and process capability analysis, calculating the Cp and Cpk indices following the methodology described by Montgomery (2021). As an acceptance criterion, a

process was considered statistically capable when Cp or Cpk values were greater than or equal to 1.33, indicating stable performance and variability within specification limits. The results are presented in Table 2.

Table 2. Statistical Process Control (SPC) applied to filament diameter

Run	Cp	Cpk
1	0.293	-0.297
2	1.529	1.509
3	0.245	-0.442
4	1.486	1.407
5	0.451	-0.599
6	0.347	-0.630
7	0.372	-0.888
8	1.354	1.246
9	0.488	-0.650

Although the Taguchi L9 design enabled the identification of the most influential factor and the estimation of overall optimal processing parameters, the aim of this research was not to restrict the analysis to a single optimal condition, but rather to comparatively evaluate the performance of three distinct formulations (F0, F5, and F10). Consequently, a set of stable conditions was selected for formulations corresponding to runs 2, 4 and 8 for F0, F5 and F10 respectively (validated by SPC), from which a representative roll was produced for each formulation (Figure 4), ensuring dimensional homogeneity and adequate surface quality.

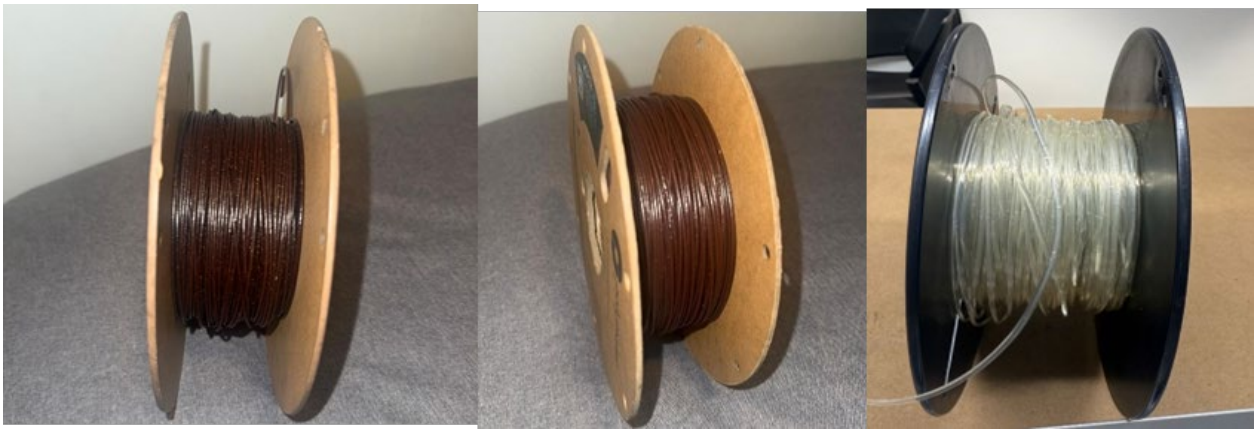

Figure 4. Final PLA–pecan shell filament spools obtained for each formulation (F10, F5, and F0)

The filaments obtained were subjected to thermal drying at 60 °C for 4 hours prior to being used for the fabrication of standardised test specimens through 3D printing. The printing process was conducted using a MakerBot Replicator 2 printer, configured with the following parameters: nozzle temperature of 210 °C, nozzle diameter of 1.2 mm, layer height of 0.3 mm, and printing speed of 90 mm/s. The samples were prepared in accordance with ASTM D638 (Type I) for tensile testing and ASTM D256 for impact testing, employing five replicates per formulation. The printed specimens produced from the developed filaments are shown in Figure 5.

