SPECIAL SYMPOSIA | BIOPOLYMERS, BIOCOMPOSITES AND BIOPROCESSING

Transforming Agriculture: Advancements in Compost-Biopolymers Composites for Enhanced Sustainability

Yina-Ortega and Felipe-Salcedo

DOI: https://doi.org/10.51573/Andes.PPS.SS.BBB.11

December 2024

View Online

Export Citation

Transforming Agriculture: Advancements in Compost-Biopolymers Composites for Enhanced Sustainability

Yina-Ortega and Felipe-Salcedo[1]

Abstract : In the Cesar Department in Colombia, sustainable agriculture faces critical challenges despite its thriving livestock sector. Extensive cattle ranching and poor soil management worsen soil conditions. Additionally, significant environmental concerns arise from the substantial discharge of wastewater in the dairy processing industry. This study aims to valorize biosolids derived from wastewater during dairy plant disinfection to enhance sustainable livestock production. It explores the use of biosolids as the primary matrix for creating composted compounds and biopolymers for agricultural purposes. These composite materials utilize composted biosolids from the dairy industry as the main matrix (60%), blended with biodegradable polymers such as polybutylene adipate succinate (PBSA) and polylactic acid (PLA) as functional agents. This innovative approach contrasts with the prevailing literature on composite polymer materials, where polymers typically form the matrix and agricultural residues serve as fillers. Biosolids from Freskaleche S.A. undergo controlled thermal treatment and composting before being combined with PBSA and PLA polymers using an internal mixer. The development of these new compounds includes the evaluation of thermal stability, Fourier-transform infrared spectroscopy (FTIR), and mechanical properties. It is noteworthy that Compost/PBSA composites exhibit promising compatibility and thermomechanical properties resembling pure PBSA, suggesting their potential as thermoplastic materials for producing plastic-based agricultural products. Regarding Compost/PLA blends, compost enhances chemical reactions in PLA polymeric chains, significantly altering their thermomechanical properties. This research provides valuable insights into the use of biosolids for manufacturing composite materials, offering a sustainable approach to address agricultural challenges in the Cesar region.

Keywords: Composite materials, Dairy industry, Polymers, Soil erosion, Waste treatment

---

[1] Yina Ortega (yp.ortega@uniandes.edu.co) is affiliated with the Department of Agroindustrial Sciences at the Universidad Popular in Colombia. Felipe Salcedo is affiliated with the Department of Chemical and Food Engineering at the Universidad de los Andes in Colombia.

DOI: https://doi.org/10.51573/Andes.PPS.SS.BBB.11

Introduction

Livestock farming in the department of Cesar, Colombia, is distinguished by the generation of wastewater, known as "biosolids," with a high load of organic contaminants, representing approximately 90% of processed milk, depending on the type of plant [1]. This wastewater mainly originates from the cleaning of equipment, machines, and production plants containing residues of dairy and chemical products such as acids, alkalis, detergents, and disinfectants.

Biosolids, composed primarily of water (> 90%) and organic matter (around 30% on a dry basis) [2], have been studied globally for over half a century. In developed countries, these materials have been regulated for over a decade for use in various activities, both agricultural and non-agricultural, including soil recovery, landfill cover, and utilization in the forestry industry [3]. Therefore, the dairy industry must focus on managing these wastewater streams, both due to their volume and organic content.

The excessive use of non-degradable plastics poses another significant challenge to agricultural sustainability, as these materials are widely used in greenhouses, irrigation systems, and packaging due to their durability and environmental resistance. Although bioplastics offer the advantage of biodegradability, their adoption in agricultural applications has been limited compared to conventional plastics, which remain dominant due to their lower cost and established performance [4]. The research focuses on utilizing biosolids as a rich source of essential nutrients for crop development, leveraging their high biodegradation capacity due to significant organic content.

