BIODEGRADABILITY STARCH-BASED FILMS FROM DIFFERENT SOURCES: INFLUENCE OF EDAPHIC FACTORS

BIODEGRADABILIDADE DE FILMES À BASE DE AMIDO DE DIFERENTES ORIGENS: INFLUÊNCIA DE FATORES EDÁFICOS

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Abstract: Biodegradability determines how long the film can be fully degraded in the environment. However, current methodologies require time and high cost and ignore the interference of the soil. Therefore, this work aimed to evaluate the effect of the edaphic and starch factors on elaborated films, and the introduction of image analysis as a tool to measure the biodegradability of films. Starches from *parreira-do-mato* roots, wolf-fruit, butterfly lily rhizomes, cassava, corn, yam, and glycerol were used to prepare starch films by means of the casting technique. From the films were determined their solubility and biodegradation. The biodegradation tests were carried out at random, in a 6 x 5 factorial scheme, in triplicate. The factors being the type of starch used to prepare the films and the type of soil used for the biodegradation process. The yam and cassava starch films were the ones that were more susceptible to biodegradability, while the butterfly lily and *parreira-do-mato* starch films showed better performance, resisting the biodegradation process for a longer time. Loose textured soil was the most favorable environment for the occurrence of biodegradability. In view of this study, it was verified that the degraded area was a good indicator of biodegradability and the attributes of the soil together with the type of starch showed a clear interference in the evolution of biodegradation of the starch films.
Keywords: Biodegradable films; Soil properties; Image analysis.

Resumo: A biodegradabilidade determina por quanto tempo o filme pode ser totalmente degradado no ambiente. No entanto, as metodologias atuais demandam tempo e alto custo e ignoram a interferência do solo. Portanto,

este trabalho teve como objetivo avaliar o efeito dos fatores edáfico e amido em filmes elaborados e a introdução da análise de imagem como ferramenta para medir a biodegradabilidade de filmes. Amidos de raízes de parreira-do-mato, fruta-lobo, rizomas de lírio-do-brejo, mandioca, milho, inhame e glicerol foram utilizados para preparar filmes de amido por meio da técnica de casting. A partir dos filmes foi determinada sua solubilidade e biodegradação. Os testes de biodegradação foram realizados aleatoriamente, em esquema fatorial 6 x 5, em triplicata. Os fatores foram o tipo de amido utilizado para preparar os filmes e o tipo de solo utilizado para o processo de biodegradação. Os filmes de fécula de inhame e mandioca foram os mais suscetíveis à biodegradabilidade, enquanto os filmes de amido de lírio-do-brejo e parreira-do-mato apresentaram melhor desempenho, resistindo ao processo de biodegradação por mais tempo. O solo de textura franca foi o ambiente mais favorável para a ocorrência de biodegradabilidade. Diante deste estudo, verificou-se que a área degradada foi um bom indicador de biodegradabilidade e os atributos do solo juntamente com o tipo de amido mostraram uma clara interferência na evolução da biodegradação dos filmes de amido.
Palavras-chaves: Filmes biodegradáveis; Propriedades do solo; Análise de imagem.

Introduction

Within the prevailing accelerated pace of illegal logging in the Amazon and other regions, the world is experiencing irreparable losses in its biome, indulging deforested areas to cattle ranching and monoculture. However, part of the issue can be reversed by making use of the Brazilian biome for the exploration of plant species that contain high technological and economic potential in favor of family farming through extractive and self-sustainable practices, without degradation of the environment.

Among the plant species that can be explored are the wolf-fruit (*Solanum lycocarpum*), the roots of the bush wine (*Cissus simsiana Schult. and Schult. f.*), and the rhizomes of the butterfly lily (*Hedychium coronarium*), which contain easily extractable starches (ASCHERI et al., 2010; ASCHERI et al., 2014). These kinds of starches can have great potential for use in the elaboration of biodegradable films and can compete with the major starches traditionally commercialized such as corn and cassava starches, which are highly sought after in commerce, mostly in the food industry and other industrial sectors, which make them expensive raw materials for film production.

It is important to highlight that, starches from different botanical sources have different functional properties and they can influence the formation of films and their biodegradation. In addition, other factors can influence the biodegradability of the films, such as the type of soil.

In Brazil, several types of soil are found (latosols, organosols, argisols, neosols, etc.) (EMBRAPA, 2018) which differ from one another by aspects related to their structure, fertility, origin, depth, permeability, presence or not of nutrients, among others. The soil effect on a variable is called the edaphic factor effects. The edaphic factors are the soil properties that affect the diversity of organisms living in the soil environment. These include soil structure, temperature, pH, and salinity (FURTAK; GALAZKA, 2019).

Other physical and physical-chemical aspects such as granulometry, organic substance content, pH and porosity, as well as environmental conditions, can interfere with the biodegradability of the film (FRANCHETTI; MARCONATO, 2006).