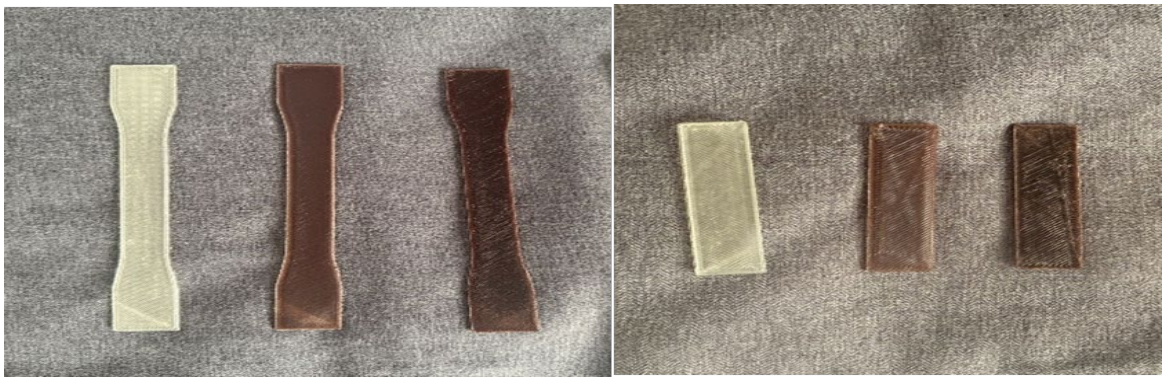

Figure 5. 3D-printed specimens for tensile and impact testing by formulation (F0, F5, and F10)

The functional validation included the following tests:

Tensile testing: performed in a Testometric X500-50 universal testing machine (50 kN load cell, 1 mm/min speed), recording Stress Peak (MPa), Young's Modulus (MPa) and Strain Break (%).

Izod impact testing: conducted on a Zwick Roell RKP450 pendulum (450 J capacity), using single-notch method and recording energy absorbed (kJ/m²).

Thermogravimetric analysis (TGA): carried out in a Bruker TGA-IR instrument under nitrogen atmosphere (50 ml/min), with a heating rate of 10 °C/min from 30 °C to 850 °C, determining onset degradation temperature (Tonset), maximum degradation temperature (Tmax) and residual mass (%).

The equipment used is depicted in Figure 6.

Figure 6. Equipment used for tensile, impact, and TGA testing respectively

The experimental results were processed using Minitab 19, applying descriptive statistical analysis of mean values and standard deviations, along with a one-way ANOVA to determine the presence of statistically significant differences among the formulations evaluated, at a 95 % confidence level. The methodology sequentially integrated the tools QFD, Taguchi, SPC, and ANOVA, linking the voice of the customer, process parameter optimisation, process stability control, and statistical validation of the results. This combination ensured the reproducibility, consistency, and technical feasibility of the biocomposite filaments developed, consolidating a robust experimental approach oriented toward sustainable additive manufacturing.

4. Data Collection

The experimental data collection was conducted with the purpose of quantitatively assessing the mechanical and thermal performance of the developed filaments, ensuring the reliability of the results and their subsequent statistical

analysis. All tests were performed under controlled environmental conditions of 23 ± 2 °C and 50 ± 10 % relative humidity, in accordance with ASTM D618 for the conditioning of plastic materials.

For the mechanical tests, five specimens per formulation were evaluated, obtained from the final batch of extruded filaments validated in Chapter 3, following the requirements of their corresponding technical standards. The recorded values served as the basis for the comparative analysis between pure PLA (F0) and the biocomposite formulations F5 and F10. The methods and variables measured in each test are described below.

In the tensile test (ASTM D638, Type I), three key parameters were determined:

Stress Peak (σ): the maximum stress sustained at the fracture point of the specimen, calculated as the maximum force divided by the initial cross-sectional area.

$$Stress\ Peak\ (MPa)\ \rightarrow \sigma_{\max} = \frac{F_{\max}}{A_0}$$

where:

$$F_{\max} = \text{maximum force recorded during the test (N)}$$
$$A_0 = \text{initial cross} - \text{sectional area of the specimen (mm}^2\text{)}$$

Young's Modulus (E): the slope of the elastic region of the stress–strain curve, representing the material's stiffness.

$$Young's\ Modulus\ (MPa)\ \rightarrow E = \frac{\Delta\sigma}{\Delta\varepsilon}$$

where:

$$\Delta\sigma = \sigma_2 - \sigma_1 = \text{stress increment between two points in the linear region (MPa)}$$
$$\Delta\varepsilon = \varepsilon_2 - \varepsilon_1 = \text{ corresponding strain increment (dimensionless)}$$

Strain Break (ε): the percentage of elongation relative to the initial gauge length of the specimen.

$$Strain\ Break\ (\%) \rightarrow \varepsilon_{\text{break}} = \frac{L_f - L_0}{L_0} \times 100$$

where:

$$L_f = \text{final length of the specimen at fracture (mm)}$$
$$L_0 = \text{initial reference length (mm)}$$

The stress–strain curves were generated using the winTest Analysis EC software, which automatically recorded force and displacement in real time and processed the data to obtain the mechanical parameters for each specimen. All samples were printed with their longitudinal axis aligned with the load direction to ensure uniform stress distribution. The dimensions of the Type I tensile specimens are shown in Figure 7.

