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Correlation study between the mechanical properties of Polylactic acid (PLA)-based biocomposites and the chemical composition of their plant fibers

N. Outaleb [1,2*], L. Laasri [1], M. Tahiri [2], Y. Naimi [1]
[1] *Department of chemistry, Faculty of Sciences Ben M'Sik, Hassan II University, Casablanca, Morocco*
[2] *Department of chemistry, Ain Chock's Sciences, Hassan II University, Casablanca, Morocco*
**Corresponding author, Email address: outaleb.naimavarenape@gmail.com*

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Abstract: Polylactic acid (PLA)-based composites have gained increasing interest in recent years, the use of such biodegradable polymer as matrix in the reinforcement of vegetables fibers enables to minimize the environmental impact of this material, which further encouraged its use in the current market. Previous studies have highlighted the importance of hybridizing the PLA polymer, generally brittle and not very resistant to shock with plant fibers, to improve the mechanical properties of the composite. Thus, knowledge of the effect of the chemical composition of each variety of fibers on the PLA, will allow us to orient the selection of the fiber according to the desired mechanical properties sought. The objective of this study is to determine to what extent the mechanical properties of the PLA-based bio-composites can be influenced by the chemical composition of plant fibers such as abaca, flax, jute, ramie, fiber coconut, hemp, and sisal. We also used the Pearson correlation test between these two continuous quantitative variables to check the existence of possible correlation.

Keywords: PLA-based composites; Plant fibers; Correlation study; Fibers chemical composition; Mechanical properties.

1. Introduction

Generally, composite materials reinforced by synthetic fibers are designed for high performance, which does not facilitate their recycling because the separation of the components they contain is quite difficult. Thus, the disposal of this composite is done by unsatisfactory means such as landfilling and incineration which has a considerable impact on the environment. This has prompted researchers to find new alternatives such as composites reinforced with natural fibers of plant origin called "Bio-composites" which are characterized by their low cost, low density, lightness, biodegradability and recyclability, and, therefore, they have a low environmental impact (Conroy et *al*., 2006; Mohanty et *al*., 2000; A. K. Bledzki & Gassan, 1999; John & Thomas, 2008;).

Biomass constitutes all the organic issue whether of vegetable or animal origin. Biomass resources are lots and varied (agriculture, forest, marine and aquatic environments, hedges, parks or gardens, industries and human activities…). The use of biodegradable polymer (PLA) as a matrix in the reinforcement of plant fibers has been the subject of great interest in materials science. Currently,

plant fibers have been widely used together with PLA as fiber reinforced biocomposites to increase the biodegradation rate of PLA, this type of reinforcement enables to facility access for micro-organisms into material structure. We cite as an example: abaca, bamboo, cocoa, coconut, flax, hemp, jute and kenaf, ramie, and sisal. The use of reinforcing materials is principally responsible for the strength and hardness of composites (Rytlewski et *al*., 2022; Naimi et *al*., 2017; A K Bledzki & Jaszkiewicz, 2010; Long et *al*., 2019; Takahashi & Bettini, 2019; Young et *al*., 2012; Ejaz et *al*., 2020; Mazzanti et *al*., 2019; Yan et *al*., 2020; Hassan, 2010; Yang et *al*., 2021; Lagazzo et *al*., 2019; Zhu et *al*., 2020; Ezeh & Onukwuli, 2021).

One of the possible solutions to increase the biodegradation rate of PLA is mixing it with natural lignin/cellulose materials such as wood flour or plant fibers. This type of filler enables easier access for micro-organisms to penetrate into all volume instead of only the outer (surface layer) material structure.