The methodologies used to determine biodegradability are linked to the quantification of carbon dioxide released (ISROI; SYAMSU, 2018), and proliferation rate of microorganisms and respirometry. Furthermore, all studies related to biodegradation emphasize the evaluation methodology using simulated soil, that is, with a defined percentage of organic substance, sand, clay, and silt, monitoring pH and humidity (MARINHO et al., 2018). What makes the study limited to the laboratory environment since in nature there are different types of soils with different physical, physicochemical, and other characteristics.

The characteristics concerning soil types such as pH, temperature and humidity must also be considered in the biodegradation of the films (KALE et al., 2007). These and other features require periodic monitoring, longer time and high cost to obtain results on the biodegradation variable. However, there are more sophisticated tools that can monitor the biodegradation of films in less time and cost, including image analysis.

Therefore, it is essential to correlate the various chemical, physical and biological factors of the soil, which can influence the biodegradation of starch films, acquiring this information through data analysis, using image processing as an essential tool. in the monitoring of biodegradability that will contribute to the elucidation of the biodegradation process in natural soils, a subject underexplored.

For this reason, the present work aimed to evaluate the effect of the edaphic and starch factors on elaborated films through image analysis as a tool to measure the biodegradability of films.

Material and Methods

The experiments were carried out on the premises of the Exact and Technological Sciences Campus of Anápolis – Henrique Santillo (Anápolis-GO).

Corn and cassava starches were purchased at the local market in the city of Anápolis. wolf-fruit, yam, butterfly lily, and *parreira-do-mato* starches were extracted according to the methodology described by Ascheri et al. (2010).

Five soil samples were collected at different points in the municipality of Gameleira de Goiás (Goiás, Brazil) and sent to the soil analysis laboratory where performed physical (granulometry, texture, porosity, degree of saturation, and humidity) and chemical (pH, phosphorus, aluminum, cation exchange capacity, organic matter, and base saturation) analysis according to the methodology by Teixeira et al. (2017), and microbiological analysis: Nitrogen from Microbial Biomass and Carbon from Microbial Biomass according to the fumigation-extraction method by Vance, Brookes and Jenkinson (1987), and Basal Soil Respiration, Organic Carbon, Soil Microbial Quotient and Quotient soil metabolism according to the methodology by Islam and Weil (2000).

The films were made by preparing filmogenic solution of starch (2% w/v), added with 0.8 mL of glycerol. The filmogenic solutions were heated at 90 °C for 5 min. After starch gelatinization, 25 ml of filmogenic solution were transferred to an 8 cm diameter polyethylene Petri plate and dried at 30 °C for 12 h.

The thickness of the films was measured with an external hand micrometer (ISOMASTER®, Tesa, Swiss) with an accuracy of 0.01 mm. The water solubility of the films was determined according to the methodology proposed by Gontard et al. (1994).

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The biodegradation analysis of the films was performed randomly in a 6 x 5 full factorial arrangement, the first factor being the type of starch (wolf-fruit, yam, butterfly lily, cassava, corn, and *parreira-do-mato* starches). The second factor was the type of soil (S1, S2, S3, S4, S5). Each treatment was applied in triplicate, totaling 90 experimental units.

The soils used were sifted in a sieve with 4 mesh holes. The starch films cut 2 cm on a side, previously dried and packed in polyethylene nets were buried in approximately 100 g of soil contained in disposable 250 ml transparent polyethylene containers. Then, the pots were stored in a BOD incubator at 30 °C, with a humidity of around 40%.

Therefore, previous studies were carried out, in which the soils were moistened to approximately 40% humidity with distilled water and stored in the BOD (under the same temperature and humidity conditions described above) for 2 days. Right after the storage, soil moisture contents were determined in an oven at 105 °C (AOAC, 2005), resulting in a reduction of up to 15% in moisture, approximately.

Biodegradability was evaluated every 8 days up to 40 days of analysis, removing the films from the containers and carefully removing the excess soil with the aid of a brush. Then, both films were placed on a sheet of paper with a white background, and the images of the 8 and 40 days were recorded with a Sony Cyber-shot 12.1-megapixel camera, with a standardized height of 10 cm. A measuring tape was placed on the sheet, used as a standard scale in the ImageJ software (National Institute of Health, 2019) and the distance in pixels was calculated. After image processing, with the acquired data, the percentages of the area of the films [Bio (%)] were calculated:

$$\text{Bio}\,(\%) = 100 \times \frac{A_{\text{Total}} - A_{\text{Degraded}}}{A_{\text{Total}}} \qquad (1)$$

Which: A_{Total} and A_{Degraded} are non-biodegraded and biodegraded film area in a given storage time in the BOD incubator, respectively.

The data were subjected to analysis of variance, the means were compared using the Scott and Knott test, and regression was analyzed using the Least Squares Method, at the level of 5% probability, using the Sisvar 5.7 software (FERREIRA, 2018).

Results and Discussion

Characterization of starch films

Films of wolffruit, butterfly lily, cassava, corn and *parreira-do-mato* starches (Figures 1A, 1C, 1D, 1E and 1F, respectively) showed a more homogeneous, transparent and flexible appearance.

