

Physicochemical Potential Of Biodegradable Packaging Biofilms With Starch And Plant Fiber Matrix

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Abstract

Background: Non-biodegradable plastic waste poses a risk to human health, animal health, the environment, and the economy. The aim of this study was to contribute to reducing environmental pollution by developing biodegradable packaging biofilms based on local materials.

Materials and Methods: Various powders of *T. daniellii*, *M. paradisiaca*, and starch were obtained from plant material by drying, grinding, and sieving. Reference methods were used to formulate biodegradable packaging biofilms and determine their mechanical and physicochemical properties, including friability, opacity, water absorption, moisture content, and pH.

Results: In order of increasing importance, the friability of fiber-supplemented packaging biofilms ranged from 20 to 50 min (biofilms supplemented with *M. paradisiaca* and *T. daniellii*) and from 15 to 45 min (biofilms supplemented with *T. daniellii*) between 60°C and 120°C. Packaging biofilms supplemented only with *T. daniellii* fibers had the best opacity scores at wavelengths DO330 (0.52 ± 0.01), DO500 (0.37 ± 0.01), and DO820 (0.64 ± 0.05). On average, packaging biofilms supplemented with *T. daniellii* fibers and *M. paradisiaca* fibers (2.46 g) absorbed less water than those formulated with *T. daniellii* (6.27 g) and *M. paradisiaca* (5.27 g) alone. With pH values ranging from 6.79 to 7.44, the moisture content of fiber-supplemented packaging biofilms was 47.8% (*T. daniellii* + *M. paradisiaca*); 52.6% (*M. paradisiaca*) and 61.6% (*T. daniellii*).

Conclusion: The toxicological study could assess the biotechnological potential of these biofilms in the design of biodegradable, photoprotective, and environmentally friendly packaging.

Keywords : Packaging biofilms ; biodegradable ; fibers, *T. daniellii* ; *M. paradisiaca* ; starch.

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I. Introduction

Plastic pollution is a major global environmental challenge. In West African coastal areas, nearly 80% of non-biodegradable plastic waste is poorly managed¹. Due to a lack of adequate resources and infrastructure for treating this waste, developing countries are particularly affected by this type of pollution as a result of insufficient plastic waste management¹.

In addition, the degradation of these petrochemical-based plastic packaging materials takes a very long time. It can range from several decades to several hundred years, or even millennia, depending on the type of plastic and environmental conditions². Conventional plastics, such as petrochemical polymers, take between 500 and 1,000 years to biodegrade³.

As a result, this plastic waste clogs pipes and promotes the spread of diseases such as malaria, typhoid fever, and cholera⁴. In addition, this plastic waste has caused various types of pollution affecting not only the soil, water, and air, but also living beings, thereby compromising biodiversity and human health⁴. According to

research by Kanatt et al.⁵, untreated plastic waste contributes to global warming through greenhouse gas emissions estimated at 1.8 billion tons in 2019, or 3.4% of global emissions.

In order to reduce the production and proliferation of synthetic plastic packaging, large companies such as Unilever, Coca Cola, Ahlstrom-Munksjo, and the Ellen MacArthur Foundation's New Plastics Economy program have proposed several solutions such as recycling, reuse, and composting⁶.

Despite considerable efforts in this area, the situation has not shown significant improvement in Africa. Plastics are found everywhere: in homes, on roads, in drainage systems, markets, and other public places. Given the ineffectiveness of these solutions in reducing the proliferation of this waste, the use of bioplastics is being proposed as an alternative.

These biodegradable bioplastics or biofilms are made from materials that can be broken down by microorganisms such as bacteria, fungi, and algae. The most commonly used types of biodegradable packaging materials include starch, cane fiber, paper, cardboard, polylactic acid (PLA) bioplastics, and parts of plant species^{4,5}.

Among these natural biopolymers, starch is considered the most promising raw material for the development of new, more environmentally friendly packaging materials⁷. This is due to its low density, renewable nature, complete biodegradability, availability in various forms, and low cost.