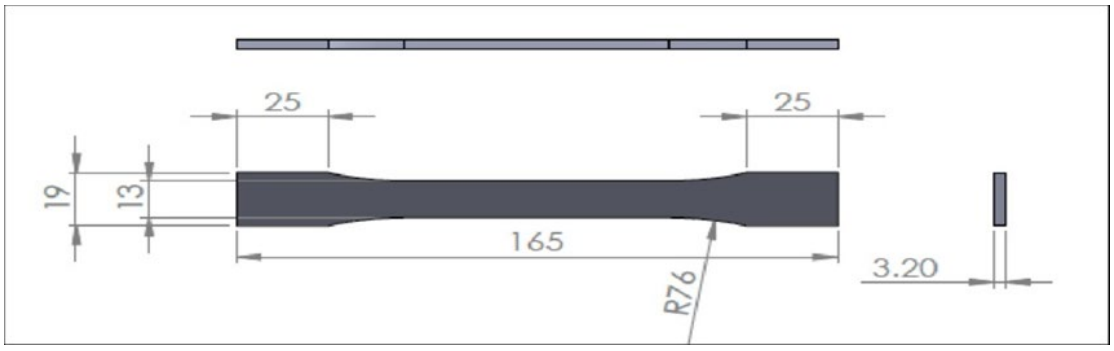

Figure 7. Dimensions of type I specimen according to ASTM D638

The Izod impact test, performed following ASTM D256 (Method A), enabled the determination of the energy absorbed during fracture of a single-notch specimen, a parameter indicative of the filament's toughness and resistance to

dynamic loading. The absorbed energy values were obtained directly from the digital system of the impact pendulum, expressed in kJ/m² and averaged for each formulation. Specimens were printed in the longitudinal direction to minimise variability in energy absorption. The standardised geometry of the impact specimen is presented in Figure 8.

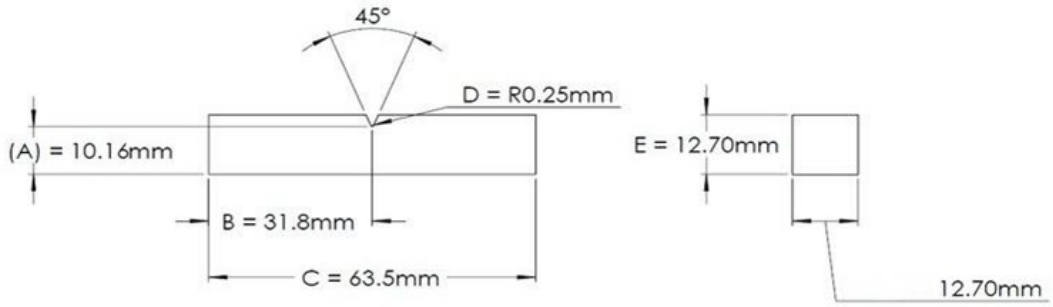

Figure 8. Specimen dimensions for Izod impact test according to ASTM D256

Lastly, thermogravimetric analysis (TGA) was carried out to evaluate the thermal behaviour of the materials in accordance with ISO 11358-1. The parameters recorded were:

Tonset (°C): temperature at which thermal degradation begins, calculated using the tangent extrapolation method.
Tmax (°C): temperature corresponding to the maximum degradation rate.
Residual mass (%): remaining mass after complete decomposition at 850 °C.

The analysis was conducted under nitrogen atmosphere (50 mL/min) with a heating rate of 10 °C/min, generating TGA and DTG curves subsequently used in the comparative evaluation presented in Chapter 5. This test provided insight into the thermal stability of the composites and their resistance to temperature-induced degradation.

For clarity, Table 3 summarises the test conditions, applicable standards, variables measured, and total number of valid samples collected.

Table 3. Summary of experimental data collected by type of test

Test	Technical Standard	Variables Measured	Valid Samples (n)
Tensile	ASTM D638 (Type I)	Stress Peak (MPa), Young's Modulus (MPa), Strain Break (%)	15 (5 per formulation)
Izod Impact	ASTM D256 (Method A)	Impact Strength (kJ/m²)	15 (5 per formulation)
TGA	ISO 11358-1	Tonset (°C), Tmax (°C), Residual mass (%)	3 (1 per formulation)

The collected data constitute the dataset for the statistical processing and comparative interpretation of results, which are developed in the following chapter.