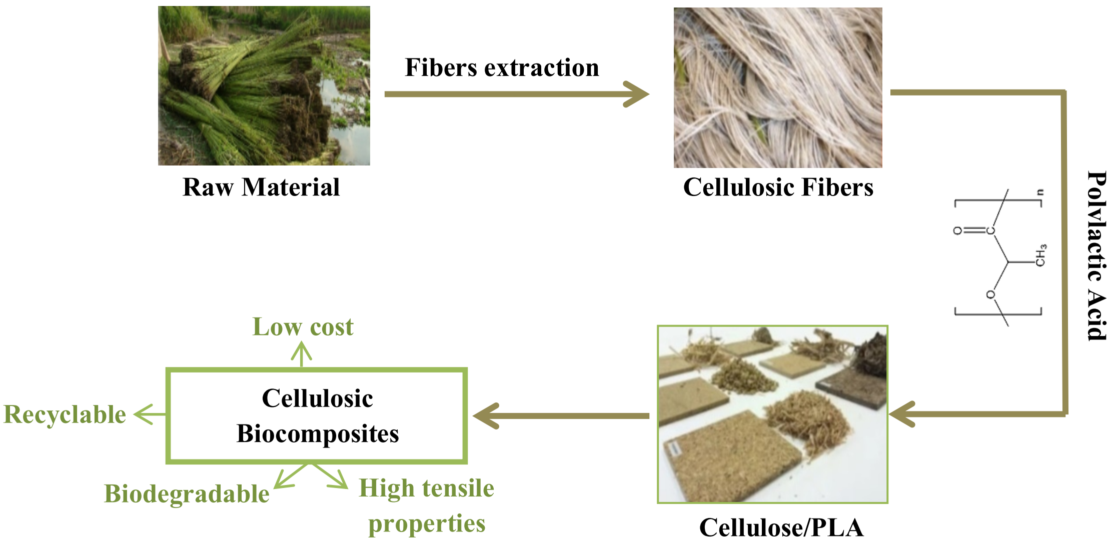

Figure 1. Diagram of PLA-based biocomposites development

The synthetic representation for the development of cellulosic fibers reinforced biocomposites is shown in Figure 1. The reinforcement is extracted from the stalk, seed, leaf, bast, reeds/grass or fruit fibers of the plant. These used fibers are obtained from a mechanical extraction from plant fibers followed by a chemical treatment. The fibers are mixed with the polymer matrix to finally give a desired shape to the product. Biomass procures immense significance as chemical raw material because consists of cellulose fibers which are high crystalloid, insoluble in water and common organic solvent, hemicelluloses and lignin. Cellulose fibers reinforced PLA-based biocomposites were developed using extrusion where the fillers are incorporated into the molten thermoplastic using a screw system and injection molding process that consists in softening the plastic material to make it melt and injecting it in a mold to shape it. Both methods are green because they don't use aggressive solvents or chemicals products in the process, which makes them more interesting on an industrial scale. So plant fibers have used with PLA as fiber reinforced biocomposites due to their high tensile strength, low cost, biodegradability and recyclability) (Barkany et *al*., 2017; Ezahra et *al*., 2019; Bouzidi et *al*., 2018; Essabir et *al*., 2017).

Therefore, this paper will focus on studying the behavior of the vegetable fiber after its combination with the PLA and compare the performance of each component such as cellulose, hemicelluloses, lignin, pectin, ash and waxes on the mechanical properties of polylactic acid (PLA)-

based composite. Also, we evaluate what limit the mechanical properties of the based-PLA composite can influenced by the chemical composition of the plant fibers.

2. Methodology

A descriptive-correlation study was conducted on eight PLA-based biocomposite samples with 30% fiber loading. The selected fibers are: abaca, coir, flax, hemp, jute, kenaf, ramie and sisal. The chemical compositions of these fibers as well as the mechanical properties of the composite materials produced using these fibers are determined by previous researchers.

Data were analyzed by SPSS software version 26 using descriptive statistics, independent test, ANOVA, Pearson's correlation coefficient, and bivariate linear regression. In analyzing the data, a P-value of 0.05 % considered the critical significance level, provided by SPSS software.

In Tables 1 and 2, we respectively report the chemical composition of plant fibers used in this study and the mechanical properties of the corresponding polylactic acid (PLA)-based composites. The chemical composition of fibers is not always the same within the same species, because there are slight differences due to the nature of soil, age, and climatic conditions. According to the values mentioned in Table 1, the fibers consist mainly of cellulose fibrils which are held together by a matrix composed mainly of lignin and hemicellulose and lesser amounts of extractable materials, proteins, starch, and inorganic materials (Bouzidi et *al*., 2018; Essabir et *al*., 2017; Faruk et *al*., 2012; Güven et *al*., 2016) .

Table 1. Chemical composition of plant fibers

Vegetable fibers	Chemical composition (wt %)						References
	Cellulose	Hemicellulose	Lignin	Pectin	Ash	Waxes	
Abacca	56-63	20-25	7-9	-	3	3	(Asim et *al*., 2015) (Thakur et *al*., 2014) (Fuqua et *al*., 2016)
Coir	32-43	0.15-0.25	40-45	-	2.7-10.2	-	(Faruk et al., 2012) (Asim et *al*., 2015) (Gurunathan et *al*., 2015)
Flax	71	18.6-20.6	2.2	2.3	-	-	(Gowda et *al*., 2019)
Hemp	68	15	10	1	0.8	0.8	(Gowda et *al*., 2019)
Jute	61-71	14-25.56	11.10-13	-	0.67-0.8	0.5-3.89	(Gowda et *al*., 2019) (Wang et *al*., 2019)
Kenaf	45-57	21.5	8-13	3-5	2.5	-	(Thakur et *al*., 2014)
Ramie	68.6-76.2	13-16	0.6-0.7	1.9	-	0.3	(Thakur et *al*., 2014)
Sisal	65-78	10-14.2	8-11	10	0.6-1	2	(Gowda et *al*., 2019) (Kim & Netravali, 2010)

Additionally, we noticed that the tensile strength of PLA biocomposites reinforced with abaca, flax, and ramie is higher than that of other plant fibers, and the coir/PLA has the lowest value, knowing that the fibers of coir contain the lowest percentage of cellulose and hemicellulose with a large percentage of lignin. In addition, the young modulus of Abaca is higher than of other plant

fibers, but the impact strength of the latter is lower than of other fibers such as coir, flax, jute and ramie so plant fibers have high tensile properties and low impact properties.

Table 1. Mechanical properties of plant fibers reinforced polymer composite

Plant fibers/PLA	Fiber loading (wt %)	Tensile strength (MPa)	Young's modulus (GPa)	Flexural strength (MPa)	Impact strength (Kj/m^2)	References
Abacca/PLA	30	74	5.85	124	5.3	(Bajpai et *al*., 2012)
Coir/PLA		16	0.8	25	-	(Dong et *al*., 2014)
Flax/PLA		54.15	6.31	-	11.13	(Sawpan et *al*., 2012)
Hemp/PLA		-	-	87.1±5.1	-	(Sawpan et *al*., 2012)
Jute/PLA		49	-	101	8.5	(Bajpai et *al*., 2012)
Kenaf/PLA		32	4.5	-	-	(E. Hassan, 2012)
Ramie/PLA		52.5	-	170	10	(Siakeng et *al*., 2018)
Sisal/PLA		35	2	110	7	(Liang et *al*., 2021)

Figure 2 shows the chemical structure of the main components of vegetable fibers: cellulose, hemicelluloses and lignin.