The advantage of these biodegradable biofilms made from starch and/or plant leaves lies in their ability and flexibility to be used for food packaging, reducing carcinogenic and toxicological effects⁸. These natural starch-based biofilms, which have the comparative advantage of being biodegradable, could be a way to reduce pollution^{9,10}. Among these leaves, those of *Thaumatococcus daniellii*, *Musa paradisiaca*, *Zea mays*, and *Tectona grandis* are used in Côte d'Ivoire as packaging to protect various foodstuffs¹¹.

In Côte d'Ivoire, work has been carried out on the recovery and recycling of packaging using local materials^{12,13}. However, the formulation of biodegradable packaging materials remains a major challenge. The objective of this study is to contribute to the reduction of environmental pollution through the development of environmentally friendly biodegradable packaging biofilms based on starch supplemented with plant fibers.

II. Materials And Methods

Materials

The plant material consisted of fresh leaves from *Thaumatococcus daniellii* and *Musa paradisiaca* (figure 1). Starch extracted from one-year-old *Manihot esculenta* cassava tubers was also used as a base polymer matrix for biofilm production. In addition, water was used as a solvent and glycerol as a plasticizer in the production of packaging biofilms.

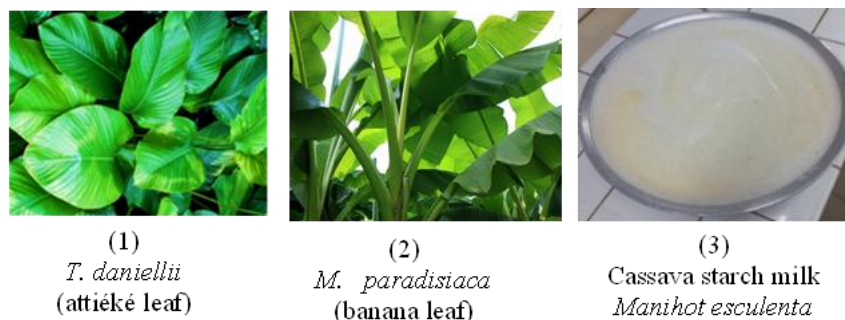


Figure 1 : Plant material

Methods

Sampling

Samples from two different batches of two kilograms (2) each, consisting of leaves from *Thaumatococcus daniellii* and *Musa paradisiaca*, were collected from the harvest fields. A mass of five kilograms (5) of *Manihot esculenta* cassava, one (1) year old, was also collected from the harvest fields. All these samples were stored in coolers and transported to the biotechnology laboratory for various tests to be carried out.

Criteria for selecting samples

Starch extracted from cassava is considered the most promising raw material for developing new, more environmentally friendly packaging materials^{14,15}. This is due to its low density, renewable nature, complete biodegradability, availability, and low cost. Among plant leaf species, the leaves of *Thaumatococcus daniellii* and *Musa paradisiaca* are the most widely used and fully characterized plant-based packaging materials in the agri-food industry in Côte d'Ivoire¹⁶.

Starch extraction

Once in the laboratory, the *Manihot esculenta* cassava was peeled and the inner and outer skins were removed from the root. Next, all impurities were removed from the stripped root by washing it five (5) times with tap water and rinsing it with distilled water. The root was then reduced to a fine pulp by grating it with a grater. The pulp obtained was dissolved in water and filtered to remove fibers and other coarse debris from the starch milk. The starch milk obtained was then subjected to decantation (Figure 1). The solid phase obtained is dried in the shade at 37°C for 24 hours to prevent the starch from browning. The various starch lumps obtained from this operation are ground using a grinder or mixer and then sieved to produce a whitish starch powder. This whitish starch powder constitutes the starch extract.