More opaque films formed by the yam starch, with a lumpy and crumbly appearance, probably the heating time used was not enough to break the starch granules that were characteristic of its gelatinization. Its was also observed by Narvaez-Gomez et al. (2021) showing that the agglomeration of yam starch provided greater opacity and reduced film elasticity.
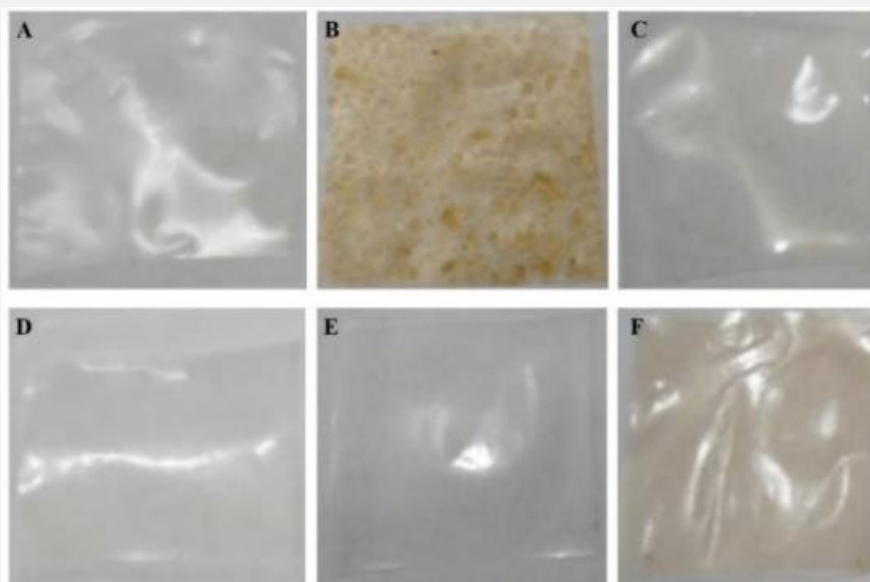

Figure 1: Films made from filmogenic solution with: A) wolf-fruit, B) yam, C) butterfly lily, D) cassava, E) corn, and F) *parreira-do-mato* starches. Fonte: Autores, 2023.

Parreira-do-mato starch formed films with a homogeneous appearance and flexible reddish color (Figure 1F) due to the presence of carotenoids (3.12 10-3 mg/g, ASCHERI et al., 2014).

The thickness of the films ranged from 0.06 to 0.12 mm (Table 1). This parameter influences the properties of the films. In addition to being directly related to solubility, thickness also influences mechanical and water steam barrier properties (FERNANDES et al., 2019).

According to Wang et al. (2017), the increase in thickness is mainly due to the greater amount of mass that, with a greater number of granules, gives a greater surface area, improving the interaction with the plasticizer. However, in the present study it has been observed that the increase in thickness was due to the size of the starch granules. Wolf-fruit, cassava, and corn starches contain relatively small granules between 0-27.3 µm (ASCHERI; PEREIRA; BASTOS, 2014), 1–10 µm (Falade; Ibanga-Bamijoko; Ayetigbo, 2019) and 13.3-21.4 µm (Bustillos-Rodríguez et al., 2019), respectively, and formed thinner films compared to yam, butterfly lily and *parreira-do-mato* films. (Table 1).

Table 1: Mean values ± standard deviation of thickness and solubility of films made from wolf-fruit, yam, butterfly lily, cassava, corn and *parreira-do-mato* (P-M).

Variable	Starch					
	Wolf-fruit	Yam	Butterfly lily	Cassava	Corn	*P-M*
Thickness (mm)	0.08±0.01	0.11±0.02	0.12±0.01	0.06±0.01	0.08±0.01	0.12±0.01
Solubility (%)	54.10±2.53	72.64±4.87	20.01±3.21	72.49±5.59	24.18±2.15	13.29±2.59

Fonte: Autores, 2023.

Butterfly lily and *parreira-do-mato* starches formed thicker films due to the size of their relatively large granules of 23.6-38.2 µm (ASCHERI et al., 2010) and 60 µm (ASCHERI et al., 2014), respectively, while the average size of yam starch granules was approximately 23 µm (ANDRADE; BARBOSA; PEREIRA, 2017). The relationship between the thickness and the size of the starch granules was better evidenced in Figure 2.