This makes them ideal for compost production and agricultural application; however, it is crucial to extend their beneficial effects on soil and plants. Thus, integrating biodegradable and compostable biopolymers such as polybutylene succinate-co-adipate (PBSA) and polylactic acid (PLA), known for their biodegradability, strength, and flexibility, into compost-polymers composites derived from composted dairy biosolids could offer a promising pathway to explore potential agricultural applications that enhance soil and crop improvement over an extended period [5].

Materials and Methods

Materials

The biosolids from Freskaleche S.A.'s wastewater formulation plant, constituting 18% of the compost, underwent thermal treatment at 200°C for 90 minutes. Subsequently, its were composted with other organic components: black soil (54%), rice husks (7%), cocoa husks (13%), orange peel (3.5%), and Efficient Microorganisms (4.5%) to improve the compost. For the composite material, Poly butylene succinate adipate (PBSA) pellets from Mitsubishi Chemical Performance Polymers, USA, and Luminy® LX175, a transparent, amorphous, low-flow, high-viscosity polylactic acid (PLA) (96% L-Isomer) from Total Corbion PLA, were used.

Blend and samples preparation

The production process of the composite material began with drying composted biosolids at 65°C for 3 hours, followed by grinding to 0.25 mm using a blade mill. Polymer and compost homogenization was made by a C.W. Brabender 2553 internal mixer, where PBSA and PLA polymers were first incorporated, followed by the compost after one minute of mixer operation. The mixtures, as detailed in Table 1, were processed at 40 rpm and 120°C for PBSA, and 170°C for PLA, until the torque stabilized, typically taking about 10 minutes.

Characterization of blends

Thermal analysis (DSC and TGA):

Thermal analysis utilized a TA Instruments SDT-Q600 Thermogravimetric Analyzer in a nitrogen atmosphere (100 ml/min). Samples (~10 mg) were heated at 10°C/min to 500°C, measuring residual weight, moisture loss, and the DTG peak.

Differential Scanning Calorimetry (DSC) with an SDT-Q2000 analyzer assessed thermal properties: Glass Transition Temperature (Tg) during heating (25°C to 250°C at 10°C/min), Crystallization Temperature (Tc) during cooling (with a 1-minute hold at 250°C) and Melting Temperature ™ from the second endothermic peak. Enthalpy of Crystallization (ΔHc) was calculated based on the area under the crystallization peak.

Comparing ΔHc values between biocomposites and pure polymers provides insights into their relative crystallinity, offering a detailed assessment of how the crystalline characteristics of biocomposites compare to those of pure polymers.

$$\text{Equation} = \text{Ci (\%)} = \frac{\mathbf{\Delta Hm - \Delta Hc}}{\mathbf{\Delta H^{0}} * \boldsymbol{f}} * 100\% \tag*{1)}$$

$$\text{Equation} = \quad \text{Ci (\%)} = \frac{\mathbf{\Delta Hm}}{\mathbf{\Delta H^{0}} * \boldsymbol{f}} * 100\% \tag*{2)}$$

The crystallinity index (Ci) of the films was determined by DSC, according to equation 1 for PLA, and equation 2 for PBS, where ΔH^0 is 135.0 J/g for PBSA [6] and 93,0 J/g for PLA [7].

Fourier Transform Infrared Spectroscopy (FTIR):

FTIR-ATR was used to study the bonds and interactions between the components of the prepared composites. The FTIR spectra of the composite materials were collected with a resolution of 4 cm-1 using a Nicolet™ iS50 spectrometer in the range of 400-4000 cm-1. Three measurements were taken for each specimen, and the average spectrum is shown.

