

Biocomposite films utilizing sugar cane bagasse and banana peel aiming seedling applications

Thiago Torres Matta Neves^{1*} , Simone Taguchi Borges¹ , Luiz Antonio Borges Junior¹ ,
Edla Maria Bezerra Lima² , Cristiane Hess de Azevedo Meleiro¹ , Ana Paula Duarte Moreira³ ,
Antonieta Middea⁴  and Renata Nunes Oliveira¹ 

¹*Programa de Pós-Graduação em Engenharia Química, Universidade Federal Rural do Rio de Janeiro – UFRRJ, Rio de Janeiro, RJ, Brasil*

²*Embrapa Agroindústria de Alimentos, Empresa Brasileira de Pesquisa Agropecuária – EMBRAPA, Rio de Janeiro, RJ, Brasil*

³*Laboratório de Propriedades Mecânicas, Universidade Federal do Rio de Janeiro – UFRJ, Rio de Janeiro, RJ, Brasil*

⁴*Departamento de Caracterização Tecnológica, Centro de Tecnologia Mineral – CETEM, Rio de Janeiro, RJ, Brasil*

*thiagotmneves@metalmat.ufrj.br

Abstract

This study developed biocomposite films from poly(vinyl alcohol) (PVA), sugar cane bagasse (SCB), and banana peel fibers (BF) or starch-rich banana flour (BS). Morphological analysis revealed filler distribution and fluid percolation within the polymer matrix. Physicochemical analysis indicated stronger interactions between components in BF-containing films. Mechanical strength decreased significantly in SCB-containing films, while biodegradation increased, particularly with banana waste. Water absorption was higher in PVA-BF-SCB and PVA-BS-CSB biocomposites. Incorporating SCB and banana waste into PVA films presents a promising approach for developing biodegradable composite packaging materials, potentially replacing low-density polyethylene in applications like seedling production. This biodegradable material can be applied directly to the environment.

Keywords: *degradation, PVA, sugar cane bagasse, banana, hydrogel.*

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1. Introduction

Polymeric packaging contributes significantly to worldwide waste. Polymers are used as packaging due to critical properties such as resistance to permeation, physical integrity, and economic imperatives^[1]. However, the current usage levels of such packaging are creating serious and urgent sustainability concerns. These impacts include accumulating polymeric waste in landfills and, notably, in oceanic environments. Biodegradable polymers continue to be investigated as potential alternatives for packaging with a short shelf life or single-use^[2].

Traditionally, in producing seedlings or as soil cover for growing vegetables and certain fruits (such as strawberries), low-density polyethylene (LDPE) is used to suppress weeds and maintain humidity and soil heating to favor plant growth^[3]. To reduce the use of these polymers with a limited period of use in the agroindustry, studies have been carried out to develop biodegradable packaging for producing and planting seedlings^[4].

Poly (vinyl alcohol) (PVA) is a non-toxic, hydrophilic, biodegradable biopolymer. It is classically used as a base material to produce hydrogels with high water uptake properties^[5]. PVA films can be classified according to their cross-linking process as physical or chemical. In physical cross-linking, the cryogelation process promotes, in a milder and more environmentally friendly way, the formation of a physical network of cross-links and secondary bonds, such as hydrogen or ionic bonds, which are reversible and facilitate the degradation process. This process also causes phase separation during freezing, forming a water-rich phase, which upon thawing forms small pores, and another phase rich in polymer. In contrast, chemical cross-linking forms covalent bonds, which hinder the degradation process. Furthermore, cross-linking agents, typically toxic, can impart toxicity to the soil when degraded^[6-8]. Films produced from ternary mixtures of PVA/polysaccharides/water, for example, through the cryogelation process, can transform the water-rich phase into a phase composed of the biopolymer, while the PVA-rich phase becomes composed of a phase of both

polymers. In this case, the crystalline phase is formed by the approximation of the macromolecular chains that interact through hydrogen bonding via the cryostructuring process^[7].

PVA's hydrolytic degradability is related to the proportion of its acetate groups remaining unsubstituted by hydroxyl groups following manufacture. 12% is viewed as being sufficient to contribute to degradability in water^[9]. Hydrolytic degradation is considered advantageous in some applications, such as soluble packaging ready for consumption, or aiming to introduce fertilizers and moisture regulators into the soil, without increasing pollution^[10]. PVA also slowly degrades in soil^[11] with some microorganisms able to consume PVA^[12].

The main disadvantage in replacing single-use plastic packaging with this material is related to its high cost. One alternative is to mix this polymer with natural products, biofillers (starches or fibers), which can be derived from abundant agro-industrial waste, helping to reduce production costs. The use of these biofillers can increase or decrease the mechanical strength of the material, depending on the granulometry, proportion used, and whether or not chemical treatment of the fibers is performed. Additionally, it favors the biodegradation of these materials due to their hydrophilic nature^[8,13-15]. It is essential that the plant-based materials used in biocomposites are not intended for human consumption.

Natural fibers stand out due to their notable properties, such as low density, low cost, easy availability, biodegradability, and ease of processing, as well as their good thermal stability^[13].

SCB is a considerable source of waste in Brazil, where 174 million tons of bagasse residues (from sugar cane juice extraction) were generated in 2012. SCB is a degradable lignocellulosic material rich in carbohydrates^[16]. Among the microorganisms that decompose/consume sugar cane bagasse are bacteria and fungi, but SCB can be a substrate for the growth of earthworms^[17]. PVA has previously been combined with lignocellulosic natural material, such as biodegradable nanofibers of purified coir cellulose (from coconut waste husks)^[18]. Cellulose nanocrystals and chitin nanofibers loaded to PVA increased the samples' mechanical properties and thermal resistance to degradation^[19].