The solubility values of the films decreased with the increase in the size of the granules (Figure 2). The butterfly lily, *parreira-do-mato* and corn films showed the lowest solubility values, while the yam, cassava and wolffruit films were more soluble in water, indicating that these types of films can be easily biodegraded. The yam starch film presented an atypical case, the solubility value was higher than the other films (Figure 2). This was due to the presence of lumps, as shown in Figure 1B. Part of this solubility must also be attributed to the plasticizer used, glycerol, due to its hydrophilic character (BALLESTEROS-MÁRTINEZ; PÉREZ-CERVERA; ANDRADE-PIZARRO, 2020).
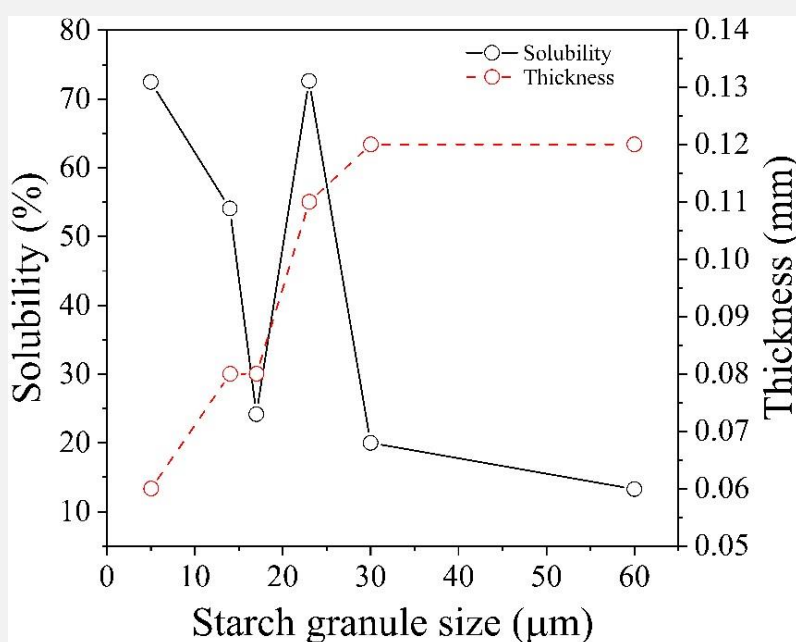

Figure 2: Variation of the average values of thickness and solubility of the films produced based on different types of starch, as a function of the size of the starch granules of wolffruit, yam, butterfly lily, cassava, corn and *parreira-do-mato*. Fonte: Autores, 2023.

Film biodegradation process

Biodegradability is often measured by gravimetric methods, in which the mass of the film is recorded at time intervals. If this type of test is applied to soils, they are adhered to the films and must be carefully removed before each weighing to ensure the integrity of the film and the removal of foreign substances, avoiding large measurement errors. For this reason, the

evaluation of the degraded area of the films by means of photographic monitoring followed by image processing is presented as a good alternative for the quantification of biodegradation. The photographic images of the films recorded on the eighth and fortieth days of biodegradation are shown in Figure 3.

After the eighth day, the films showed changes in color, tone and physical integrity due to their hydrophilic nature, suggesting the beginning of the biodegradation process. Films of wolf fruit, butterfly lily, cassava, corn, and *parreira-do-mato* starches submitted to S1 to S4 soils showed a rough appearance due to water adsorption. However, this absorbed water was not enough for the proliferation of microorganisms, despite having the films as a carbon source for their metabolism (PRAKASH et al., 2014) and starting to degrade, generating an increase in the biodegraded area.
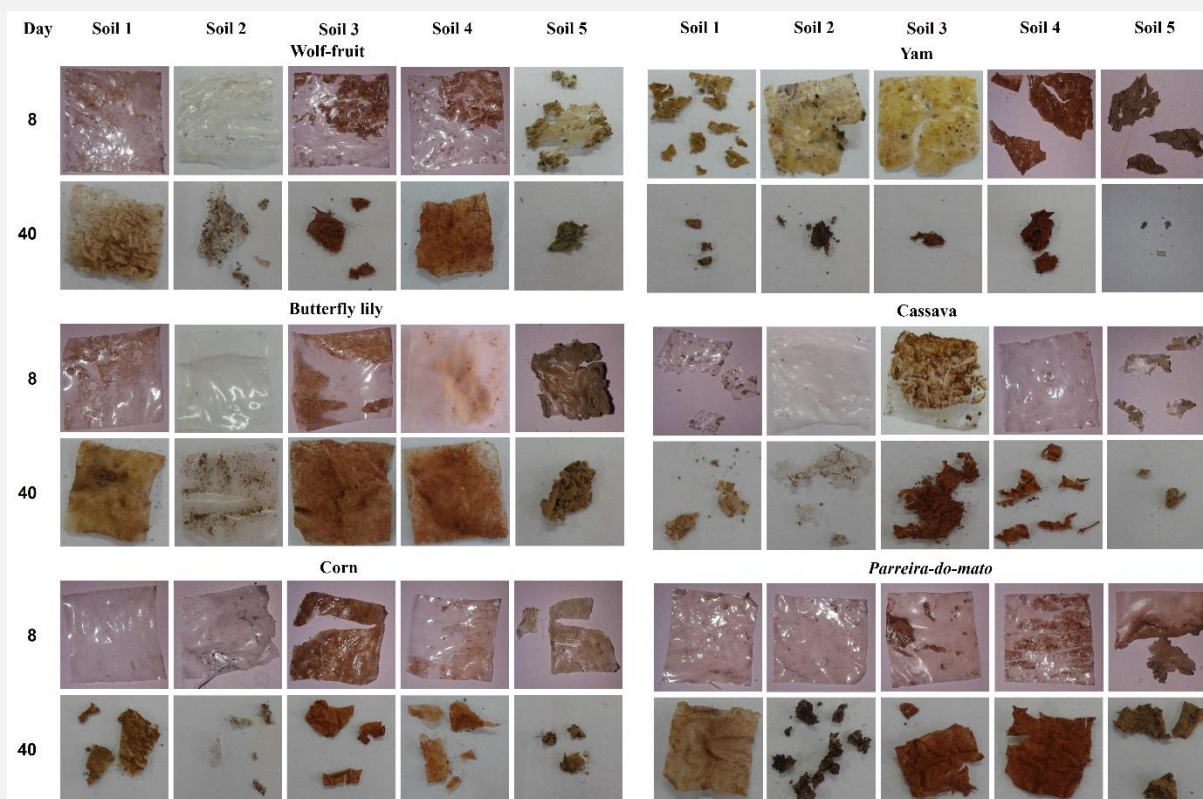