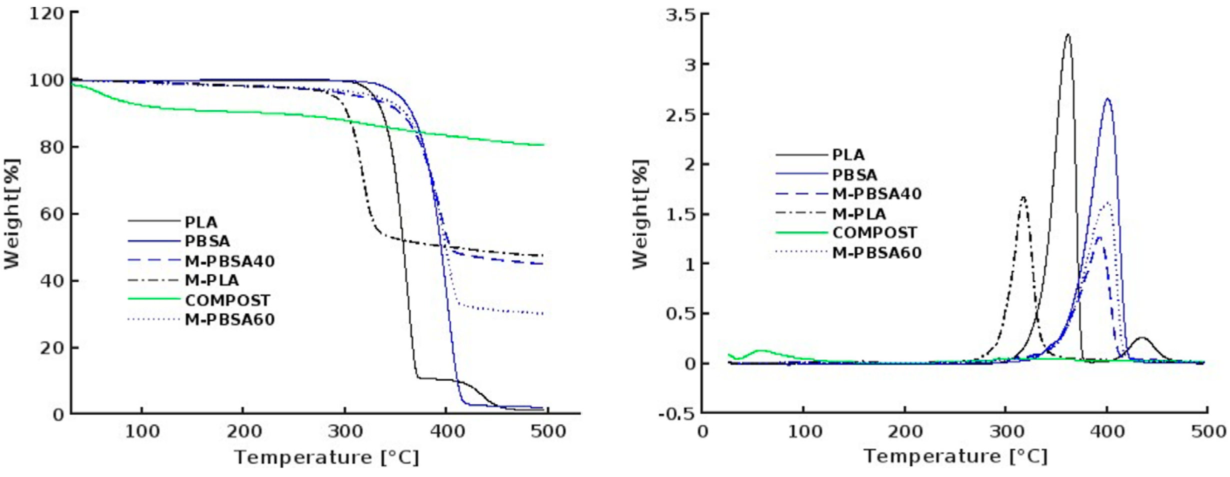
Analysis of Mechanical Properties:

Mechanical properties (tensile strength, percentage elongation at break, and Young's modulus) were evaluated using an INSTRON 3367 universal testing machine per ASTM D638, with results averaged from ten specimens.

Results and Discussion

Thermal analysis (DSC and TGA)

The thermal stability of all prepared samples was evaluated by thermogravimetric analysis. Figures 1(a) and (b) show the TG and DTG curves of the pure polymers and composites produced under the different compost ratios.



(a) (b)

Figure 1. Thermograms of the proposed mixtures. (a) Temperature and weight % Thermogram; (b) Weight % Derivative /°C Thermogram.

DOI: https://doi.org/10.51573/Andes.PPS.SS.BBB.11

When examining the thermograms in Figure 1, it is evident that the thermal degradation temperatures for pure PBSA and the blends with 60% and 40% compost do not differ significantly. For example, pure PBSA has a degradation temperature of 400°C, while the PBS biocomposite blends with 40% and 60% compost show degradation temperatures of 391°C and 398°C, respectively.

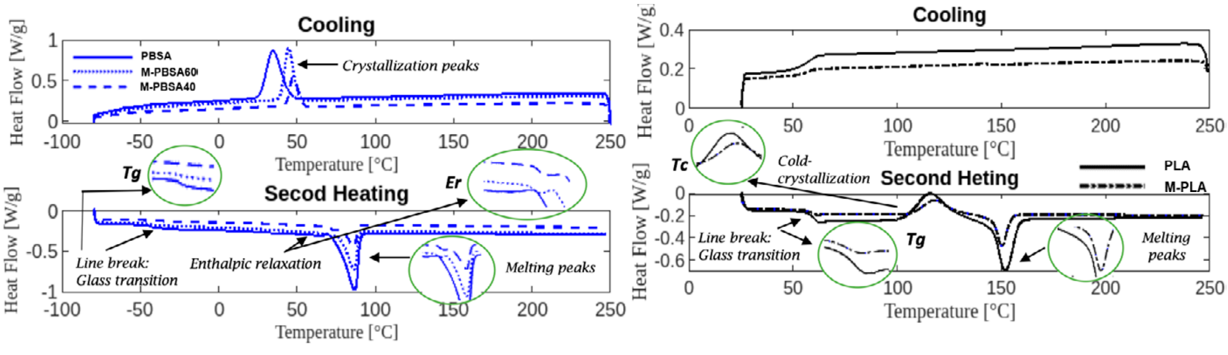
Table 1. TGA and DSC thermal properties of PBSA , PLA and Biocomposites.