Brazil is an important producer of bananas, where the banana industry produces over 57.6 million metric tons of banana peels per year. Banana flour is rich in starch^[20], while banana peel flour is rich in carbohydrates and hemicelluloses^[21]. Banana peels are used as a culture medium for microorganisms^[22]. Chitosan mixed with banana flour led to biodegradable films, where starch makes the films water soluble, and chitosan adds a bactericidal characteristic^[23]. PVA-starch films degraded considerably (~70%) in 30 days when exposed to fungi compared to the films in contact with compost (~60%)^[24]. In addition, PVA-starch samples are susceptible to enzymatic degradation^[25].

PVA/starch or PVA/hydroxypropylmethylcellulose films have been proposed as an alternative to replace traditional mulching films in agriculture, helping to control soil temperature and retain moisture. Additionally, these materials do not require removal after serving their purpose, as they can naturally deteriorate, releasing nitrogen-rich elements/ions that act as fertilizers, influencing plant growth^[26,27]. Due to their hydrophilic nature, the presence of starch and/or cellulose and their derivatives as added components to PVA tends to increase

water solubility, facilitating bacterial diffusion through the material and, consequently, accelerating the biodegradation process of these materials in soil^[26]. On the other hand, this addition can also promote a reduction in mechanical properties compared to pure PVA, due to the increase in amorphous regions and weak intermolecular bonds^[26].

The present study aims to develop, manufacture and characterize low-cost biodegradable samples based on PVA mixed with SCB and/or banana parts (banana peel flour (BF) and banana flour (BS)). As a sustainable alternative to PE, particularly in agricultural applications with single-use packaging and low mechanical stress, various formulations were tested intending to evaluate its physical-chemical characteristics, mechanical behavior, and degradation properties.

2. Materials and Methods

2.1 Materials

Poly (vinyl alcohol) (PVA) (Mw 85.000-124.000 Da, 99% hydrolyzed) was purchased from SIGMA-ALDRICH, Germany; Food Engineering Department / UFRRJ donated 24 bananas (*Musa* spp.); sugar cane bagasse was collected from a local market (Seropédica / RJ / Brazil) as waste material.

The bananas were cut and dried in an oven (FABBE – PRIMAR, Brazil) at 52°C for 48h, as was the SCB. Both bananas and SCB were ground using a PERTEN LABORATORY MILL 3600 from Sweden. The ground banana was washed several times with distilled water using a sieve (aperture of 106µm). The retained material (banana fibers - BF) was dried in the oven as described. The washing fluid presented sedimentation, and after 2h, it was centrifuged using a HETTICH ZENTRIFUGEN, 320 R model, Germany, at 9000 RPM for 15 min, obtaining a solid fraction of banana starch (BS). The different granulometries used in organic loads have the function of assisting in the biodegradability process, creating preferential pathways that facilitate the entry of microorganisms, passing through the PVA and reaching the interior of the composite, favoring the proliferation of colonies of these microorganisms and accelerating the biodegradation process.

2.1.1 Samples manufacturing

The samples were prepared using a casting method according to the compositions described in Table 1, with the added fillers always corresponding to 20% of the mass value (g) in the loaded films. The PVA was poured into 100 mL of distilled water under mechanical stirring (equipment FISATOM 710, Brazil) and kept at ~80°C for 4h to achieve a homogeneous polymeric solution. The

Table 1. Composition of Samples.

Material	PVA (g)	SCB (g)	BF (g)	BS (g)
PVA	10	-	-	-
PVA-SCB	8	2	-	-
PVA-BF	8	-	2	-
PVA-BS	8	-	-	2
PVA-SCB-BF	8	1	1	-
PVA-SCB-BS	8	1	-	1

loaded material (e.g., banana peel, SCB) was added when the solutions reached room temperature while stirring was maintained. At room temperature, 20 mL of each solution was poured into Petri dishes (ϕ 150 mm). It was followed by freeze-thawing cycles, where the samples were kept at -16°C for 18h, followed by 2 cycles of 40 min at room temperature and 1h at -16°C before final thawing and drying in an oven (NOVA INSTRUMENTS, NI 1512 model, from Brazil) at 50°C for 24h.

2.2 Characterization of materials

2.2.1 X-ray Diffraction (XRD)

The mineralogical analysis of the materials were carried out by XRD. The samples were back-loaded to avoid the preferred orientation, and the XRD patterns were obtained with a Bruker-AXS D8 Advance Eco (Germany) operating at a tube voltage and current of 40 kV and 25 mA, respectively, using Cu $k\alpha$ radiation. Diffraction patterns were recorded between $2\theta=5^{\circ}$ and 80° with a 0.02° step and a position-sensitive LynxEye detector (Germany).

2.2.2 Fourier Transformed Infrared Spectroscopy (FTIR)

The physicochemical analysis of the samples was performed by FTIR, Perkin-Elmer Spectrum 100, from USA, ATR mode, wavenumber between 4000 cm^{-1} and 600 cm^{-1} , 16 scans/sample, spectral resolution of 4 cm^{-1} .

2.2.3 Stereoscopic Photographs

Non-degraded samples were photographed using a Sony Cyber-Shot DSC-HX200V (Brazil) camera. Macrographs of the samples produced were then obtained on a black background using the Zeiss Stemi 508 stereoscope (Germany) with the Zeiss AxioCam 208 color camera (Germany) attached, with 1x magnification eyepieces, showing an accurate magnification of 6.3x.

2.2.4 Scanning Electron Microscope with Energy-Dispersive Spectroscopy (SEM/EDS)

The materials' morphology and elemental composition (coated with Ag) were identified using two scanning electron microscopes. The first one observed the samples by backscattered electron mode in an FEI Quanta 400 scanning electron microscope (USA) operating at 20 kV and small spot size with an EDS coupled using a Quantax Esprit XFlash 6160, Bruker, from Germany, for SEM. The second was a Hitachi TM3030Plus (Japan) scanning electron microscope with an integrated EDS (Bruker Quantax 70 from Germany). This SEM was operated at 15 kV, a small spot size, while images were also acquired in backscattered electron mode (BSE) for elemental contrast, and EDS maps were used to identify the elements of interest accumulated during 200s.