Figure 3: Photographs of the starch films made from wolf fruit, yam, butterfly lily, cassava, corn, and parreira-do-mato starch obtained after the eighth and fortieth day of biodegradation carried out in the different soils used. Fonte: Autores, 2023.

It should be taken into consideration that the wrinkling of the film has not yet consisted of an area of biodegradation, but an area of deformation, as it alters the original appearance and the physical structure that can affect the strength and elasticity of the film, leading to fragmentation. Fragmentation can be seen from the eighth day of biodegradation in yam films in soils S1 to S5 and, for the other films, it can be seen in soil S5 (Figure 3).

The films, by absorbing water, become more viscous and the soil begins to adhere, causing physical changes, increasing susceptibility to attack by microorganisms and mechanical breakdown. This phenomenon occurred more quickly in the films that were subjected to S5 soil, reaching a rapid increase in its biodegraded area (Figure 3).

Using the appropriate commands of the ImageJ software, it was possible to increase the photographic image, without loss of optical characters, and to calculate the biodegraded area and biodegradation as a function of time through equation 1. The results are shown in Figure 4, which shows the linear and positive trend of biodegradation.

The linear model was well suited to the experimental data, presenting values of coefficients of determination between $0.80 \leq R2 \leq 0.99$, in which angular coefficients represented the biodegradation rates of the films. The yam films acquired higher biodegradation rates, indicating rapid degradation in soils S1 and S3 to S5 (except in S2). On the other hand, *parreira-do-mato* films degraded slowlier in soils S1 to S3.

All films had different behavior in each of the studied soils, indicating that biodegradation depends on the type of starch and the type of soil applied. This behavior is confirmed by means of ANOVA applied to the final results obtained on the fortieth day of biodegradation (Table 2).
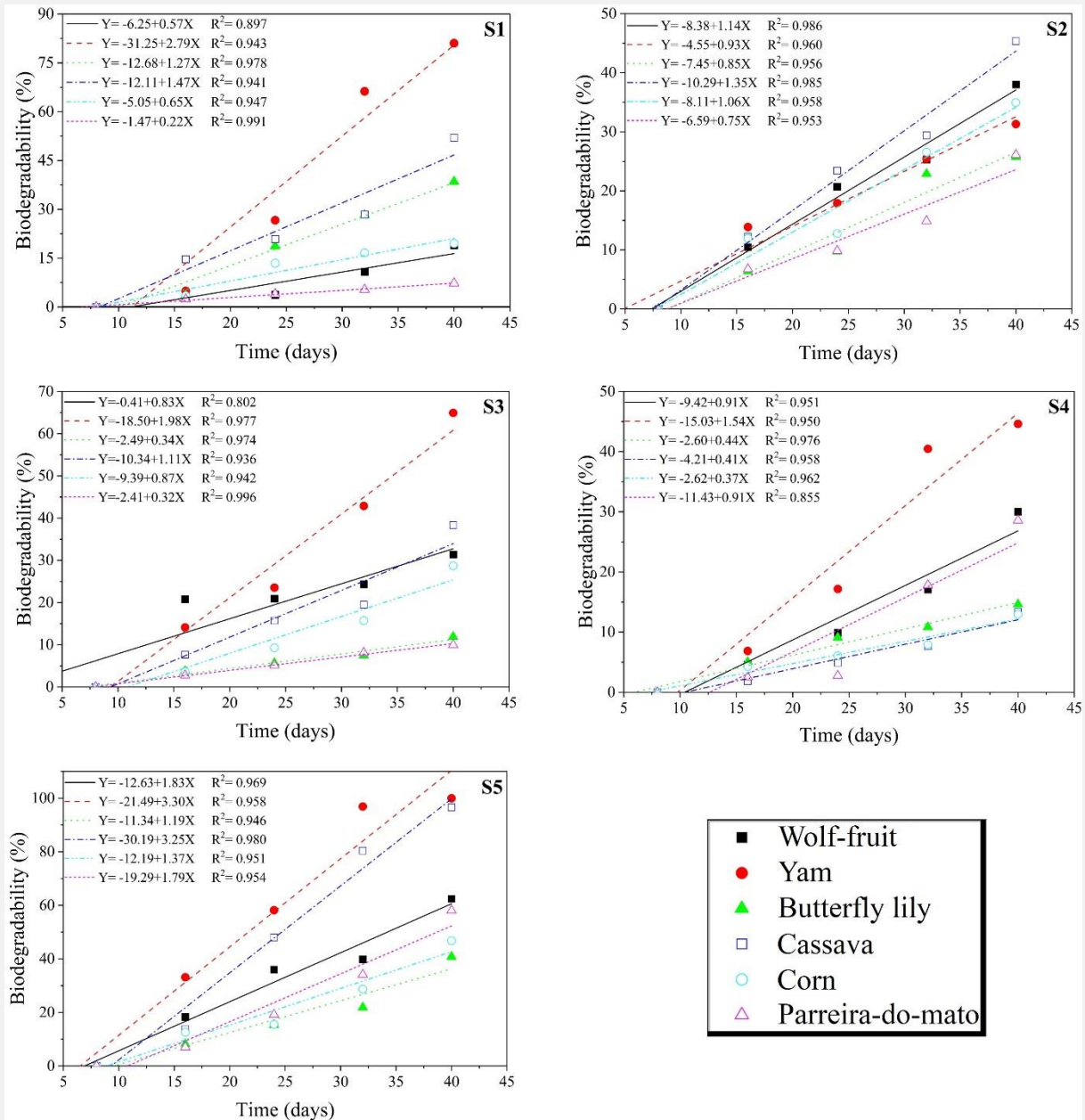
Figure 4: Biodegradability of wolf-fruit, yam, butterfly lily, cassava, corn, and *parreira-do-mato* films starch in different soils, as a function of biodegradation time. Fonte: Autores, 2023.