Formulation			TGA Parameters		DSC Parameters					
	% Compost	% Polymers	T0 (°C)	Tmax (°C)	Tg(°C)	Tc(°C)	ΔHcc (Jg-1)	Tm(°C)	ΔHm(Jg-1)	Ci (%)
PLA	0	100	329	361	55,4	104,8 (Tcc)*	18,69	145,6	21,41	2,7
M-PLA	60	40	354	400	55,2	107,4	9,139	145,7	9,145	0,8
PBSA	0	100	331	398	-43,7	44,3	41,16	52,3a – 75,3b	36,7	27,2
M-PBSA60	40	60	316	391	-45	50,4	26,77	61,9a–78,5b	19,1	23,6
M-PBSA40	60	40	290	316	-46,4	53,3	14,26	70,9a – 79,7b	8,496	15,7
COMPOST	100	0	67	58	——-	————	————	——-	——-	

* Tcc = Cold cristalization

The decrease in degradation temperature is closely related to the presence of cellulose and lignin in the compost. At temperatures above 350°C, cellulose undergoes thermal decomposition due to the random cleavage of the main chains of 1,4-β-d-glucopyranose. Furthermore, at these high temperatures, the cleavage of C-O and C-C bonds in cellulose leads to the generation of volatile compounds such as CO and CH4, as observed in previous studies incorporating pineapple and yucca flour fillers [8].

On the other hand, when exposed to high temperatures, PLA exhibits a notably different thermal response. In its thermogram (Figure 1[a]), two distinct decomposition zones are identified, linked to various reactions occurring during PLA's thermal degradation. Typically, PLA degrades in a single step around 350 °C [9,10]. However, in some cases, two degradation stages have been reported, at 300 °C and 340-360 °C [4], as presented in this study. This difference could be explained by the complexity of PLA's thermal degradation or its chemical structure in terms of polymer stereoisomerism. The stereoisomerism of PLA, influenced by the presence of L or Disomers, can impact the polymer's thermal properties [11].



(a)

(b)

Figure 2. Thermal transitions observed for formulations. (a) Thermogram of PBSA, M-PBSA60 and M-PBSA40; (b) Thermogram of PLA and M-PLA.

Differential scanning calorimetry (DSC) identified the melting temperatures (Tm) and crystallization temperatures (Tc) and calculated the melting enthalpies (ΔHm) and crystallization enthalpies (ΔHc), as well as the crystallinity index (CI) of the samples (Table 1, Figure 2[a] and [b]). The addition of 60% compost does not significantly alter the Tg of pure PLA or its biocomposites, which remains around 55°C, nor does it affect the Tm values, maintaining them at 145°C [12], as reported by Cacciotti et al. in their study on PLA with functional diatomite loaded with coffee extracts [13].