2.2.5 Mechanical analysis

A texture analyzer TA.XT Plus, from England, (P5S, diameter of 4.85 mm, EMBRAPA Agroindustry) was used with a load cell of 0.05N and speed of 1 mm/s. Three samples of each composition were tested, with a sample size of $(2.5 \times 2.5)\text{ cm}^2$. The results were analyzed using ANOVA to verify whether the films presented significant

variations, applying the Scott-Knot test ($p < 0.05$) to detect differences between the average results.

2.2.6 Degradation analysis

The samples of $2.5 \times 2.5\text{ cm}^2$ were cut and weighed to perform the hydrolytic degradation and biodegradation tests. Triplicates with similar weights were tested for each composition. Samples were immersed in a constant volume of sterile saline solution (20 mL) at room temperature for 50 days, after the first 30 days, the solution was changed weekly. The samples were then dried in an oven (50°C , 24h) and weighed. The samples' weight loss (WL) was calculated according to Equation 1^[28], where W_D is the samples' original dry weight and W_{DS} is the samples' dry weight after 50 days of immersion in media/water.

$$WL(\%) = 100 \frac{W_D - W_{DS}}{W_D} \quad (1)$$

For biodegradation tests^[29], the triplicates were weighed and buried in humus from southeast Brazil. The samples were placed on humus soil with a height of 5 cm and covered with humus soil to a depth of 5 cm. The soil was hydrated weekly with $\sim 75\text{ mL/sample}$ of rainwater (450mL). After 50 days, the samples were removed from the soil, washed with running water, dried in the oven as described, and weighed. The weight loss of the samples was calculated according to Equation 1. Once more, the results were analyzed using ANOVA to verify whether the loss of mass observed after the degradation tests was significant between the films by applying the Scott-Knot test ($p < 0.05$).

2.2.7 Water absorption

The samples used in the hydrolytic degradation tests were also used for the water absorption test. Before undergoing the drying process, with the aid of paper towels, the samples were lightly dried to remove excess water and then weighed to obtain the wet mass of the samples. Subsequently, the percentage of water absorption was calculated as a function of the wet mass gain and in relation to the dry mass of the samples, following the equation 1.

3. Results and Discussions

3.1 XRD – Mineralogical analysis

The XRD patterns of six biocomposites (PVA, PVA/Banana starch-BS, PVA/SCB, PVA/Banana Fiber-BF, PVA/BS/SCB, PVA/BF/SCB) are shown in Figure 1(1) and their respective crystallinities in Table 2. The diffractogram of the pure PVA film was used as a reference to investigate the behavior of the other five different compositions. The maximum intensity diffraction of the PVA peak observed was at $2\theta = 19.8^{\circ}$ corresponding to d-spacing 4.4801 \AA and indicated the presence of a typical semicrystalline structure, exhibiting a low degree of crystallinity^[30].

The biocomposite fillers present two compositions in their majority: Starch is found in higher concentration in banana starch (BS) and SCB fillers, and cellulose is representative in banana fiber (BF). Both starch, cellulose, and PVA separately present semicrystalline structures^[31].

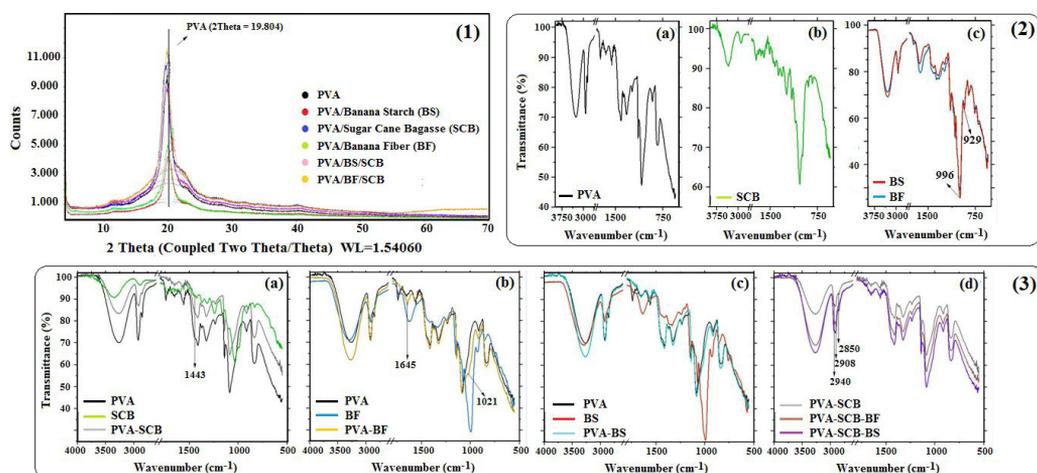


Figure 1. (1) X-ray diffraction patterns of six biocomposites. (2) FTIR spectra of: (a) PVA; (b) sugar cane bagasse (SCB) and (c) banana fibers (BF) and starch (BS). (3) FTIR spectra of: (a) PVA-SCB sample compared to PVA and SCB spectra; (b) PVA-BF and (c) PVA-BS compared to PVA and BF or BS spectra, respectively; (d) PVA-SCB-BF and PVA-SCB-BS spectra compared to PVA-SCB spectrum.

Table 2. Crystallinities of six biocomposites: PVA, PVA/Banana starch-BS, PVA/SCB, PVA/Banana Fiber-BF, PVA/BS/SCB and PVA/BF/SCB.