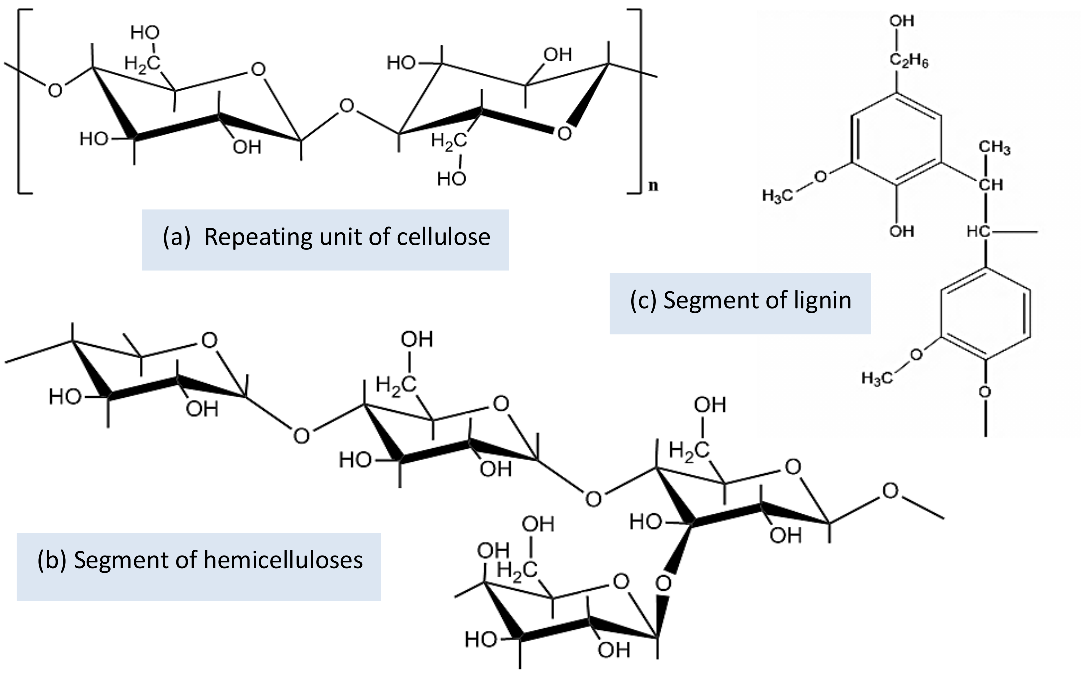

Figure 2. Chemical structure of the main components of vegetable fibers: (a) repeating unit of cellulose, (b) segment of hemicelluloses, and (c) a segment of lignin (Bouzidi et *al*., 2018) .

1.1. Statistical analysis

We investigated the relationship between the chemical composition of plant fibers, and the mechanical properties of the corresponding plant fibers reinforced PLA. Both variables were included in the simple linear regression model. Statistical analysis was carried out to verify a possible correlation between the chemical composition and the mechanical properties of PLA reinforced with vegetable fibers. The examination of the rank correlation between two variables is carried out using the Pearson correlation test. The first variable noted I, corresponds in our calculations to the mechanical properties of natural fibers of vegetable origin, such as tensile strength, young's modulus, flexural strength and impact strength. The second variable noted II, corresponds to the chemical composition of vegetable fibers such as cellulose, hemicelluloses, lignin, ash, pectin and waxes. From the use of mean values, we can find the rank correlation between the two variables (I and II) since the values used in the tables are scattered in an interval.

2. Results

Tables 3, 4, 5, and 6 present the correlation coefficient R, the number of variables, the regression coefficient R2, and the linear regression equation of each plant fibers component as well as the mechanical properties of the corresponding PLA-based composites. The correlation coefficient (R=95%) indicates a perfect correlation between two variables I and II. The graphs of regressions of the measured mechanical properties (tensile strength, young's modulus, flexural strength, and impact strength) as function of the percentage of the chemical composition (cellulose, hemicellulose, pectin, ash and waxes) are shown in Figures 3, 4, 5, 6, 7, and 8.

In Figure 3, we report the influence of the percentage of cellulose on the mechanical properties of PLA-base composites. Similar shapes are almost observed for the correlation curves between the cellulose content and tensile strength and flexural strength. These two properties reach their maximum values of 74 and 170 MPa for cellulose contents of at 59.5 and 72.4wt% of respectively. Beyond these values, the properties decrease drastically. Young's modulus, and impact strength, also varies similarly. However, it appears that the Young's modulus decreases with increasing cellulose content and this correlation can be considered linear. This result is consistent with the relatively large regression coefficient of 0.618 and low P-value (Table 3).