Obtaining leaf powders

The two species of leaves, *Thaumatococcus daniellii* and *Musa paradisiaca*, were sorted to remove foreign matter. They were then washed three times with tap water. Each leaf was dried and stored in the laboratory at room temperature (25±2°C) for three weeks. For the formulation of packaging biofilms, the dried leaves were ground using a grinder (blender) and then sieved to a particle size of 2 mm¹⁷ (figure 2). The fine powder obtained was stored in airtight bottles and used as the leaf powder for the formulation of biofilms.

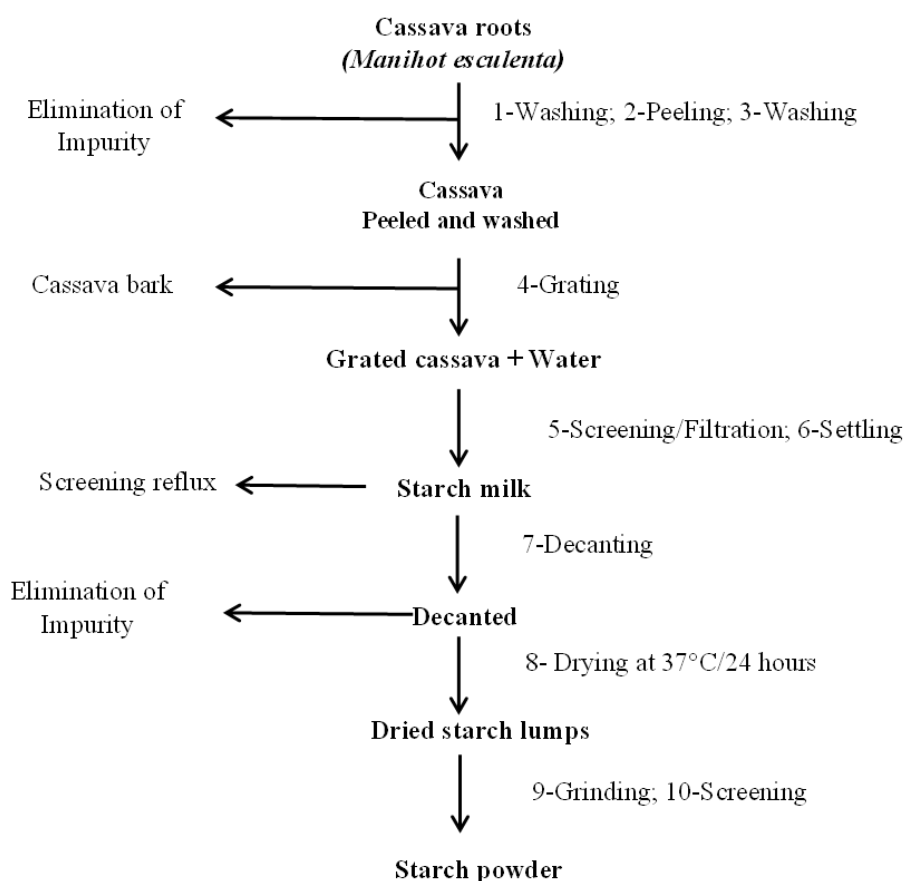


Figure 2 : Diagram of the steps involved in producing starch powder,

Formulation of biodegradable packaging biofilms

The biodegradable starch-based packaging biofilms supplemented with plant fibers were formulated according to the Delamarche method¹⁸ (2021). An initial formulation was made using only *Thaumatococcus daniellii* leaf powder; the second using *Musa paradisiaca* leaf powder; and the final formulation using both leaf powders simultaneously. Thus, 70 mL of distilled water was introduced into a 150 mL Erlenmeyer flask. To this volume contained in the Erlenmeyer flask, the starch powder, plant fiber powder, and glycerol were gradually added according to the proportions indicated in the table below. After homogenization, the mixture was heated gently on a hot plate at a moderate temperature between 45°C and 50°C. The mixture was stirred until a paste corresponding to the gelatinization temperature of starch, which is 72°C, was obtained. These different preparations were poured into plates or defined surfaces to form the various biodegradable biofilms (Table 1).