Biocomposites	Crystallinity (%)	Amorphous (%)
PVA	37.6	62.4
PVA/Banana Starch (BS)	29.8	70.2
PVA/Sugar Cane Bagasse (SCB)	36.4	63.6
PVA/Banana Fiber (BF)	26.1	73.9
PVA/BS/SCB	27.3	72.7
PVA/BF/SCB	26.7	73.3

An attempt to improve the degree of crystallinity of these films and, possibly, their mechanical strength, can be made by optimizing the starch (BS) content added to the mixture, since at high concentrations, the presence of the polysaccharide can disrupt the crystalline nature of the film instead of increasing this phase^[7]. Another factor is the chemical treatment performed on the fibers, which removes the amorphous part of the fibers, increasing the crystalline phase and improving the molecular ordering in the amorphous phase of PVA. The larger the crystal size, the more hydrophobic the fiber becomes, influencing the mechanical and physical properties of the composite^[13,15].

From the XRD analysis, it is essential to highlight that the amount of vegetal filler in the biocomposite represents 20%; thus, the well-defined structure of these fillers will influence the intensity of the X-ray peaks without highly varying the crystallinity x amorphous ratio. The variation in granulometry between the fillers can also affect the response, considering whether the point where the analysis was performed had a higher or lower agglomeration of fillers, thus allowing the matrix (PVA) to influence more the degree of crystallinity compared to other components.

3.2 FTIR studies

3.2.1 Raw materials

FTIR data regarding the individual raw materials are displayed in Figure 1(2), and their vibration modes are in

Table 3. SCB^[32], and PVA^[33] exhibit their characteristic bands, as do banana-sourced samples^[34], although there are some differences in the intensity of bands for fibers and starch. Starch and banana bagasse/fibers usually present bands of 300-2800 cm⁻¹ with varied intensity due to different amylose and amylopectin contents. The banana fibers and banana starch spectra are similar, where the presence of fibers and starch in both banana samples indicates incomplete separation. The band at 996 cm⁻¹ can be referred to as the starch and fibers spectra, where it can be not only related to Starch's δ(C-O-C) of the α-(1-4) glycosidic bonds^[35], but also to the amount of amorphous phase, where the amount of water interacting with intramolecular hydrogen bonds vary according to the crystallinity of the samples. Differences in the position of the band around 929 cm⁻¹ in fibers and starch could be due to varied amounts of amylopectin α-1,6 bonds^[36].

3.2.2 Biocomposites

The blended samples present most bands usually associated with PVA (Figure 1(3)), besides slight contribution of the other added materials. By evaluating composites of PVA-nanocellulose (obtained from sugar cane bagasse), they presented bands of both components, suggesting that only physical interactions took place^[48]. Physical interactions were observed in the composite samples and humidity in the samples containing banana parts.

3.3 Morphological aspects

3.3.1 PVA blended films

The films produced appear to show homogeneous dispersion of the incorporated natural materials, as seen in the photographs in Figure 2(1) since no phase separation is apparent. Although the matrix remains relatively transparent in the films, the addition of banana compounds (BF and BS) rendered the films slightly opaque compared to the pure PVA film. The addition of SCB resulted in a rougher surface due to the large particle size of the component used, on a millimetric and centimetric scale^[49,50]. Figure 2(2) shows macrographs with 6.3x magnification of biocomposites.

Table 3. FTIR wavenumbers and respective vibration modes of PVA, SCB, and banana (fibers and starch) samples.

PVA bands vibrations		SCB bands vibrations		Banana bands vibrations	
(cm ⁻¹)	Mode	(cm ⁻¹)	Mode	(cm ⁻¹)	Mode
3263	v(-OH) of hydrogen bonds ^[37]	3344	v(OH) of lignin, carbohydrates, phenols ^[32]	3299	v(O-H) bonds; hydrogen bond between OH groups ^[45]
2921	v(C-H) ^[38]	2899	C-H _n (aliphatic, aromatic) ^[44]	2926	v(C-H) of CH, CH ₂ groups in cellulose, hemicelluloses ^[34]
2850	v _s (C-H) from alkyl groups ^[39]	1727	v(CO) (ketone, carbonyl) ^[44]	1734	C=O of ketone, carbonyl groups, mainly related to lignin, but also waxes, pectins ^[46] or of ester, anhydride ^[34]
1744	C=O from remaining acetate groups ^[40]	1684	v(C=O) of carbonyl groups ^[32]	1626	Related mainly to banana fibres: v(C=O) of amide I of proteins ^[36]
1653	δ(HOH) ^[41]	1654	v _{AS} (C=O) of carboxylic groups ^[32]	1412	δ(N-H) of -NH ₂ ^[34]
1560	v(C=C) ^[33]	1633	v(C=C) (benzene ring of lignin) ^[44]	1372	δ(OH) ^[34]
1414	δ(CH ₂) ^[38]	1603	aromatic skeletal modes of lignin ^[44]	1241	Starch's flexion and torsional of C-O-H and C-H groups ^[35] and fibers C-H ^[34]
1376	ω(-CH ₂) ^[42]	1559	CO + aromatic of carbohydrates and lignin ^[44]	1148	Fibers' amide III of proteins ^[36]
1325	δ(-C-H-), δ(-O-H-) ^[42]	1516	skeletal modes of carbohydrates and lignin ^[44]	1077	Starch's v(C-C); ^[35] fibres' v(C-O) of ester or ether ^[34]
1236	v(C-C) ^[43]	1508	v(C=C) of aromatic rings ^[32]	996	Starch's δ(C-O-C) of the α-(1-4) glycosidic bonds ^[35]
1143	PVA crystallites ^[36] , v(C-C) and v(C-O-C) ^[38]	1426	v(C-O) of carboxylic groups ^[32]	929	Starch's α-(1-6) bond of amylopectin, ^[35] glycosidic bonds; ^[36] amorphous phase of fibres and starch ^[36]
1089	out of plane C-O vibration ^[43]	1372	C-H deformation of -CH ₃ , -CH ₂ - ^[32]	860	Amine groups of proteins ^[47]
916	ρ(CH ₂) ^[38]	1318	v(C-O) of carboxylate groups ^[32]	760	δ(C=C); ^[34] phenols' aromatic structures ^[36]
835	v(C-C) ^[38]	1240	C=O deformation of carboxylic acids ^[32]	706	Phenols' aromatic structures ^[36]
		1116	-		
		1105	O-H ass., CO def. (lignin) ^[44]		
		1034	v(C-O-C, O-H) of polysaccharides ^[32]		
		988	C-H aromatic of carbohydrates and lignin ^[44]		
		898			
		833			