5. Results and Discussion

5.1 Numerical Results

The experimental results obtained allowed the mechanical and thermal behaviour of the three filament formulations developed to be quantified. The average properties and their respective standard deviations are presented in Table 4.

Table 4. Average mechanical and thermal properties by formulation (mean ± standard deviation)

Test	Variable	F0	F5	F10	F0 vs F5	F0 vs F10	F5 vs F10
Tensile Test	Stress Peak (MPa)	32.9 ± 2.4	38.9 ± 0.7	32.7 ± 2.2	18.2 %	–0.8 %	–16.1 %
	Young´s Modulus (MPa)	2457.1 ± 125.2	2670.4 ± 70.9	2581.5 ± 51.0	8.7 %	5.1 %	–3.3 %
	Strain at Break (%)	3.21 ± 0.56	2.28 ± 0.26	1.84 ± 0.19	–28.8 %	–42.5 %	–19.3 %
Impact Test	Impact Strength (kJ/m²)	2.86 ± 0.65	3.03 ± 0.46	2.72 ± 0.54	5.9 %	–4.9 %	–10.2 %
TGA	Tonset (°C)	320	323	327	0.9 %	2.2 %	1.2 %
	Tmax (°C)	351	353	357	0.6 %	1.7 %	1.1 %

The results reveal a significant increase in the mechanical properties for the formulation reinforced with 5 % pecan shell. Specifically, the Stress Peak increased by 18.2 %, Young's Modulus by 8.7 %, and Impact Strength by 5.9 % compared to pure PLA. However, a progressive reduction in Strain Break was observed as the additive content increased, suggesting a decrease in the material's ductility. At the thermal level, the TGA analysis showed a slight shift of the degradation temperatures (Tonset and Tmax) toward higher values, indicating an improvement in the thermal stability of the biocomposite as the reinforcement percentage increased.

The analysis of variance (ANOVA) performed for each variable allowed the identification of statistically significant differences ($\alpha = 0.05$) among the formulations, as presented in Table 5. The variables Stress Peak, Young's Modulus, and Strain Break showed p-values below 0.05, confirming the influence of pecan shell content on the mechanical properties of the material. In contrast, the Impact Strength exhibited a high p-value (0.678) and low statistical power (0.10), attributable to the high data dispersion and limited sample size, which prevents establishing conclusive differences among formulations for this variable.

Table 5. Results of the ANOVA analysis and statistical power for the evaluated variables

Variable	p-value	Statistical Power
Stress Peak	0.000	0.985
Young's Modulus	0.008	0.861
Strain Break	0.000	0.997
Impact Strength	0.678	0.100

The numerical results confirm that the moderate addition of 5 % pecan shell enhances the mechanical performance of the filament without compromising its thermal stability, whereas higher additive proportions tend to reduce the material's ductility and homogeneity. The graphical analyses and detailed interpretation of these trends are presented in the following section.

5.2 Graphical Results

Prior to the comparative analysis, the normality assumption was verified through visual inspection of the normal probability plots generated for each variable. In all cases, the experimental data points aligned adequately with the reference diagonal, confirming an approximately normal distribution of the datasets. This result validates the use of the ANOVA model for the statistical comparison among the three formulations and supports the reliability of the graphically represented outcomes.

Figures 9 and 10 present the graphical evaluation of the experimental results, enabling the visualisation of data variability, statistical differences, and the combined behaviour of the mechanical and thermal properties for the three formulations.

Figure 9 displays the box plots corresponding to Stress Peak, Young's Modulus, and Strain Break. The low dispersion observed in the data confirms experimental consistency, particularly for formulation F5, which exhibits higher strength and stiffness compared to F0 and F10. The general trend indicates a simultaneous increase in Stress Peak and Young's Modulus with 5 % additive content, accompanied by a reduction in Strain Break, reflecting a shift toward a less ductile behaviour that is characteristic of lignocellulosic biocomposites.