Table 1 : Different masses for the formulation of packaging biofilms

Products	Amount to be withdrawn
Starch	20 g
Leaves	5 g
Glycerol	3 mL
Distilled Water	70 mL

Physicochemical characterization of formulated packaging

Determination of the thickness of packaging biofilms

The thickness of the packaging biofilms was measured with a caliper in accordance with standard NF EN ISO 13385-1. This thickness, which also depends on proportions, was given by formula **no.1** defined according to the response surface design (Minitab Box-Bokhen design). The sensitivity of the packaging was also given according to the proportions using formula no.2. With **E**: thickness; **S**: sensitivity; **X**: sheet mass; **Y**: starch mass; and **Z**: glycerol mass in both formulas.

$$n^{\circ}1: E = 2,51 + 1,37x + 0,165y + 0,530z - 0,434x^2 - 0,0089y^2 - 0,0697z^2 - 0,0048 x.y - 0,005 x.z + 0,0007 y.z$$

$$n^{\circ}2: S = 1,44 - 0,382x + 0,158y - 0,870z - 0,042x^2 - 0,00124y^2 + 0,1085z^2 - 0,0038x.y + 0,173 x.z - 0,0283 y.z \%$$

Determination of the moisture content of packaging biofilms

The moisture content (**H**) of the packaging biofilms was determined using the **AOAC**¹⁹ method. A 5 g sample of each formulated packaging biofilm was weighed using a SARTORUIS BP 310S precision balance (Göttingen, West Germany) and this initial mass was recorded as (**mi**). This mass of packaging biofilms (5 g) was dried in a MEMERT oven (Schwabach, West Germany) at 105°C for 24 hours. Upon removal from the oven, the samples were cooled in a desiccator and weighed (**mf**); the moisture content was calculated using the following formula:

$$H(\%) = \frac{mi - mf}{mi} \times 100$$

pH determination

The pH of the packaging biofilms was determined by immersing the glass electrode of the pre-calibrated pH meter in 10 mL of supernatant. The supernatant was obtained after macerating 10 g of sample in 75 mL of distilled water. The pH value displayed on the pH meter screen was recorded for each sample²⁰.

Determination of the opacity of packaging biofilms

Opacity is a physical parameter that determines the relative permeability of packaging biofilms to monochromatic light radiation. Opacity is used to evaluate the photoprotective effect of packaging on packaged foodstuffs. High opacity in packaging provides better protection for foodstuffs against phytochemical deterioration¹³. It was determined by spectrophotometric measurements at wavelengths of: 330 nm (ultraviolet), 500 nm (infrared), and 820 nm (visible). These wavelengths were used to assess the spectrum range in which each packaging is most or least opaque.

To determine opacity, a volume of two (2 mL) of extract from packaging biofilms obtained after grinding each piece of packaging was diluted to 1/10th and 1/100th. The glass cuvettes, filled to 2/3 capacity, were placed in the receiving tube of the spectrophotometer, which had been set to the wavelengths (330 nm, 500 nm, and 820 nm), and the optical densities were determined against a blank or control. The monochromatic beam was emitted onto the sample as soon as the command was given, and the opacity (**OP**) was determined from the optical density (**OD**) such that **OD** = log (OP) with **OP** = 10^{OD}¹³.

Determination of the permeability of packaging biofilms

The water permeability (**Pe**) of packaging biofilms was determined in order to assess the level of protection these packaging materials provide to foodstuffs against moisture. The test consisted of measuring the amount of water that flowed through the packaging biofilms at 25°C after 4, 8, 16, and 24 hours of flow. Thus, a quantity of 5 mL of water was introduced into a filter container containing 25 cm² of packaging and equipped with a support for collecting the quantity of water drained over time, which made it possible to determine the water permeability¹³. Permeability was determined using the formula below. **Pe** : water flow rate (g.cm⁻².h⁻¹), **me**: mass of water flow (g), **Sf** : cross-sectional area of the packaging (cm²), **te**: time taken for water to flow through the sheet (**h**).