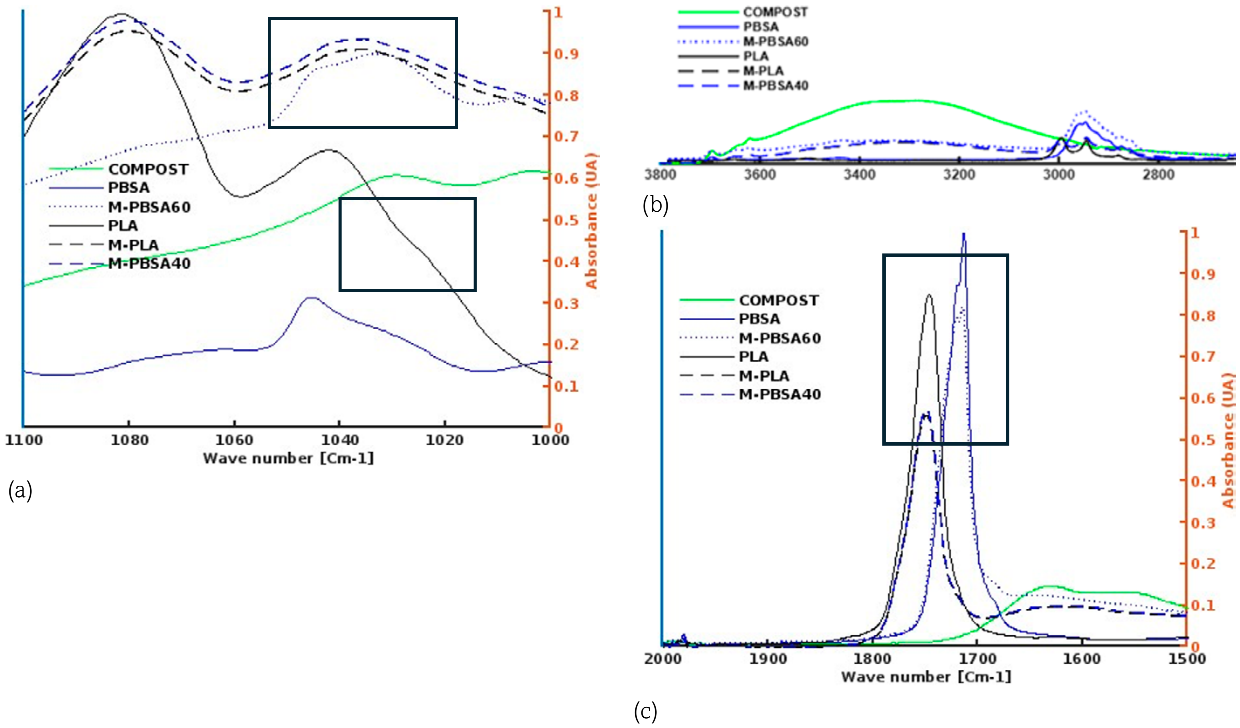
Similarly, the Tg of PBSA and its biocomposites remains close to -45°C, with the Tm also remaining constant in the range of 75°C to 78°C, consistent with previous studies that show no significant differences with additions of up to 20% cellulose [14].

For PBSA, the exothermic peak associated with crystallinity is found at 44.3°C, shifting to 50.4°C and 53.3°C in M-PBS60 and M-PBS40, respectively, indicating that compost acts as a nucleating agent [15]. Cellulose in the compost promotes crystal formation at higher temperatures [16,17]. In the formulation with 40% compost (M-PBSA40), the crystallinity index (CI) is barely affected, with a reduction of approximately 3%. However, with compost content increased to 60% (M-PBSA60), the CI decreases by around 12%, possibly due to homogeneous dispersion hindering lamellar crystallization compared to the pure polymer. Additionally, both in PBSA and its biocomposites, a multiple melting behavior (identified as Tma in Table 1) associated with partial melting, recrystallization, and re-melting phenomena is observed, characteristic of PBSA as reported in previous studies [18].

The thermogram of PLA shows a sharp endothermic peak just before the glass transition, due to enthalpy relaxation from the polymer's physical aging [19]. This peak is more pronounced in pure PLA than in the M-PLA composite, likely because the addition of compost results in lower molecular weight products with higher mobility. The cold crystallization process of PLA has a peak at 104.8°C. Adding 60% compost raises the Tcc to 107.4°C, possibly due to stereocomplexation. However, a decrease in the material's crystallinity index is observed, similar to that seen in PBSA, due to the difficulty in forming crystallites [12,20].