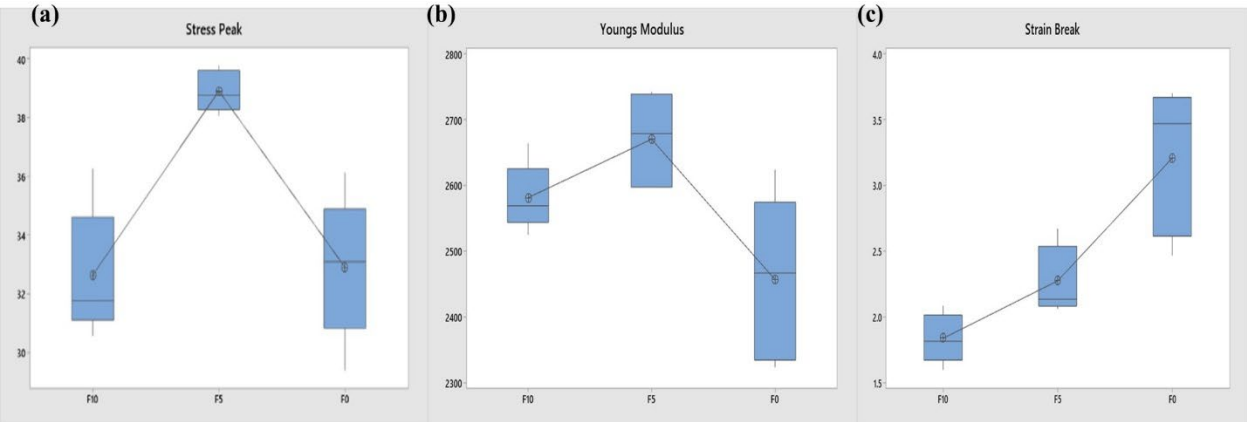

Figure 9. Box plots of tensile test results for each formulation (F10, F5, and F0): (a) Stress Peak, (b) Young's Modulus, and (c) Strain Break

Figure 10 shows the confidence intervals obtained through Tukey's post hoc analysis. It can be observed that the differences between F5 and F0 are statistically significant ($p < 0.05$) for Stress Peak and Young's Modulus, whereas the difference between F10 and F5 is minimal. In the case of Strain Break, a significant reduction is observed compared to pure PLA, indicating a lower plastic deformation capacity attributable to the incorporation of the lignocellulosic reinforcement.

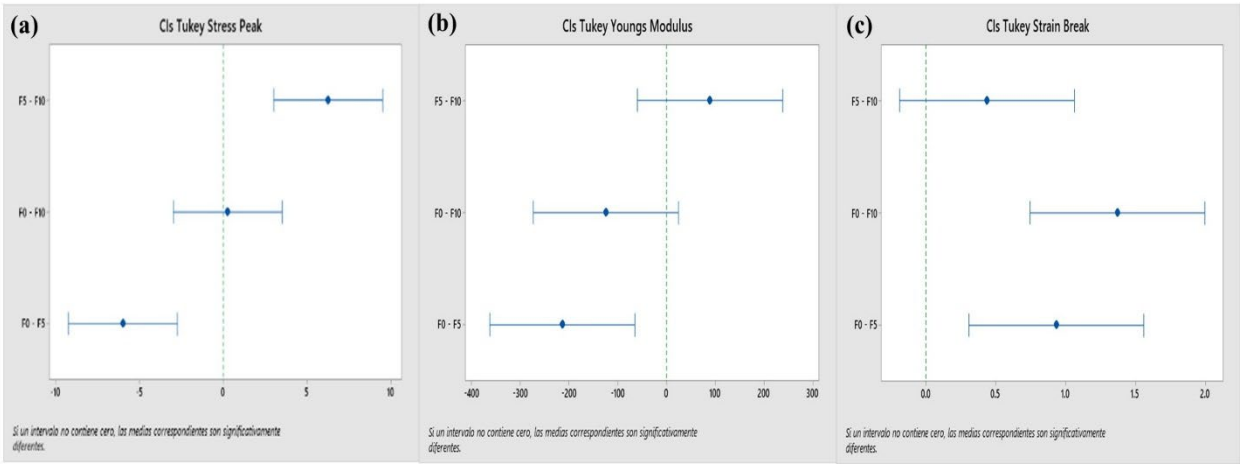

Figure 10. 95 % confidence intervals from Tukey's analysis: (a) Stress Peak, (b) Young's Modulus, and (c) Strain Break

Figure 11 illustrates the results of the impact test. The box plot reveals a relatively high dispersion of data, particularly for formulation F5, which explains the high p-value and low statistical power observed in the ANOVA. This variability is mainly attributed to the limited number of replicates and to the inherent sensitivity of the impact test to small

variations in printing conditions or specimen notching. Nevertheless, the mean value for F5 is slightly higher than that of F0, suggesting a marginal improvement in energy absorption capacity without compromising the structural integrity of the material.