$$Pe = \frac{me}{Sf. te}$$

Determination of packaging fragility

Friability characterizes the ease with which packaging biofilms crumble under the effect of heat. The packaging biofilms were first cut and dried in a Pasteur oven at temperatures of 60, 80, 100, and 120 °C. The different times required to achieve packaging friability were determined for each temperature¹³. The values obtained were used to plot the correlation curve between the temperature and the time required to achieve a given degree of friability ($f(t) = T$).

Determination of water absorption capacity

The water absorption test for packaging biofilms allows the ability of these packages to keep food fresh to be evaluated. This absorption measurement was carried out over a temperature range of 35 to 105°C. The range was obtained by adding 15°C to the minimum temperature each time. This temperature range was chosen based on the actual conditions of use of packaging biofilms as food packaging¹³.

Thus, 5x5 cm² square surfaces of each sectioned packaging biofilm were weighed (**mi**) and batches were formed. Each batch was then immersed in a water bath at the specified temperature for 20 minutes. After removal, all square surfaces were weighed again (**mf**). The change in mass (**Δm**) of each packaging biofilm between before and after immersion was determined¹³ (**Onzo et al., 2014b**). This variation, which reflects absorption, is either a gain (+**Δm**) or a loss (-**Δm**) and was measured as follows: **Δm = (mf-mi) (g)**; where mf is the final mass of the immersed packaging biofilms and mi is the initial mass of the packaging (unimmersed).

Statistical analyses

The information obtained from the surveys was processed manually and electronically. SPSS 20.0 software was used to analyze the questionnaire data. Excel was used to plot the graphs. SPSS 20.0 software was used for statistical analysis.

III. Results

Characteristics of formulated packaging biofilms

The yields of plant material powders showed variations with significant differences at the 0.05% threshold, ranging from 12.17±0.61a% (*M. paradisiaca* leaves) to 15.43±0.86b% (*M. esculenta* roots) (**Table 2**). The results showed different varieties of packaging biofilms formulated with different thicknesses ranging from 0.96 mm to 1.59 mm and identical sensitivity (15.75%) (**Figure 3**) (**Table 3**).

Table 2 : Extraction yields

Plant matrices	Powder yield (%)
Leaves of <i>M. paradisiaca</i>	12,17±0,61 ^a
Leaves of <i>T. daniellii</i>	14,50±0,76 ^b
Roots of <i>M. esculenta</i>	15,43±0,86 ^b

Values with different letters in the same column are significantly different at the 0.05% level.

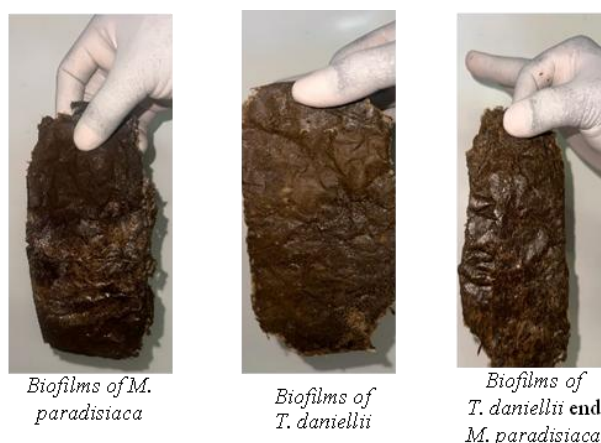


Figure 3 : Formulated packaging biofilm varieties

Table 3 : Thickness and sensitivity of packaging biofilms

Physical characteristics of packaging biofilms	<i>T. daniellii</i>	<i>M. paradisiaca</i>	<i>T. daniellii</i> + <i>M. paradisiaca</i>
Thickness (mm)	0.96	1.34	1.59
Sensitivity (%)	15.75	15.75	15.75

Physicochemical characteristics of formulated packaging biofilms

Friability of packaging biofilms

The results indicate that for temperatures ranging from 60 to 120°C, the friability time of packaging biofilms supplemented with both plant fibers (*Musa paradisiaca* and *Thaumatococcus daniellii*) varies from 20 to 50 min, followed by that formulated solely from *Thaumatococcus daniellii* fibers (15 to 45 min). Between 60 and 120 °C, packaging biofilms supplemented only with *Musa paradisiaca* fibers wilt faster (2 to 15 min) (Figure 4).