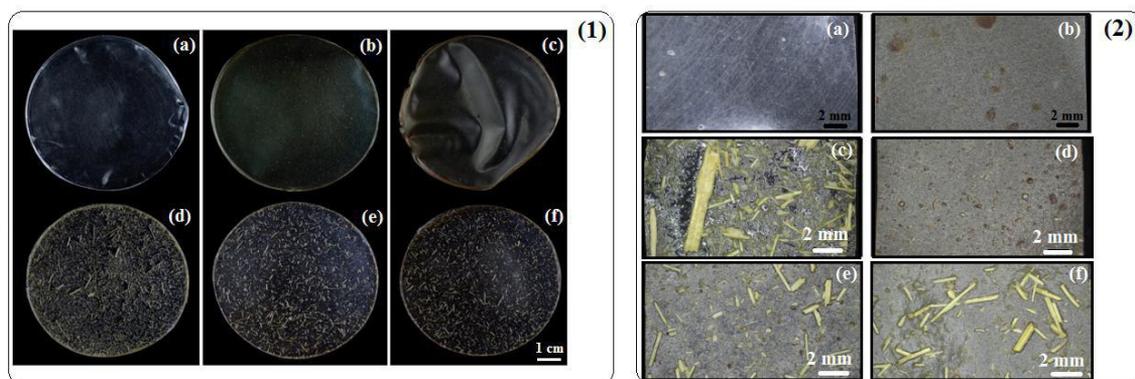


Figure 2. (1) Samples morphology: a) PVA; b) PVA-BF; c) PVA-BS; d) PVA-SCB; e) PVA-SCB-BS; and f) PVA-SCB-BF. (2) Macrographs with 6.3x magnification of biocomposites: (a) PVA; (b) PVA/BS; (c) PVA/SCB; (d) PVA/BF; (e) PVA/SCB/BS and (f) PVA/SCB/BF.

3.3.2 SEM - microtexture studies

The SEM images of the six biocomposites obtained in this study presented characteristic morphology and can be observed in Figure 3. The BSE images reveal that small

PVA particles were not dissolved during the casting process. Starch's presence is evident in the PVA-BS sample (Figure 3b) by its characteristic circular geometry. Despite the washing and starch extraction procedures applied to the green banana

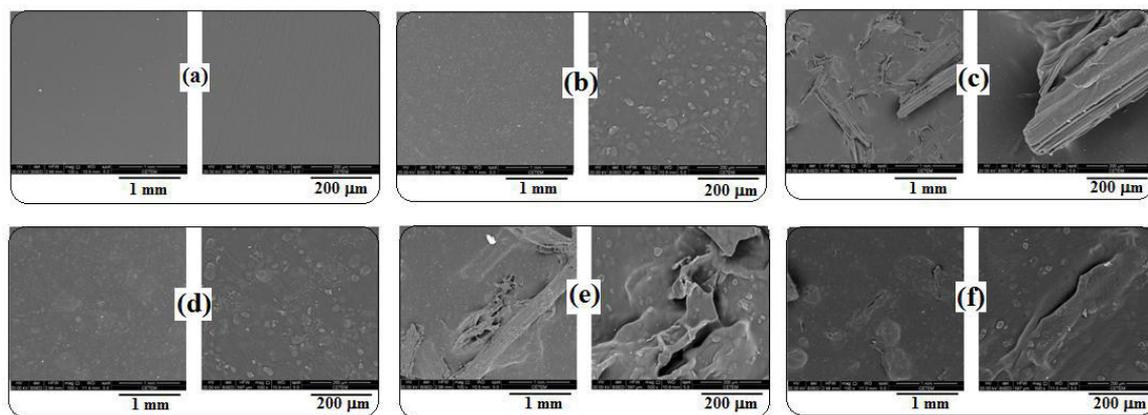


Figure 3. SEM images of six biocomposites: (a) PVA; (b) PVA/BS; (c) PVA/SCB; (d) PVA/BF; (e) PVA/SCB/BS and (f) PVA/SCB/BF.

peels, significant amounts of starch are still visible in the samples loaded with banana fiber. Furthermore, the banana fibers are not evenly distributed, resulting in concentrated fiber veins within the film.

Through SEM, it is possible to observe spaces between the filler and the matrix. The dispersion of the fillers on a micrometric scale (BF and BS) favored the encapsulation of these fillers by the polymeric matrix (Figures 3b and 3d). Compared to the samples containing SCB with a centimetric particle size, a decrease in the specific surface area is observed in relation to the other samples, where the interaction with the polymeric matrix occurs through anchoring (Figures 3c and 3f).

The low adhesion between the organic components (BS, BF, and SCB) with the PVA film can also be observed by FTIR (Figure 4).

3.4 Mechanical properties

The PVA and PVA composite films presented similar Young's modulus ($p > 0.05$). However, there is a significant difference between the failure strengths (σ), where \int_{PVA} (2.34 ± 0.93 MPa) is higher than the failure strength of all samples containing sugar cane bagasse, PVA-SCB (0.64 ± 0.15 MPa), PVA-SCB-BS (0.55 ± 0.10 MPa) and PVA-SCB-BF (0.81 ± 0.21 MPa), $p < 0.05$ (Figure 4(1)). In addition, there is a difference ($p < 0.05$) between the strength of \int_{PVA-BF} (1.43 ± 0.07 MPa) $>$ $\int_{PVA-SCB}$ and \int_{PVA-BS} (1.86 ± 0.36 MPa) $>$ $\int_{PVA-SCB-BS}$. The addition of fibers disrupts the intermolecular interactions, mainly hydrogen bonds, between PVA and amylose/amylopectin. Some researchers^[10,50], also observed a reduction in mechanical properties when adding fruit flour as fillers to PVA films, due to the low compatibility of the flours with PVA.