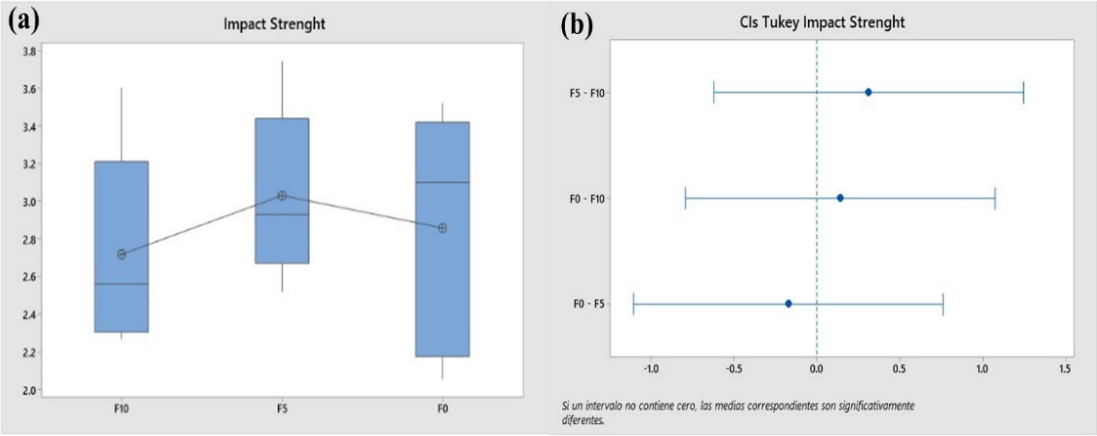

Figure 11. Statistical results of the impact test: (a) box plot of Impact Strength and (b) Tukey analysis with 95 % confidence intervals

Finally, Figure 12 summarises the overall results in a radar chart with normalised values. The F5 formulation exhibits the most balanced performance, achieving the highest relative values in Stress Peak, Young's Modulus, and Impact Strength, without compromising thermal stability. In contrast, F10 shows a slight improvement in Tonset and Tmax, but with a loss of ductility and strength. Pure PLA (F0) maintains a higher Strain Break, although with lower stiffness. This multivariable visualisation confirms that the incorporation of 5 % pecan shell enhances both the mechanical and thermal responses of the material simultaneously.

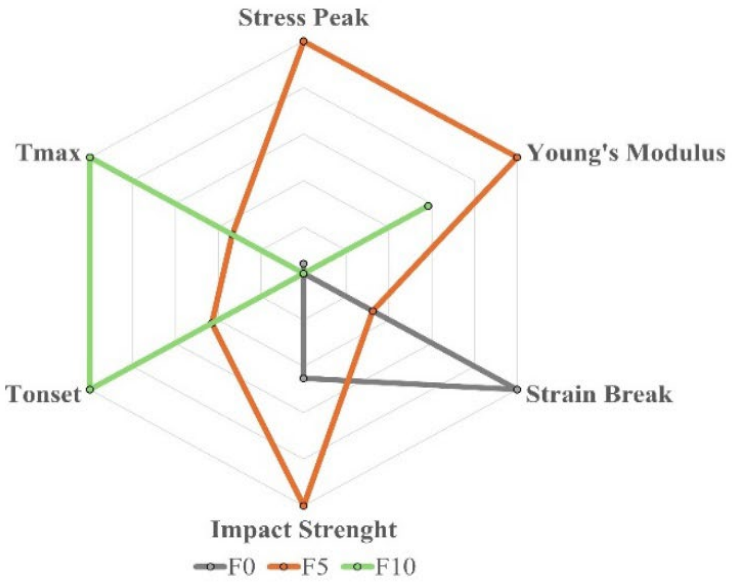

Figure 12. Normalised multivariable comparison of the mechanical and thermal properties for the F0, F5, and F10 formulations

5.3 Proposed Improvements

The results obtained from the Taguchi design provided a robust foundation for process optimisation, identifying an optimal configuration of 5 % pecan shell additive, an extrusion temperature of 150 °C, and a winding speed of 11 rpm,

which ensured the dimensional stability of the filament. Winding speed was identified as the factor with the highest relative variability, highlighting the need to implement automated tension and temperature control during production, complemented by real-time diameter monitoring to minimise deviations and enhance process reproducibility.

From a material standpoint, the main technical challenge identified was the dispersion and interfacial adhesion between the lignocellulosic additive and the PLA matrix, both of which directly influence the mechanical performance of the biocomposite. In agreement with Ahmad et al. (2023), future research should explore the use of compatibilising agents or surface treatments (such as maleic anhydride or alkaline treatment) to strengthen interfacial bonding and promote a more homogeneous microstructure. Likewise, the adoption of a twin-screw extruder and the incorporation of a pre-pelletising stage, as suggested by Morales et al. (2023) and Almeida et al. (2024), are recommended to improve mixing uniformity and prevent clogging during extrusion and 3D printing. These adjustments would significantly reduce dimensional variability and enhance the surface quality of the filament.