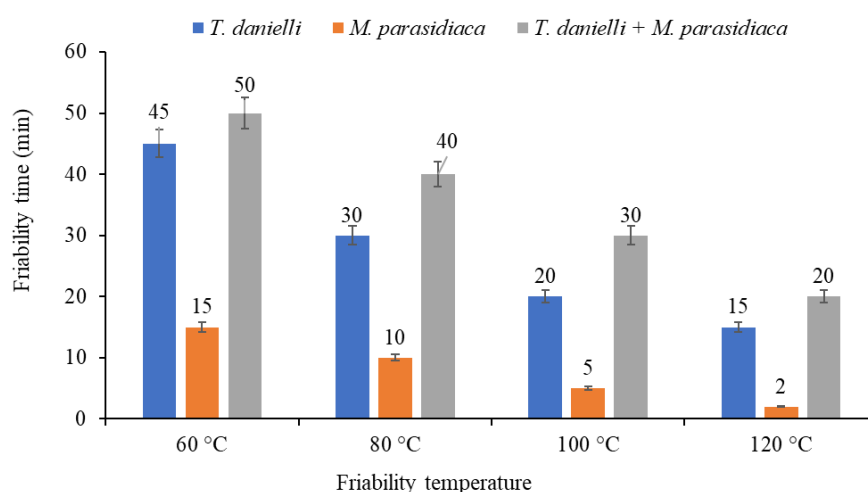


Figure 4 : Friability of different fiber-supplemented packaging biofilms

Opacity of packaging biofilms

In order of increasing importance, packaging biofilms supplemented solely with *T. daniellii* fibers exhibited the highest opacity scores at wavelengths DO₃₃₀ (0.52 ± 0.01), DO₅₀₀ (0.37 ± 0.01), and DO₈₂₀ (0.64 ± 0.05). Biofilms formulated from *M. paradisiaca* (0.16 ± 0.01 to 0.35 ± 0.1) and the mixture of the two (2) fibers (*T. daniellii* + *M. paradisiaca*) were the least opaque (0.12 ± 0.01 to 0.51 ± 0.01) at the wavelengths studied. The results indicated that there was no significant difference between the opacities of the *T. daniellii* and *T. daniellii* + *M. paradisiaca* biofilms at DO₃₃₀ wavelengths (0.52 ± 0.01 ; 0.51 ± 0.01) for $p < 0.0001$. For $p = 0.005$, there was no significant difference between the opacities of *M. paradisiaca* and *T. daniellii* + *M. paradisiaca* biofilms at wavelengths of DO₈₂₀ (0.52 ± 0.01 ; 0.51 ± 0.01) DO₈₂₀ (0.35 ± 0.1 ; 0.47 ± 0.01) (Table 4).

Table 4 : Opacity of different packaging biofilms

Packaging biofilms	Wavelength range (nm)		
	DO ₃₃₀	DO ₅₀₀	DO ₈₂₀
<i>T. daniellii</i>	0.52 ± 0.01^a	0.37 ± 0.01^a	0.64 ± 0.05^a
<i>M. paradisiaca</i>	0.16 ± 0.01^b	0.33 ± 0.01^b	0.35 ± 0.1^b
<i>T. daniellii</i> + <i>M. paradisiaca</i>	0.51 ± 0.01^a	0.12 ± 0.01^c	0.47 ± 0.01^b
Pr > F (Model)	< 0.0001	< 0.0001	0.005

Values with the same letters in the same column are not significantly different at the 0.005 level.