Table 3. Correlation between the percentage of cellulose and the mechanical properties

Mechanical properties	Correlation coefficient	Number of variables	P-value	Regression R^2
Tensile strength	-0.204	7	0.661	0.042
Young's modulus	-0.786	5	0.115	0.618
Flexural strength	-0.064	6	0.904	0.004
Impact strength	-0.488	5	0.404	0.238

In Figure 4, we report the influence of the percentage of hemicellulose on the mechanical properties of PLA-based composites. The calculated P-value, shown in table 4, indiquate that there is no relationship between hemicelluloses and mechanical properties

Table 4. Correlation between the percentage of hemicellulose and the mechanical properties

Mechanical properties	Correlation coefficient	Number of variables	P-value	Regression R^2
Tensile strength	-0.105	7	0.823	0.011
Young's modulus	-0.294	5	0.631	0.086
Flexural strength	-0.249	6	0.634	0.062
Impact strength	-0.750	5	0.144	0.562

In Figure 5, we report the influence of the lignin percent on the mechanical properties of PLA-base composites. According to the obtained results, we can state that the evolution of the cellulose contents does not have any influence on the aforementioned mechanical properties of the composites. The calculated P-value presented in table 5 confirms the above findings.

Table 5. Correlation between the lignin percent and the mechanical properties

Mechanical properties	Correlation coefficient	Number of variables	P-value	Regression R^2
Tensile strength	0.105	7	0.822	0.011
Young's modulus	0.495	5	0.397	0.245
Flexural strength	-0.040	6	0.940	0.002
Impact strength	-0.658	5	0.227	0.433

In Figure 6, we report the influence of the pectin percent on the mechanical properties of PLA-base composites. Generally, Young's modulus, tensile strength, and impact strength decrease similarly with increasing pectin content, unlike flexural strength which evolves randomly. Although the regression coefficient R^2 equals 1 for Young's modulus (Table 6), we cannot confirm the linearity of this correlation because we only relied on two values. It is insufficient to draw such a conclusion.

Table 6. Correlation between the pectin percent and the mechanical properties

Mechanical properties	Correlation coefficient	Number of variables	P-value	Regression R^2
Tensile strength	-0.707	4	0.293	0.500
Young's modulus	-1.000	2	0.000	1.000
Flexural strength	-0.243	4	0.757	0.059
Impact strength	-0.925	3	0.248	0.856

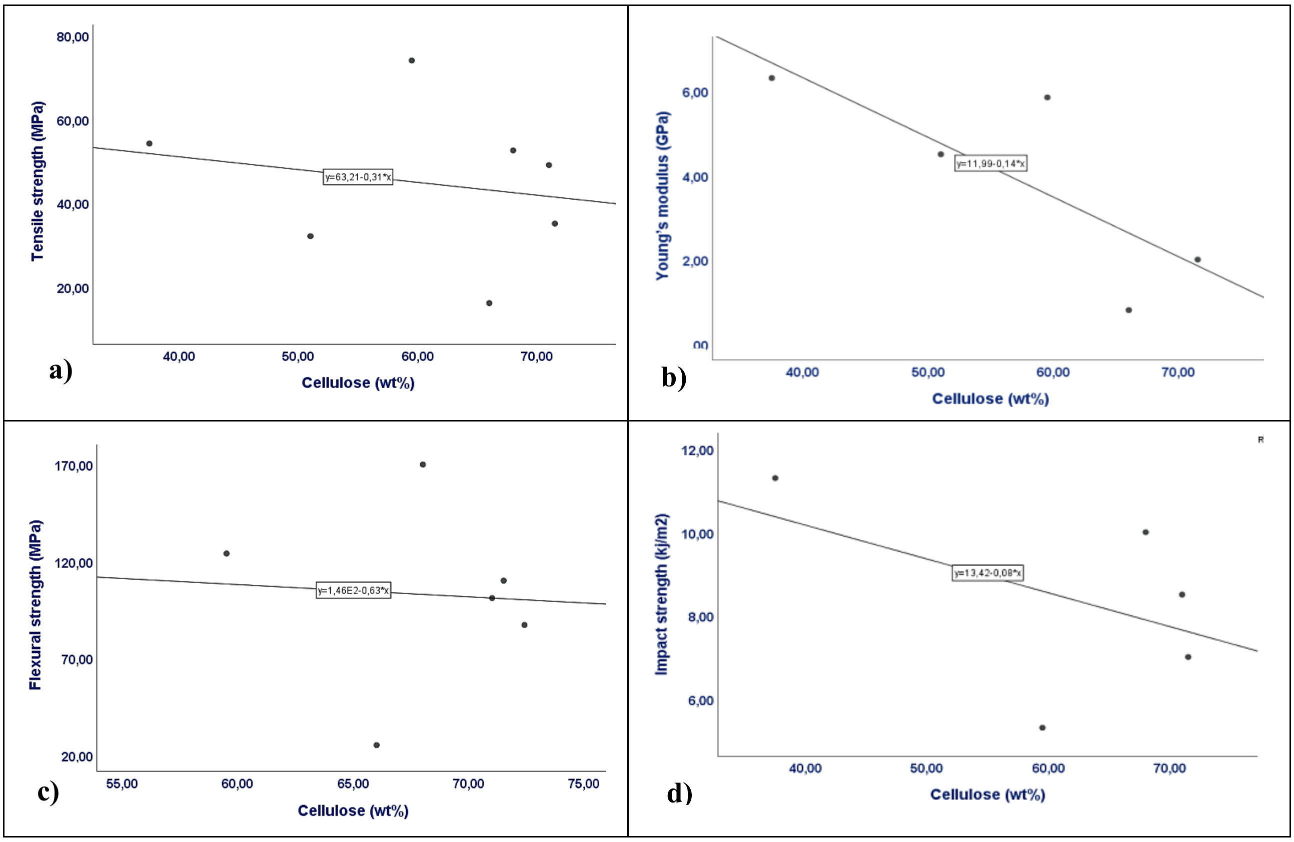

Figure 3. Evolution of the mechanical properties versus the percentage of cellulose: a) Tensile strength, b) Young's modulus, c) Flexural strength, and d) Impact strength.