Table 2: Average values on the Biodegradability of starch-based films in different types of soils and results of the analysis of variance.

Type of starch	Type of soil				
	S1	S2	S3	S4	S5
Wolf-fruit	18.9 [cD]	38.0 [bA]	31.2 [bB]	30.0 [bB]	81,1 [aB]
Yam	81.0 [bA]	31.3 [eB]	64.9 [cA]	44.6 [dA]	100,0 [aA]
Butterfly lily	38.6 [aC]	25.7 [bB]	11.9 [bC]	14.6 [bC]	40,8 [aD]
Cassava	52.0 [bB]	45.4 [bA]	38.4 [bB]	13.4 [cC]	96,6 [cA]
Corn	19.5 [cD]	35.0 [bA]	28.7 [bB]	13.0 [cC]	46,8 [aD]
Parreira-do-mato	6.7 [cD]	26.2 [bB]	10.0 [cC]	29.0 [bB]	58,1 [aC]

Source	Degrees of Freedom	Sum of squares	Mean Squares	F Test
Starch	5	17789.9	3558.0	56.30[**]
Soil	4	23798.6	5949.7	94.15**
Starch x Soil	20	12459.1	623.0	9.86**
Residual	60	3791.6	63.2	--

*Average of three repetitions. ** Significant ($p\text{-}value < 0.015$). [ns] No significant.

Averages followed by lowercase letters in the rows and uppercase letters in the columns do not differ statistically by the Scott and Knott test at the 5% probability level. Fonte: Autores, 2023.

The starch x soil interaction shows that the changes in the biodegradation process were recurrent from the simultaneous action of these factors, that is, the effect that soil characteristics exert on the biodegradation of the films depends on the type of starch used, and vice versa.

When unfolding the starch x soil interaction, it was observed that the edaphic factor has a very important role in the biodegradation of starch films. The highest percentages of biodegradation were obtained by fixing the soil factor within the yam, cassava and wolffruit starches (Table 2). The lowest percentages of biodegradation were found in the soils within the butterfly lily, corn and *parreira-do-mato* starches.

Soil S5 was able to degrade 100% the initial area of the yam film, followed by soils S1, S3, S4 and S2 with biodegradation percentages of 81.0, 64.9, 44.6 and 31.3%. In the cassava starch film, soil S5 degraded 96.6% if its initial area and soils S1 to S4 were able to degrade 52.0, 45.4, 38.4 and 13.4%, respectively.

In the other films, it was observed that the highest percentage of the initial area was degraded by the S5 soil classified as loam soil due to its high percentage of fine sand (45.9%, Table 3), forming a massive structure that allowed the absorption of moisture and adhesion to the surface of the films.

In addition to sand, in S5 soil there is a predominance of macropores (0.52) that are responsible for aeration and for the greater contribution of water infiltration into the soil, providing an exchange of moisture and gases between the biodegradable films and the atmosphere (GANOT; DAHLKE, 2021).

Furthermore, other edaphic factors contributed to the effect of S5 soil on film biodegradation. The pH of 5.8 influenced the solubility of nutrients and many of the chemical transformations in the soil (Cardoso; Andreote, 2016). At high acidity, there are limitations in the availability of Ca, Mg and K and favors the availability of phytotoxic Al^{3+} at pH lower than 5.6 (BATISTA et al., 2018). Silva et al. (2016) stated that in soils with pH values between 5.6 and 6.2 aluminum precipitates and most nutrients are in the soluble form, and Rosa et al. (2015) reported that aluminum availability is inversely correlated with soil microbiological activity.