Water absorption capacity of packaging biofilms

Analysis of the results shows that for all temperatures between 35 and 95°C, the three types of packaging biofilms supplemented with fibers absorb water with $\Delta m_{\text{moy}} > 0$ (Table 5). On average, packaging biofilms formulated with a mixture of *T. daniellii* and *M. paradisiaca* fibers (2.31 g) absorb less water than those formulated with *T. daniellii* alone (6.23 g) and *M. paradisiaca* alone (5.05 g). The results indicate that there is no significant difference between the mass variations of packaging biofilms supplemented with *T. daniellii* fibers and *M. paradisiaca* fibers at temperatures of 35°C, 50°C, and 80°C (with $p = 0.001$ and $p < 0.0001$) (Table 5).

Table 5 : Mass variation as a function of temperature

Biofilms d'emballage	Mass variation (Δ mmoy (g)) as a function of temperature					Moyenne
	35°C	50°C	65°C	80°C	95°C	
<i>T. daniellii</i>	1,200 ^a	5,400 ^a	6,300 ^a	7,000 ^a	11,267 ^a	6,230 ^a
<i>M. paradisiaca</i>	1,200 ^a	5,300 ^a	4,800 ^b	6,333 ^a	7,600 ^b	5,050 ^a
<i>T. daniellii</i> + <i>M. paradisiaca</i>	0,467 ^b	1,300 ^b	1,467 ^c	3,233 ^b	5,067 ^c	2,310 ^b
Pr > F(Model)	0,001	< 0,0001	< 0,0001	0,001	< 0,0001	

Values with the same letters in the same column are not significantly different at the 0,001 level.

Distribution of pH and moisture content in packaging biofilms

It can be seen that for all formulated packaging biofilms, the pH is approximately neutral, with values ranging from 6.7 to 7.3 (Figure 5a). Normalized residual analysis showed that the pH values for *M. paradisiaca* and *T. daniellii* range from 7.1 to 7.3. In order of increasing importance, the normalized residual analysis indicates that the moisture content for packaging biofilms supplemented with *T. daniellii* + *M. paradisiaca* fibers and *M. paradisiaca* fibers is between 47% and 52%. This moisture content for packaging biofilms supplemented with *T. daniellii* fibers is greater than 57% (Figure 5b).

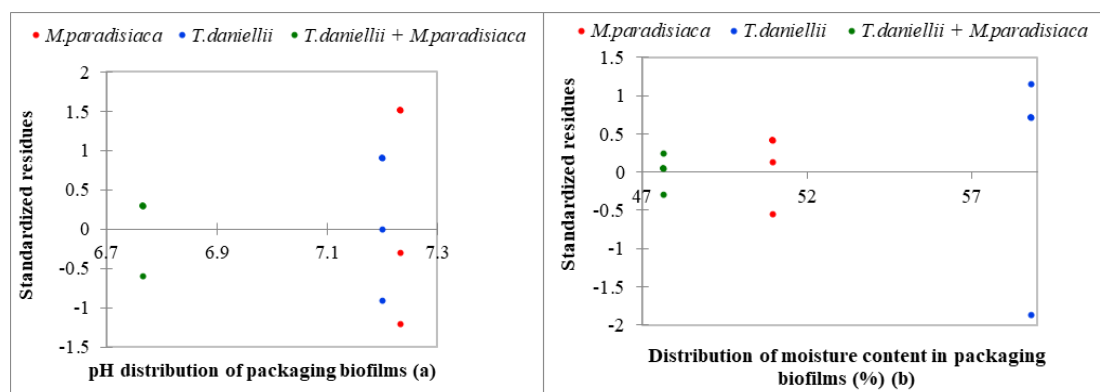


Figure 2 : Distribution of moisture content and pH in packaging biofilms

IV. Discussion

Air pollution and environmental degradation pose significant risks to human health, biodiversity, economic growth, and the survival of ecosystems. Today, several countries around the world, particularly in Africa, are very concerned about pollution and the state of their local environment. In this context, several studies are focusing on the production of biodegradable packaging and the recycling of plastic waste using local materials. This study is part of the drive to contribute to the production of environmentally friendly biodegradable packaging.