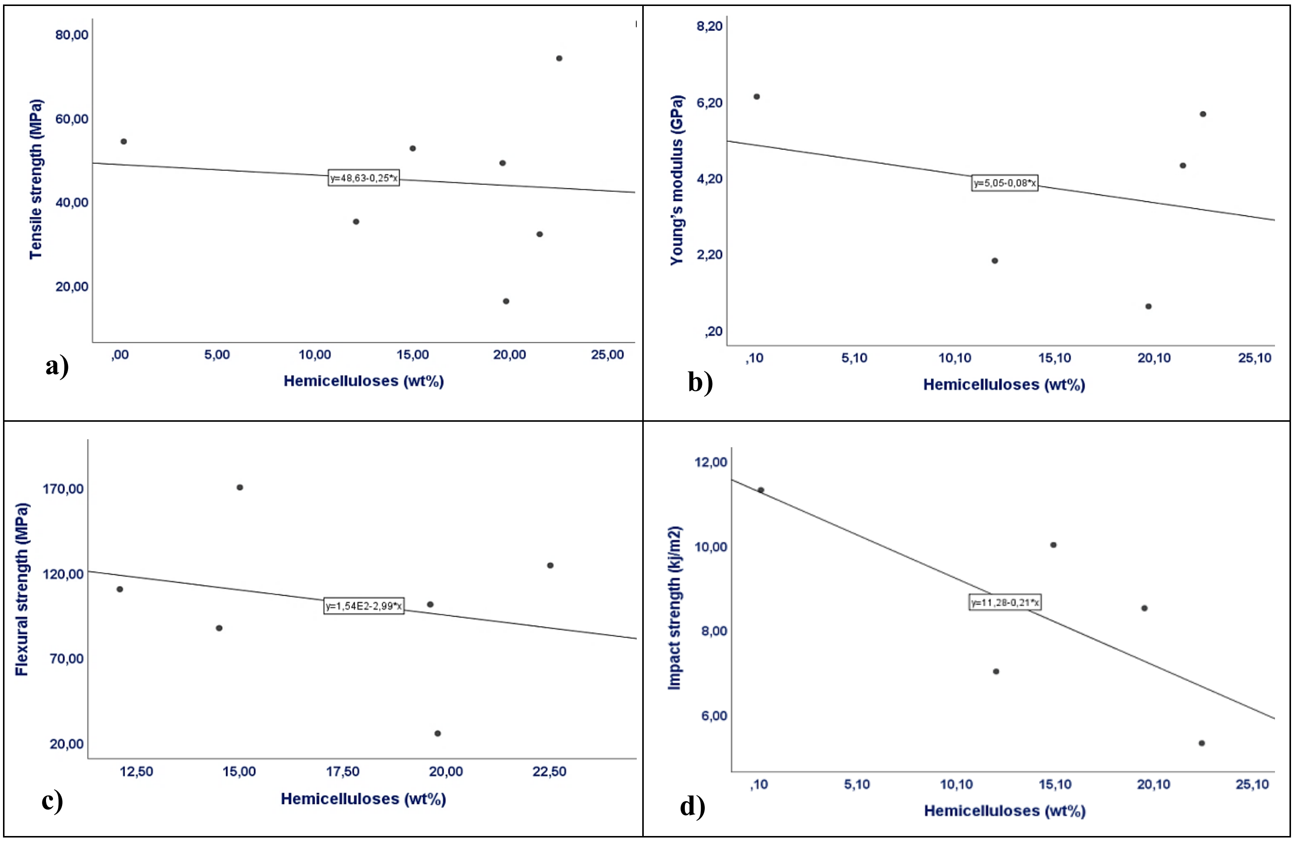

Figure 4. Evolution of the mechanical properties versus the percentage of hemicelluloses: a) Tensile strength, b) Young's modulus, c) Flexural strength and d) Impact strength.