From an analytical perspective, it is proposed to integrate advanced characterisation techniques such as FTIR, SEM and moisture absorption tests to gain a deeper understanding of the physicochemical interactions between the matrix and the reinforcement. It is also recommended to increase the number of experimental replicates, especially in the impact test, to increase statistical power and reduce the observed variability.

Finally, in terms of sustainability and industrial scalability, strengthening collaboration with local pecan producers in southern Peru is encouraged to ensure a continuous supply of agricultural residues, reduce raw material costs, and foster the revaluation of agro-industrial by-products. In a hypothetical production scenario, the volume of pecan shell required would represent only 0.29 % of the estimated national supply for 2024, demonstrating high availability and supply feasibility without compromising other potential uses of this resource.

The implementation of a Life Cycle Assessment (LCA) will make it possible to accurately quantify the environmental impacts associated with the production, use, and end-of-life of the filament, identifying the stages with the highest energy demand or emissions, and comparing its performance against conventional PLA. This approach will support the optimisation of the process towards cleaner and more efficient manufacturing, revealing potential reductions in carbon footprint and non-renewable resource consumption. Together, these actions consolidate the development of PLA–pecan shell filament within the principles of the circular economy and in full accordance with Sustainable Development Goal No. 12: Responsible Production and Consumption.

5.4 Validation

The mechanical and thermal indicators obtained in this study were validated through standardised methodologies (ASTM and ISO) and contrasted with recent scientific literature on PLA biocomposites reinforced with plant fibres, confirming the reliability of the results and their consistency with the typical ranges reported.

Validation of Mechanical Properties

The average values obtained for Stress Peak (32.9–38.9 MPa) and Young's Modulus (2457–2670 MPa) fall within the expected ranges for PLA biocomposites reinforced with natural fibres, generally reported between 30 and 50 MPa for tensile strength and 2000 to 3000 MPa for Young's Modulus. In particular, the 18.2 % increase in strength and the 8.7 % increase in stiffness recorded for the formulation containing 5 % pecan shell are aligned with the typical improvements of 20–50 % reported for analogous systems reinforced with vegetal fibres such as kenaf, hemp, or flax, which significantly enhance the strength and modulus of PLA (Plamadiala et al. 2025). These findings confirm the effectiveness of the 5 % pecan shell reinforcement, validating the mechanical performance obtained under the conditions established by ASTM D638.

However, the analysis also revealed a 28.8 % decrease in Strain Break compared to pure PLA, a phenomenon widely documented in the literature (Agaliotis et al. 2022; Pemas et al. 2024). According to Paternina et al. (2023) and Barreto et al. (2024), as the proportion of lignocellulosic additive increases, mechanical properties tend to deteriorate due to weak interfacial adhesion between the reinforcement and the polymer matrix, as well as the higher intrinsic stiffness of the material. This pattern was also observed in the formulation with 10 % pecan shell, which showed a slight reduction in strength and ductility compared to F5, confirming that the optimal reinforcement level is around 5 %, consistent with the findings of Pemas et al. (2024).

Validation of Impact Strength
In the Izod impact test (ASTM D256), the absorbed energy values ranged from 2.72 to 3.03 kJ/m². Despite the dispersion observed, these results are consistent with the ranges reported by Barreto et al. (2024) for PLA filaments reinforced with rice husk (2–3.5 kJ/m²) and by Pemas et al. (2024) for PLA reinforced with tomato stems (≈3 kJ/m²). The slight improvement observed in formulation F5 suggests that a moderate addition of pecan shell contributes to absorbing impacts without weakening the matrix, validating the mechanical performance obtained.

Validation of Thermal Behaviour
Thermogravimetric analysis (TGA) showed initial degradation temperatures (Tonset) between 320 and 327 °C and maximum degradation temperatures (Tmax) between 351 and 357 °C, results that are consistent with the expected ranges for PLA biocomposites containing plant fibres (Trivedi et al. 2023; Xiao et al. 2025). This positive shift of approximately 6 °C with respect to pure PLA supports the hypothesis that the lignin and phenolic compounds present in pecan shell act as thermal stabilisers, enhancing the material's resistance to thermal degradation. This behaviour confirms the correct implementation of ISO 11358-1 and supports the thermal stability of the material for 3D printing applications.