When the cellulose fibers and starch in a film present low water content, this can be expected to increase sample strength^[51]. However, adding oil and cassava bagasse rich in starch has lowered PVA water resistance^[52]. In this case, it can be observed that the addition of sugar cane bagasse reduced the samples' strength, possibly due to SCB hydration.

It is worth noting that the variations observed in the tensile strength among the different combinations highlight the complex interactions between the various components within the biocomposite system. These interactions can be

influenced by factors such as filler-matrix compatibility, filler particle size, dispersion, and interfacial adhesion, which ultimately affect the overall mechanical performance of the developed sheets^[53].

The biocomposites with higher mechanical strengths are associated with smaller granulometric-sized fillers obtained through grinding, which indicates that the semi-crystalline regions present show a lower intensity of XRD signals due to the breakage of the amylopectin chains present in the starches. Samples incorporating BF and BS display micrometric granulometry, demonstrating enhanced mechanical strength as a result of the effective dispersion of fillers within the polymeric matrix. Notably, the addition of nanometric fillers derived from the seed coat of *Putranjiva roxburghii* yielded a significant 32.94% improvement in tensile strength^[15]. A comparative analysis with SCB samples possessing centimetric granulometry reveals a lower specific surface area for equivalent mass loadings (g), leading to an anchoring effect with the matrix that compromises mechanical strength (Figure 4-1).

These results may be primarily related to the reduction in the % crystallinity observed in the XRD characterization of the composite films concerning the PVA film (Table 2 and Figure 1(1)) and the low adhesion between the organic components (BS, BF, and SCB), as can be observed in the microscopy images obtained by SEM (Figure 3).

One approach to enhancing the mechanical resistance of natural fibers is through chemical modification^[8,13,15,53,54]. For use in composites, natural fibers like cellulose must be separated from other components, such as lignin, hemicelluloses, wax, and proteins, in the initial stage^[53]. Chemical treatments can reduce the amorphous part of the fibers, increasing cellulose content and consequently improving mechanical resistance and crystallinity degree^[13].

The addition of a chemical crosslinking agent increased the resistance to 13.38 MPa, while the addition of 1% (m/m) modified fiber further increased the resistance to 18.54 MPa. Alkaline treatment stabilizes the cellulose present in the fiber, removes non-cellulosic elements from the surface, and promotes better adhesion between the fibers and the matrix. Moreover, the greater comminution of SCB would also contribute to increasing the contact area with the PVA matrix^[8,15,53].

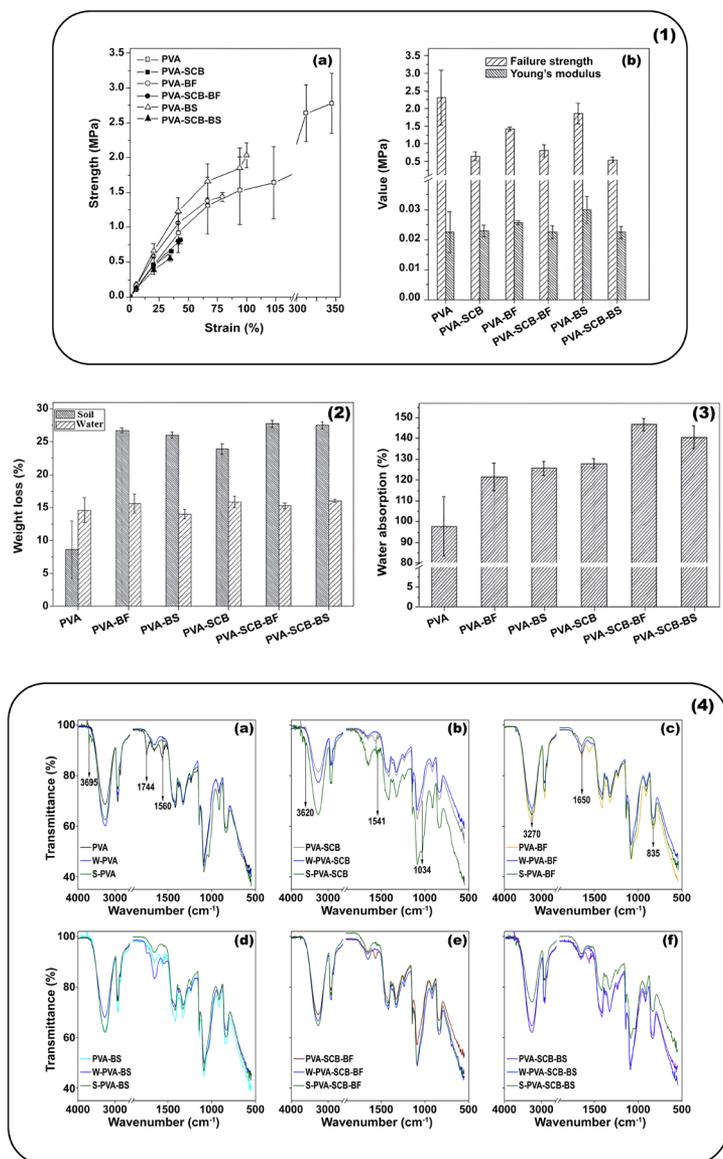


Figure 4. (1) Mechanical properties of: (a) PVA and (b) PVA-based composite films. (2) Degradation of six biocomposite samples (hydrolytic - water and biodegradation - soil). (3) Water absorption of six biocomposite films and (4) FTIR spectra of the samples after 50 days degradation in the w – water and s – soil: (a) PVA; (b) PVA-SCB; (c) PVA-BF; (d) PVA-BS; (e) PVA-SCB-BF and (f) PVA-SCB-BS.