Thus, several formulations of biodegradable packaging biofilms supplemented with starch-based plant leaf fibers were produced in this study. These different categories of packaging biofilms with different textures and physicochemical characteristics were obtained from different yields of plant powder. These same formulations were produced by Tamnou et al.²¹, who formulated packaging biofilms from potato starch. The physicochemical properties of their packaging biofilms differ from those obtained in this study. These variations could be explained by the properties of the different matrices and fibers used in the formulation of these biodegradable plant-based packaging biofilms^{22,23}.

his study also presents various physicochemical and mechanical characteristics of the formulated packaging biofilms. The heat resistance of the packaging biofilms indicated that at temperatures between 60°C and 120°C, packaging biofilms supplemented only with *Musa paradisiaca* fibers wilt faster. Those supplemented with *Thaumatococcus daniellii* fibers and combined plant fibers reach friability less quickly. This potential shows that it is prudent to package hotter foods in packaging biofilms supplemented with *Musa paradisiaca* fibers.

These results contradict those obtained by Benie et al.¹², who indicated that it was preferable to store hot food in *Thaumatococcus daniellii* leaves, which are less brittle than those of *Musa paradisiaca*. This contradiction could be explained by the fact that this study focuses on starch-based packaging biofilms supplemented with fibers, rather than on the plant leaf itself, as shown by Benie et al.¹². In addition, this difference in brittleness could be justified by the species of plant leaves, their intrinsic compositions, and their level of maturity²⁴. Hot foods need to be packaged in heat-resistant food containers to prevent the transfer of certain toxic substances from the packaging to the food²⁵.

Despite the good friability of packaging biofilms supplemented with *Musa paradisiaca*, they are less opaque than those of *T. daniellii*, which had the highest opacity scores at DO₃₃₀, DO₅₀₀, and DO₈₂₀ wavelengths. The higher the opacity of a packaging, the better it protects the food from phytochemical degradation. Indeed, prolonged exposure of certain foods to solar ultraviolet rays induces a series of reactions. Among these reactions, photo-oxidation is generally responsible for food spoilage²⁶.

In addition, water absorption is an important physicochemical factor to consider when choosing a high-quality packaging biofilm¹². This absorption provides information on the ability of packaging biofilms to keep food fresh. This study showed that for all temperatures ranging from 35 to 95°C, packaging biofilms supplemented with plant fibers in a starch matrix absorb water with a mass variation Δm_{moy} greater than zero.

This finding could be explained by the fact that these plant fibers have identical chemical compositions and properties, as well as identical levels of maturity. As a result, these biofilms could better preserve food at cool temperatures²⁷. These various findings have been made by several authors who have highlighted the potential of packaging to absorb water through several mechanisms²⁸.

In addition to this water absorption, standardized residual analysis shows that for all formulated packaging biofilms, the pH is approximately neutral with moisture levels around 50%. This finding could explain the shelf life of foodstuffs and the susceptibility of these packaging biofilms to biological degradation²⁹. This level of preservation could be justified by the presence of phytochemical compounds²⁹.

The study will present the potential of these plant fiber-supplemented packaging biofilms to be used in the design of biodegradable, photo-protective food packaging that complies with ecological requirements.

V. Conclusion

This study enabled the development of biodegradable packaging biofilms supplemented with fibers from *Thaumatococcus daniellii*, *Musa paradisiaca*, and a combination of fibers from these two plant species. It also highlighted the mechanical and physicochemical properties of these three formulations. This study could serve as a database for the valorization of these biodegradable leaf species with a view to protecting the environment.

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