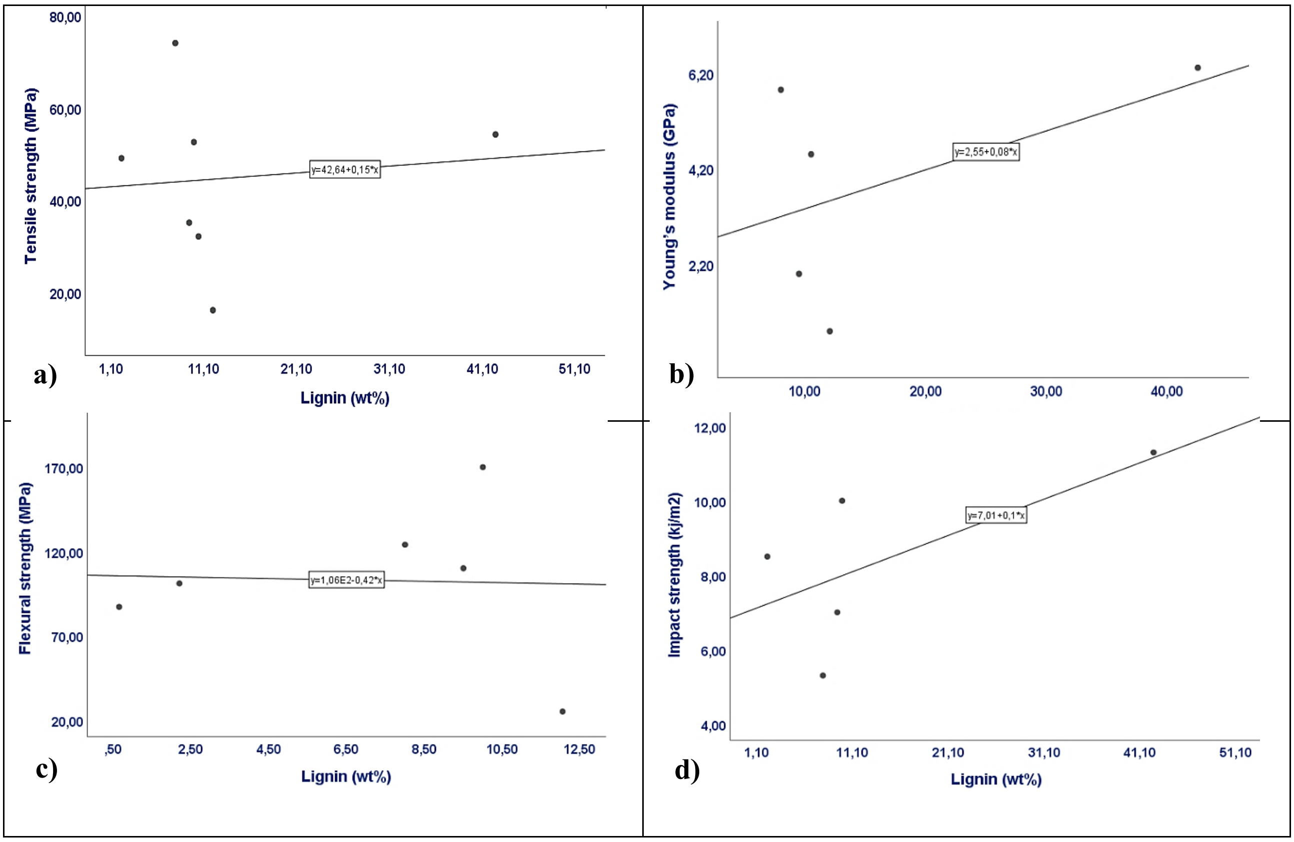

Figure 5. Evolution of the mechanical properties versus the percentage of lignin: a) Tensile strength, b) Young's modulus, c) Flexural strength and d) Impact strength.

In Figure 7, we report the influence of ash content on the mechanical properties of PLA-base composites. Young's modulus and impact strength also vary similarly. The calculated P-value (table 7) states that there is no relationship between ash content and mechanical properties, except for Young's modulus, for which the correlation is insignificant. Young's modulus increases with the ash content and this correlation can be considered linear.

Table 7. Correlation between ash content and mechanical properties

Mechanical properties	Correlation coefficient	Number of variables	P-value	Regression R^2
Tensile strength	0.464	6	0.354	0.215
Young's modulus	0.856	5	0.064	0.734
Flexural strength	0.207	4	0.793	0.043
Impact strength	0.436	4	0.564	0.190

In Figure 8, we report the influence of the waxes on the mechanical properties of PLA-base composites. The p-values presented in Table 8 show that there is no significant correlation between waxes, and tensile strength, young's modulus and flexural strength. On the other hand, we recorded a regression coefficient of 0.989 for the impact strength which shows a linear regression.

Table 8. Correlation between waxes and mechanical properties

Mechanical properties	Correlation coefficient	Number of variables	P-value	Regression R^2
Tensile strength	0.210	4	0.790	0.044
Young's modulus	0.913	3	0.267	0.834
Flexural strength	-0.204	5	0.742	0.042
Impact strength	-0.994	3	0.067	0.989