Fourier Transform Infrared Spectroscopy (FTIR):

The FTIR spectra for the biopolymer's PLA and PBSA, as well as for the biocomposites M-PLA, M-PBSA60, M-PBSA40, and Compost are shown in Figure 3.



(a)

(b)

(c)

Figure 3. FTIR Analysis for formulations. (a) Hydroxyl groups region (2800- 3800 cm-1); Cellulose region (1020–1050 cm-1); (c) Carbonyl group region (1600–1700 cm-1).

The spectral signals in Figure 3(a), identified in the range of 3600 to 3000 cm 1, indicated the presence of numerous hydroxyl (OH) groups and methyl groups in the lignocellulose section of the compost. This observation is confirmed by the strong absorption peak in the range of 1020 to 1050 cm−1 in the spectra of both the compost and the composites (Figure 3[b]), related to cellulose. These findings are consistent with previous studies on the effect of coffee husk in the production of biocomposites.

On the other hand, the region corresponding to the carbonyl group (-C=O) in the 1700 cm−1 range presents several overlapping peaks (Figure 3[c]), a characteristic behavior of semicrystalline polyesters, such as PLA and PBS. This behavior is equally evident in the biocomposites M-PLA, M-PBS40, and M-PBS60, despite the high percentage of compost added to the formulation. This phenomenon is attributed to the stretching of the ester carbonyl groups in both amorphous and crystalline regions. The confirmed presence of cellulose in the samples explains the decrease in the thermal stability of the composites provided by it [15-21].

Analysis of Mechanical Properties:

When comparing the results between the pure polymers and their derived blends, Figure 4 is obtained.

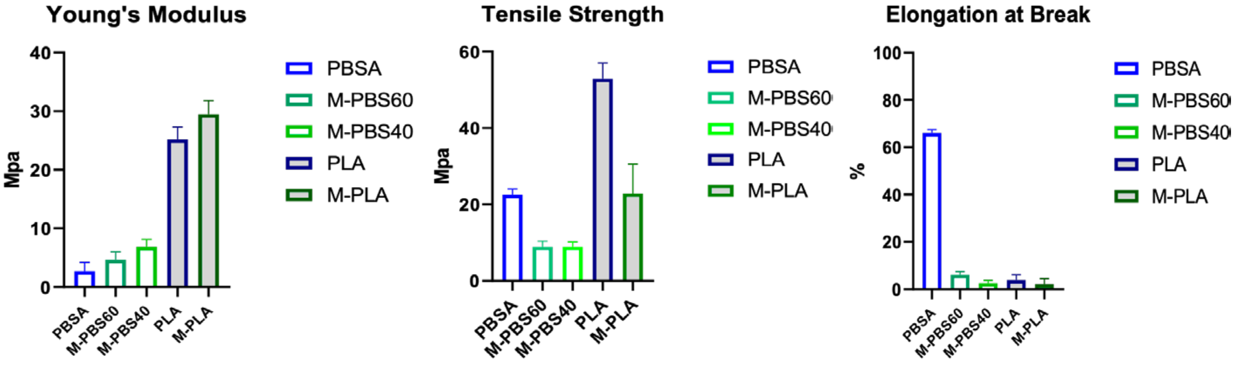


Figure 4. Mechanical properties of PBSA and PLA neat polymers and composites.

The results of the Figure 4 show that the M-PBS60, M-PBS40, and M-PLA blends are stiffer but less ductile than the pure PBS and PLA polymers. The stiffness increased by 17% for M-PLA, 70% for M-PBS60, and 150% for M-PBS40, while tensile strength decreased by 57% for M-PLA and around 48% for M-PBS60 and M-PBS40. Elongation at fracture also significantly decreased. These results align with torque rheometry tests and literature, which indicate that the excessive addition of fibrous material reduces ductility due to non-uniform load and stress transfer within biocomposites [22].

Conclusion

The results show that biocomposite blends with composted biosolids are stiffer but less ductile than pure polymers, due to the addition of fibrous material that reduces ductility.