Overall, the validation of the experimental results demonstrates that the values obtained fall within the accepted ranges for PLA-based biocomposite materials, confirming the reliability of the ASTM D638, ASTM D256, and ISO 11358-1 methods employed. Correspondence with the literature and the observed trends of greater rigidity, slight loss of ductility, and thermal improvement with moderate reinforcement contents confirm the robustness of the experimental procedure. Therefore, the validated mechanical and thermal indicators support the reproducibility of the process and strengthen the proposition of PLA–pecan shell filaments as a technically viable and environmentally sustainable alternative for additive manufacturing.

6. Conclusion
This study addressed the existing gap in research concerning the revaluation of Peruvian agro-industrial waste in additive manufacturing, demonstrating the technical feasibility of using pecan shell as a reinforcement in PLA filaments. Through the integrated application of QFD, Taguchi, SPC, and ANOVA methodologies, a reproducible experimental optimisation approach was validated, one that is transferable to the development of other polymeric biocomposites formulated with locally sourced agricultural residues.

The optimal processing configuration (150 °C and 11 rpm) and a reinforcement proportion of 5 % yielded filaments with superior properties compared to pure PLA, showing increases of 18.2 % in Stress Peak, 8.7 % in Young's Modulus, and 5.9 % in Impact Strength. The slight reduction in Strain Break confirmed the characteristic balance between stiffness and ductility associated with lignocellulosic composites. Likewise, thermal analysis revealed an improvement of approximately 5 °C in the maximum degradation temperature (Tmax), attributed to the stabilising effect of lignin present in the pecan shell, confirming an adequate balance between stiffness, thermal stability, and processability.

Overall, the findings validate the reproducibility and reliability of the developed biocomposite, consolidating its potential as a sustainable and low-cost alternative for additive manufacturing. It is recommended to scale up the process using twin-screw extrusion with prior pelletisation and to incorporate FTIR, SEM and LCA analyses to further the chemical and environmental characterisation of the material. This research also lays the foundations for the development of new sustainable biocomposites that drive innovation in 3D printing materials.

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Biographies

Gliden Jaroslav Dubois-Makarova student from the Faculty of Engineering at Universidad de Lima. He has experience in HSE and Operations.

Alejandro Antonio Lara-Ruiz student from the Faculty of Engineering at Universidad de Lima. He has experience in Sells and Marketing.

Juan Carlos Quiroz-Flores holds an MBA from Universidad ESAN. Industrial Engineer from Universidad de Lima. Ph.D. in Business Management from Universidad Nacional Mayor de San Marcos, Black Belt in Lean Six Sigma. He is currently an undergraduate professor and researcher at the University of Lima. Expert in Lean Supply Chain and Operations with more than 20 years of professional experience in the direction and management of operations, process improvement, and productivity; specialist in implementing Continuous Improvement Projects, PDCA, TOC, and Lean Six Sigma. Leader of the transformation, productivity, and change generation projects. Able to form high-performance teams aligned with the company's "Continuous Improvement" strategies and programs. He has published journal articles and conferences indexed in Scopus and Web of Science. His research interests include supply chain and logistics management, lean manufacturing, Lean Six Sigma, business process management, agribusiness, design work, facility layout design, systematic distribution planning, quality management, Industry 4.0, Digital Transformation, and Lean Manufacturing. He is a classified researcher by the National Council of Science, Technology, and Technological Innovation of Peru (CONCYTEC) and a member of IEOM, IISE, ASQ, IEEE, and CIP (College of Engineers of Peru).

Marcos Fernando Ruiz-Ruiz holds a PhD in Education Sciences from the Pontifical Catholic University of Peru, a master's degree and bachelor's degree in education from the University of Piura, as well as a degree in Industrial Engineering from the University of Lima. His research interests encompass future studies, structural analysis, and the foresight of engineering and education, with a particular emphasis on gender-focused approaches within STEM education. Doctor Ruiz-Ruiz also holds a Postgraduate Certificate (Expert Diploma) in Future Studies from UNED (Spain). He is a Renacyt researcher accredited by the National Council of Science and Technology of Peru (Concytec). Additionally, he has published several articles in prestigious journals and is a member of several professional organisations, including the Ibero-American Chapter of the World Futures Studies Federation (WFSF), the Peruvian Society for Educational Research (SIEP), the College of Teachers of Peru (CPP) and the College of Engineers of Peru (CIP-80033).