The cation exchange capacity of S5 soil was 5.5 cmol dm^{-3} (Table 3). This attribute represented the total amount of cations retained in the soil in exchangeable condition (Ca^{2+} + Mg^{2+} + K^{+} + H^{+} + $Al^{3+)}$ (Ronquim, 2010) is related to soil fertility providing nutrients and also to the activity of microorganisms responsible for the decomposition of organic substances (ZHENG et al., 2019).

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Table 3: Result of granulometric analysis and textural classification of soils.

Attribute		Soil				
		S1	S2	S3	S4	S5
Granulometry (%)	sand	18.8	36.0	31.1	36.9	45.9
	Clay	53.0	21.1	20.1	8.3	14.0
	Silt	28.8	42.9	48.8	54.8	40.1
Texture		Clay	Loam	Loam	Silty loam	Loam
Porosity		0.55	0.58	0.58	0.48	0.52
Degree of saturation		39.3	40.1	33.3	30.9	33.7
Umidity (%)		16.87	41.33	35.52	15.68	8.21
pH		4.5	4.8	4.6	4.7	5.8
CEC^1 (cmol dm^{-3})		4.6	11.2	9.6	4.6	5.5
Phosphorus (mg dm^{-3})		1	1	3	3	23
Aluminum (cmol dm^{-3})		0.1	0.1	0.2	0.1	0.0
Organic matter (%)		1.2	2.7	2.0	1.0	1.3
Base saturation (V%)		23	48	43	38	79
MBC (mgC kg^{-1}solo)^2		0.001	254.5	242.4	266.7	206.1
Basal respiration (mgC-$CO_2$$kg^{-1}$ solo h^{-1})		0.06	0.37	0.12	0.01	0.06
Microbial Biom. N (mgN kg^{-1}solo)		1.3	10.4	22.0	25.9	14.3
Total organic carbon (%)		0.8	3.5	1.4	0.1	0.5
qCO_2 (mgC-CO_2 mg^{-1}C-BM h^{-1})^3		686.6	$1\ 10^{-3}$	$5\ 10^{-4}$	$4\ 10^{-5}$	$3\ 10^{-4}$
Soil Microbial Quotient (%)		$12\ 10^{-5}$	72.7	176.9	5333.3	404.0

[1] Cation exchange capacity. [2] Microbial Biomass Carbon. [3] Quotient soil metabolism. Fonte: Autores, 2023.

The high content of phosphorus (23 mg dm^{-3}), absence of aluminum and low levels of organic substance (1.3%) seen in Table 3, also favor the development of microorganisms.

The microbiological attributes of the S5 soil (Table 3) confirm contributions of the physicochemical attributes in the deterioration process of the films studied. The microbial activity in S5 soil was confirmed by the considerable amount of carbon in the microbial biomass (206.1 mgC kg^{-1}soil) that favors the establishment of microorganisms in the soil (GUIMARÃES et al., 2017), relating well to the results of basal respiration of soil (0.06 mg of CO_2 kg^{-1} soil h^{-1}), total organic carbon (0.5%), microbial quotient (404.0%) and with the metabolic quotient (3.10^{-4} mgC-CO_2 mg^{-1}C-BM h^{-1}).

Table 3 shows that soils S1 and S3 classified as clayey and loam soils, respectively, and soil S5 were more favorable for the biodegradation of films made with yam and cassava starch, presenting biodegradation percentages between 52-81%, 38-65%, and 96-100%, respectively. Like the S5 soil, the S1 soil presented a wetter and stickier texture due to the large amount of clay (53%, Table 3) that easily adhered to the surfaces of the films.

Soils S3 and S4 showed lower biodegradation power. The butterfly lily and *parreira-do-mato* starch films and the cassava and corn films had the lowest percentage of biodegradation between 10-12% in soils S3 and S4. These soils had the highest levels of silt (48.8-54.8%), high levels of carbon in the microbial biomass (242.4 and 266.7 mgC kg^{-1} soil, respectively), nitrogen in the microbial biomass (22.0 and 25.9 mgC-CO_2 kg^{-1} soil h^{-1}, respectively), lower humidity values (35.52 and 15.68%, respectively), degree of saturation (33.3 and 30.9, respectively) and the same phosphorus content of 3 mg dm^{-3}.

The probable cause of S3 and S4 soils being less favorable environments for the conditioning of the biodegradation process is related to the nutrient imbalance that may interfere with absorption, since the excess or lack of a certain element may limit the absorption of the others, causing a breakdown in the microbiological activity of the soil, limiting or inhibiting the biological functions of microorganisms.

Other edaphic factors to take into account are the humidity and the degree of soil saturation, as environments with lower levels of these two factors determined the delay in the biodegradation of the films, showing that water is an extremely important factor for the

acceleration of the activity of biodegradation, therefore, the variation in the consistency of the soil occurs due to the influence of the forces of adhesion and cohesion.