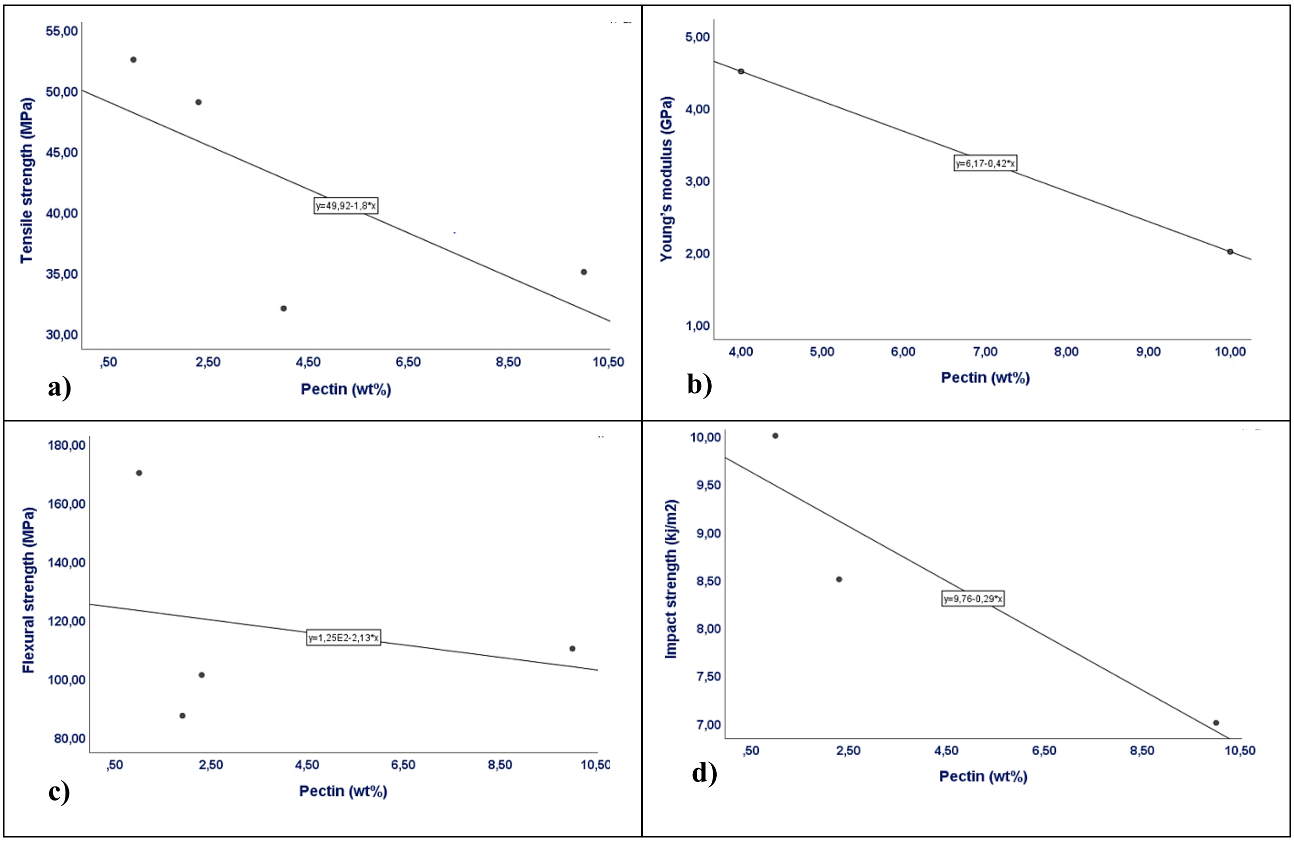

Figure 6. Evolution of the mechanical properties versus the percent of pectin: a) Tensile strength, b) Young's modulus, c) Flexural strength and d) Impact strength.