Spectral analysis confirmed the presence of cellulose in the compost, contributing to lower thermal stability but promoting crystal formation at higher temperatures. Despite the high compost concentration, the thermal and rheological properties of the biocomposites remained similar to those of pure polymers, with compost acting as a nucleating agent.

Based on these findings, the incorporation of composted biosolids as the primary matrix in biocomposites is expected not only to enhance material stiffness but also to provide additional benefits to soil and crop growth, offering a sustainable and effective solution in agriculture.

Acknowledgments

We wish to extend our sincere gratitude to the Ministry of Science, Technology, and Innovation (MINCIENCIAS) in Colombia for their invaluable contributions to this research. We are particularly thankful for the funding provided through the doctoral scholarship awarded to Yina Ortega under the "Bicentenario 2" program. Additionally, we appreciate the support of the Agroindustrial Engineers from the Universidad Popular del Cesar—Marcela Quimbayo, Melissa Segura, Ariana Sierra Vera, and Yerfry Castro—for their assistance in producing the organic amendment.

References

1. H. Escalante, L. Castro, M. P. Amaya, L. Jaimes, and J. Jaimes-Estévez, "Anaerobic digestion of cheese whey: Energetic and nutritional potential for the dairy sector in developing countries," *Waste Management*, vol. 71, pp. 711–718, 2018, https://doi.org/10.1016/j.wasman.2017.09.026
2. Y. Freiberg, P. Fine, I. Levkovitch, and S. Baram, "Effects of the origins and stabilization of biosolids and biowastes on their phosphorous composition and extractability," *Waste Management*, vol. 113, pp. 145–153, 2020, https://doi.org/10.1016/j.wasman.2020.06.002
3. M. Buta, J. Hubeny, W. Zieliński, M. Harnisz, and E. Korzeniewska, "Sewage sludge in agriculture – the effects of selected chemical pollutants and emerging genetic resistance determinants on the quality of soil and crops – a review," *Ecotoxicology and Environmental Safety*, vol. 214, 2021, https://doi.org/10.1016/j.ecoenv.2021.112070
4. S. ElHady, O. Amin, A. Elhussieny, and I. S. Fahim, "Bioplastics, biodegradable plastics, and degradation in natural environments," in *Biodegradability of Conventional Plastics: Opportunities, Challenges, and Misconceptions*, Elsevier, 2022, pp. 47–67, https://doi.org/10.1016/B978-0-323-89858-4.00008-7
5. R. Dziuba, M. Kucharska, L. Madej-Kiełbik, K. Sulak, and M. Wiśniewska-Wrona, "Biopolymers and biomaterials for special applications within the context of the circular economy," *Materials*, vol. 14, no. 24, 2021, https://doi.org/10.3390/ma14247704
6. V. Siracusa, N. Lotti, A. Munari, and M. Dalla Rosa, "Poly (butylene succinate) and poly (butylene succinate-co-adipate) for food packaging applications: Gas barrier properties after stressed treatments," *Polymer Degradation and Stability*, vol. 122, pp. 10–16, 2015, https://doi.org/10.1016/j.polymdegradstab.2015.04.026
7. S. Wang, L. Daelemans, R. Fiorio, G. Maling, R. D. D'hooge, K. De Clerck, and L. Cardon, "Improving mechanical properties for extrusion-based additive manufacturing of poly(lactic acid) by annealing and blending with poly(3-hydroxybutyrate)," *Polymers*, vol. 11, no. 9, p. 1529, 2019, https://doi.org/10.3390/polym11091529
8. G. Dorez, A. Taguet, L. Ferry, J. Lopez-Cuesta, and J. M. Lopez-Cuesta, "Thermal and fire behavior of natural fibers/PBS biocomposites," *Polymer Degradation and Stability*, vol. 98, no. 1, pp. 1–10, 2013, https://doi.org/10.1016/j.polymdegradstab.2012.10.026