3.5 Degradation

The pure PVA samples showed low biodegradation, with soil degradation of $8.65 \pm 4.35\%$ mass loss, compared to hydrolytic degradation, with water degradation of $14.58 \pm 1.88\%$ mass loss ($p < 0.05$). Hydrolytic degradation is considered advantageous in some applications, such as soluble packaging ready for consumption, which can also act as fertilizers without increasing pollution^[10]. The characteristic behavior of PVA samples may be attributed to the leaching of chains by the media, caused by water absorption and the breakage of hydrogen bonds associated with OH groups^[55].

The films containing natural fillers (BF, BS, SCB) exhibited higher degradation in soil (biodegradation, above 23% in all samples) than in water (hydrolytic degradation, where the

greatest mass loss was observed in the PVA-BS-SCB sample with $16.03 \pm 0.23\%$, $p < 0.05$, measured in terms of mass loss after 50 days (Figure 4(2)). The films containing natural materials (SCB and banana) exhibited higher biodegradation than pure PVA ($8.65 \pm 4.35\%$), indicating a significant increase in the biodegradation of composite films compared to PVA film ($p < 0.05$)^[8]. The highest biodegradation rates were observed in the PVA-SCB-BF ($27.72 \pm 0.53\%$) and PVA-SCB-BS ($27.48 \pm 0.50\%$) films. Since starch and PVA are biodegradable materials, their blends are considered potential sustainable materials for packaging and agriculture. The chemical treatment performed on the fibers, as well as the use of chemical crosslinkers, decreases biodegradation. SCB is a material that stimulates biodegrading biota, as do

banana constituents. The combination of these materials is expected to support biodegradation. Water absorption is an important property, especially for the development of biodegradable materials^[50,56]. Soil moisture is absorbed by the films, promoting their swelling, which, associated with the lack of chemical interaction between the filler and the matrix, promotes the development of preferred pathways for the passage of fluids (air, water, microorganisms, and organic waste) and natural materials increase the activity of microorganisms towards these samples^[8,29,50,54,57].

3.5.1 Water absorption

All loaded samples exhibited higher absorption than pure PVA ($97.70 \pm 14.30\%$), while all other samples showed absorption above 121%, Figure 4(3). This value is lower than that observed some works^[7,8]. This occurs because the absorption measurement was performed on the samples after 50 days of hydrolytic degradation. The highest absorption rates were observed in the PVA-BF-SCB and PVA-BS-SCB composites, with $146.72 \pm 2.95\%$ and $140.63 \pm 5.29\%$, respectively. The addition of crosslinkers and modified fibers can reduced water absorption, due to the increased crystallinity of PVA and the hydrophobicity conferred to the fiber by the treatment^[8,15].

The increased absorption of the composite films is due to the greater affinity of water with the O-H groups present in these films, either by the addition of starch (BS) or by the contamination observed in BF^[7]. The poor chemical compatibility observed between PVA and banana flour increased the free volume and, consequently, the water absorption^[50]. Additionally, the coarser granulometry of

SCB, which also showed a lack of chemical interactions with the matrix, creates pathways for water percolation, also aiding the degradation process^[29].

3.5.2 FTIR/SEM-EDS after degradation

The samples were also evaluated by FTIR (Figure 4(4)) after 50 days in the two media. The hydrolytically degraded samples presented the absence or the shift of the band at $\sim 1560\text{ cm}^{-1}$ (related to the PVA $\nu(\text{C}=\text{C})$ ^[58] vibration due to residual acetate groups^[59]) and the absence of the band at $\sim 1744\text{ cm}^{-1}$ ($\text{C}=\text{O}$ vibration of PVA residual acetate^[60]). The residual acetate groups in PVA play an essential role in promoting degradation^[61]. The absence of the bands associated with residual acetate groups is also observed in biodegradation, where the acetate groups enhance degradation in soil^[61]. In addition, it was observed that the biodegraded samples presented some soil bands, e.g., bands at 3695 cm^{-1} (vibration of OH groups from the Al-OH and Si-OH surfaces), and at 3620 cm^{-1} ($\nu(\text{O}-\text{H})$ of clay minerals)^[62]. Most samples degraded in soil presented an additional band at $(1030-1040)\text{ cm}^{-1}$, related to $\nu(\text{Si}-\text{O})$ of soil silicates^[63]. The bands at 1650 cm^{-1} and 835 cm^{-1} are present in all samples, while in some samples, they are more intense due to hydro- and biodegradation. The high intensity of the last band ($\sim 835\text{ cm}^{-1}$) would be related to the biotransformation of polysaccharides from starch and cellulose in low-molecular-weight sugars by microorganisms and due to their presence in the samples^[64].

The degraded samples presented a variety of morphologies according to the media to which they were exposed (Figures 5(1), 5(2) and 5(3)). The sample containing SCB