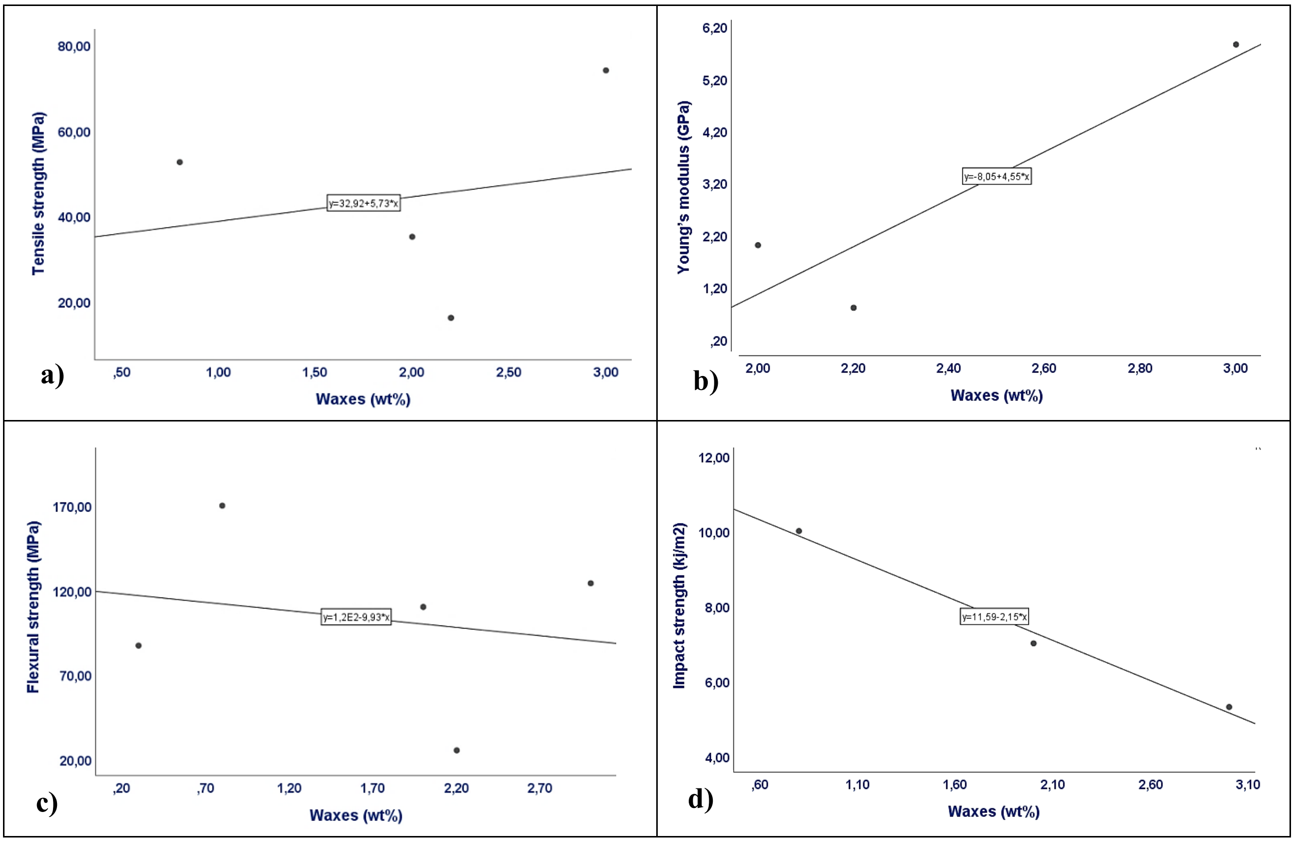

Figure 8. Evolution of the mechanical properties versus the percentage of waxes: a) Tensile strength, b) Young's modulus, c) Flexural strength and d) Impact strength.

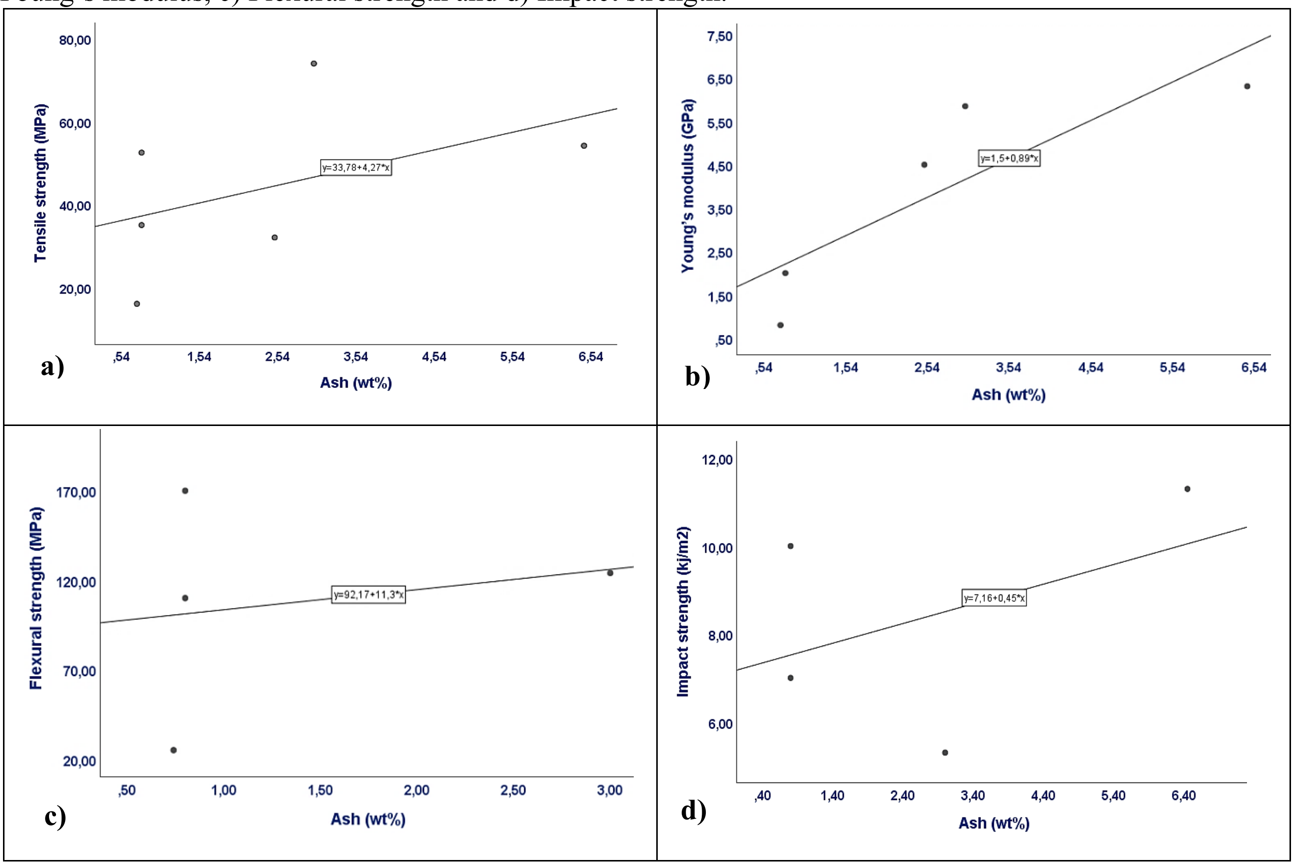

Figure 7. Evolution of the mechanical properties versus the percentage of ash: a) Tensile strength, b) Young's modulus, c) Flexural strength and d) Impact strength.

Conclusion

In conclusion, the choice of plant fiber is conditioned by the desired mechanical properties of the PLA-based composites in question. Remember that cellulose, hemicellulose, and lignin are the most presented with high proportion in the plant fibers. Although pectin, ash and waxes are present a low proportion so their influence is also weak. The results of the preliminary statistical study carried out in this work show that young's modulus varies inversely with the cellulose and ash contents. The content of hemicellulose, lignin and pectin does not significantly influence the mechanical properties of PLA-based composites. Also it appears that the Impact strength property varies inversely with the wax content.

Disclosure statement: *Conflict of Interest:* The authors declare that there are no conflicts of interest.
Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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