9. D. Aydemir and D. J. Gardner, "Biopolymer blends of polyhydroxybutyrate and polylactic acid reinforced with cellulose nanofibrils," *Carbohydrate Polymers*, vol. 250, p. 116867, Dec. 2020, https://doi.org/10.1016/j.carbpol.2020.116867

10. I. Armentano et al., "Processing and characterization of plasticized PLA/PHB blends for biodegradable multiphase systems," *Express Polymer Letters*, vol. 9, no. 7, pp. 583–596, 2015, https://doi.org/10.3144/expresspolymlett.2015.55

11. H. Tsuji and I. Fukui, "Enhanced thermal stability of poly(lactide)s in the melt by enantiomeric polymer blending," *Polymer*, vol. 44, no. 10, pp. 2891–2896, May 2003, https://doi.org/10.1016/S0032-3861(03)00175-7

12. A. Aguero, L. Quiles-Carrillo, A. Jorda-Vilaplana, O. Fenollar, and N. Montanes, "Effect of different compatibilizers on environmentally friendly composites from poly(lactic acid) and diatomaceous earth," *Polymer International*, vol. 68, no. 5, pp. 893–903, 2019, https://doi.org/10.1002/pi.5779

13. I. Cacciotti, S. Mori, V. Cherubini, and F. Nanni, "Eco-sustainable systems based on poly(lactic acid), diatomite and coffee grounds extract for food packaging," *International Journal of Biological Macromolecules*, vol. 112, pp. 567–575, 2018, https://doi.org/10.1016/j.ijbiomac.2018.01.144

14. R. Avolio et al., "Effect of cellulose structure and morphology on the properties of poly(butylene succinate-co-butylene adipate) biocomposites," *Carbohydrate Polymers*, vol. 133, pp. 408–420, 2015, https://doi.org/10.1016/j.carbpol.2015.07.072

15. Z. C. Lule and J. Kim, "Organic-inorganic hybrid filler for improved thermal conductivity and anti-dripping performance of polybutylene succinate composite," *Journal of Cleaner Production*, vol. 340, 2022, https://doi.org/10.1016/j.jclepro.2022.130781

16. M. M.-U. Haque, M. E. Errico, G. Gentile, M. Avella, and M. Pracella, "Properties of macromolecular materials," *Macromolecular Materials and Engineering*, vol. 297, pp. 985–992, 2012.

17. L. Jiang, E. Morelius, J. Zhang, M. Wolcott, and J. Holbery, "Sustainable composite materials from cellulose fiber and polylactic acid," *Journal of Composite Materials*, vol. 42, pp. 2629–2639, 2008, https://doi.org/10.1177/0021998308097740

18. J. Amaya-Pinos, "Thermo-mechanical study of the mixture of polylactic acid PLA obtained from potato starch with an aliphatic copolyester PBSA (polybutylene succinate adipate)," *Dyna*, vol. 89, no. 221, pp. 142–150, 2022

19. I. Armentano et al., "Processing and characterization of plasticized PLA/PHB blends for biodegradable multiphase systems," *Express Polymer Letters*, vol. 9, pp. 583–596, 2015.

20. A. N. Frone, S. Berlioz, J. F. Chailan, D. M. Panaitescu, and D. Donescu, "Cellulose fiber-reinforced polylactic acid composites," *Polymer Composites*, vol. 32, pp. 976–985, 2011, https://doi.org/10.1002/pc.21120

21. M. R. Nobile, A. Crocitti, M. Malinconico, G. Santagata, and P. Cerruti, "Preparation and characterization of PBS and PBAT biodegradable blends," *AIP Conference Proceedings*, vol. 1981, 2018, https://doi.org/10.1063/1.5046042

22. M. R. Rahman, "Engineering materials acacia wood bio-composites towards bio-sustainability of the environment," Springer, 2019, [Online]. Available: http://www.springer.com/series/4288