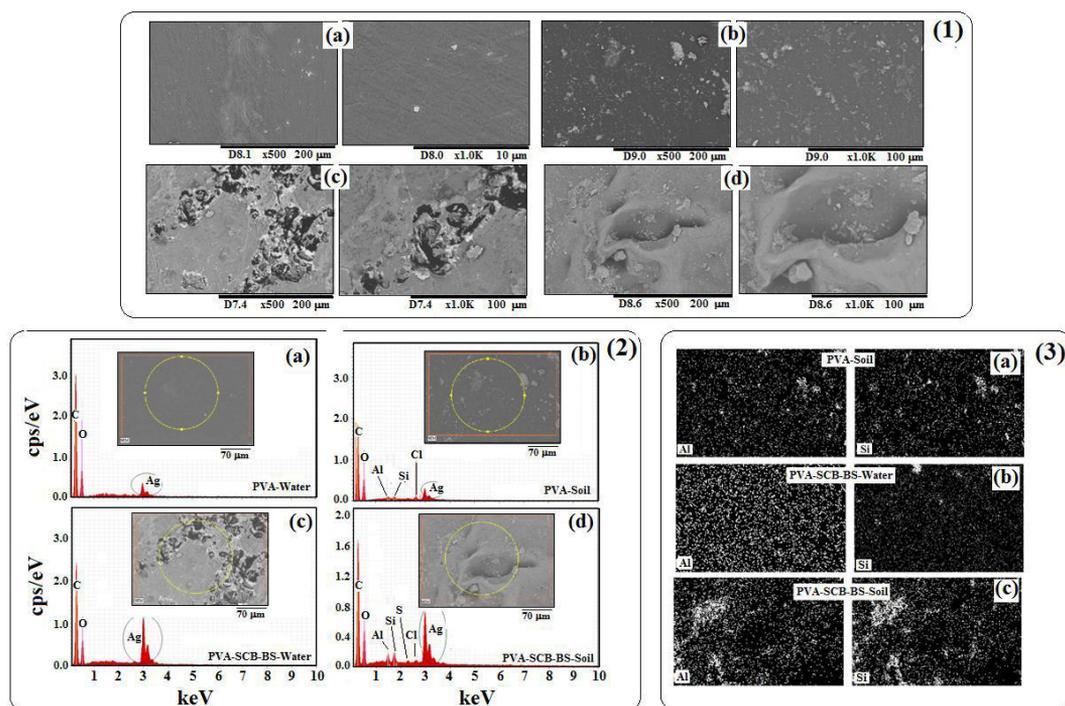


Figure 5. (1) SEM BSE images of the samples: (a) PVA degraded in water; (b) PVA degraded in soil; (c) PVA-SCB-BS degraded in water and (d) PVA-SCB-BS degraded in soil. (2): EDS spectra and the corresponding BSE images of: a) PVA degraded in water; (b) PVA degraded in soil; (c) PVA-SCB-BS degraded in water and (d) PVA-SCB-BS degraded in soil. (3): X-ray maps showing the distribution of the Al and Si elements present in the: (a) PVA degraded in soil; (b) PVA-SCB-BS degraded in water and (c) PVA-SCB-BS degraded in soil.

and banana starch became porous when degraded in water or soil (Figure 5(1)). Porosity and cracks all over the sample are evidence of degradation^[65,66], and this was also apparent in the weight loss measurements. The samples degraded in soil presented soil residues, as identified by FTIR, which could also be evaluated by EDX (Figure 5(2)). The Si and Al maps (Figure 5(3)) presented more evidence of silicon-based material on the PVA-SCB-BS sample immersed in soil. Silicon and aluminum are abundant elements in soil^[67], and are also present in sugar cane bagasse, which justifies the presence of Si in the hydrolytically degraded PVA-SCB-BS sample and the higher amount of Si in the same sample degraded in soil.

4. Conclusions

The combination of banana and SCB filler in the PVA formulation increases the amorphous phase when banana fibers are present (PVA/BF/SCB) by about 17.48% and by 16.51% when starch is present (PVA/BS/SCB) compared to the pure PVA film. FTIR analysis revealed no chemical interactions between the fillers and the matrix. Physical anchoring interactions are observed in the biocomposites and there is an indication of water content in the samples containing banana parts. The addition of sugar cane bagasse reduced the mechanical strength compared to pure PVA, possibly due to hydration of the bagasse. Although the tensile strength of films containing SCB decreased by up to 76.55% in the PVA-BS-SCB film compared to pure PVA, the PVA-SCB-BF and PVA-SCB-BS biocomposites exhibited higher biodegradation rates, approximately 27.72% and 27.48%, respectively, in mass loss after 50 days in soil, suggesting that the addition of natural materials as filler stimulates the local biota. Biodegradation (in soil) led to the absence of acetate groups, the presence of soil, and indications of degradation of the samples by microorganisms (biotransformation of polysaccharides into low molecular weight sugars). And water absorption increased significantly in these biocomposites by 146.72±2.95% and 140.63±5.29%, respectively, in mass gain. The data gathered highlight the potential of PVA composites with SCB and/or banana waste to be used as biodegradable and sustainable alternatives to current packaging materials.

5. Author's Contribution

- **Conceptualization** – Thiago Tôrres Matta Neves; Simone Taguchi Borges; Edla Maria Bezerra Lima; Cristiane Hess de Azevedo Meleiro; Antonieta Middea; Renata Nunes Oliveira.
- **Data curation** – Ana Paula Duarte Moreira.
- **Formal analysis** – Thiago Tôrres Matta Neves; Ana Paula Duarte Moreira; Antonieta Middea.
- **Funding acquisition** – Renata Nunes Oliveira.
- **Investigation** – Thiago Tôrres Matta Neves.
- **Methodology** – Thiago Tôrres Matta Neves; Cristiane Hess de Azevedo Meleiro; Antonieta Middea.
- **Project administration** – NA.
- **Resources** – Renata Nunes Oliveira.

- **Software** – NA.
- **Supervision** – Simone Taguchi Borges; Edla Maria Bezerra Lima; Renata Nunes Oliveira.
- **Validation** – Simone Taguchi Borges; Luiz Antonio Borges Junior; Edla Maria Bezerra Lima; Renata Nunes Oliveira.
- **Visualization** – Luiz Antonio Borges Junior; Edla Maria Bezerra Lima; Antonieta Middea; Renata Nunes Oliveira.
- **Writing – original draft** – Thiago Tôrres Matta Neves.
- **Writing – review & editing** – Thiago Tôrres Matta Neves; Ana Paula Duarte Moreira; Renata Nunes Oliveira.

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