Soils S2 and S3 were expected to contain higher levels of organic substances (2.7 and 2.0%, respectively) and with higher values of organic carbon (3.5 and 1.4%, respectively) and basal respiration (0.37 and 0.12 $mgC\text{-}CO_2 kg^{-1}$) soil h^{-1}, respectively), were the environments of greater biodegradation, however, the quotient of microbial activity remained stable (1.10-3 and 5.10^{-4} $mgC\text{-}CO_2$ $mg^{-1}C\text{-}BM$ h^{-1}, respectively), which means that in the evaluation of the biodegradation of the films, not only must the microbial activity be indicated, but also the set of physical, chemical and biological properties of the soil, called the edaphic factor effect, which interferes in the degradation rate.

Hence, the effect of the soil edaphic factor on biodegradation depends on the effect of the starch factor (Table 2). Fixing the starch factor in each soil studied, it was noted that these starches behave differently in each soil used, confirming once again that starches extracted from different plant sources come up with different physical, physicochemical and functional properties (ANDRADE; BARBOSA; PEREIRA, 2017) and, in this case, they produce films with different physicochemical and biodegradation properties.

The effect of starch on film properties is mainly related to the rearrangement of its solubilized polymers during the preparation of the film-forming solution, that is, during starch gelatinization at 90 °C for 5 min. During gelatinization, the viscosity of the film-forming solution increased and, after cooling and drying at 30 °C, the formation of the film (THOMAS; ATWELL, 1999). At this stage, retrogradation occurred which, according to Bemiller and Whistler (2009), was due to the reassociation of some amylose and amylopectin polymers, but the intensity of this process depends on the type of starch used.

According to Denardin and Silva (2022), retrogradation is basically a process of crystallization of starch molecules that occurs due to the strong tendency to form hydrogen bonds between adjacent molecules. Not only can this phenomenon considerably affect its mechanical properties, but also its barrier ability. High retrogradation produces more rigid films, more resistant to fracture, but less elastic, with lower oxygen permeability and greater

water absorption capacity (CANO et al., 2014), the latter was shown through the solubility of the films (Table 1). And, the solubility of the films correlated well with the size of the starch granules.

In fact, the variability of the solubility of the films attributed to the starches used was positively related to the biodegradation of the films (Figure 5) in the different soils studied. Higher correlations were obtained in soils S5, S3 and S1, followed by soils S2 and S4 with r and p-values of 0.946 and 0.004, 0.861 and 0.028, 0.745 and 0.089, 0.694 and 0.126 and 0.373 and 0.466, respectively.
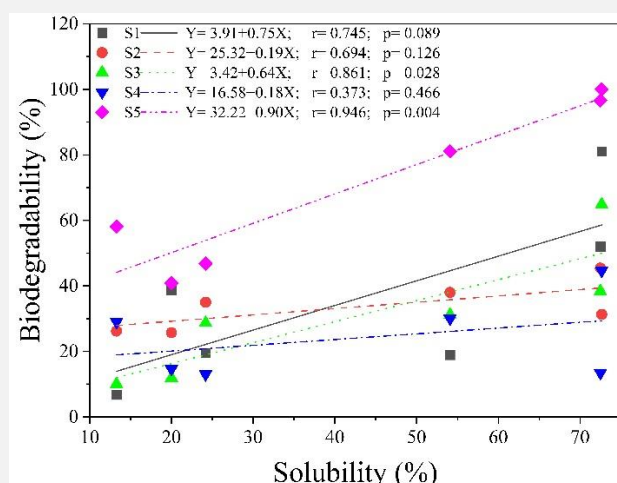

Figure 5: Variability of biodegradation as a function of the solubility of films made with wolf fruit, yam, butterfly lily, cassava, corn and *parreira-do-mato* starches. Fonte: Autores, 2023.

Films made with yam and cassava had the highest percentages of water solubility (72.64 and 72.49%, respectively seen in Table 1) and also showed the highest percentages of biodegradation, being statistically equal only in soils S2 and S5 (Table 2). While the butterfly lily and *parreira-do-mato* films showed the lowest biodegradation percentages, with only significant differences occurring in soils S1 and S3 (Table 2).

With these results, it can be inferred that it is important to analyze the biodegradation process through photographs and image analysis in order to study, mainly, physical phenomena such as film appearance, initial and final biodegradation time, wrinkling and soil adhesion. in the movies, microbial attack, disintegration etc. However, the biodegradation

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process of the films made with the studied starches can be replaced with the study of the water solubility of the films, nevertheless, in soils S1, S3 and S5, that is, in soils that can absorb water which is very important for the study of solubility and biodegradation of films.

Conclusions

The edaphic effect and starch properties had a significant influence on the starch films biodegradability. Soils with medium acidity, high phosphorus content and loam soil texture are more favorable environments for the biodegradation. The yam and cassava starch films were more susceptible than butterfly lily and parreira-do-mato starch films that resisted the biodegradation process for a longer time. The Image analysis with ImageJ software by measuring the biodegraded area proved to be a very effective tool for measuring biodegradation.

Acknowledgments

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