

RESEARCH ARTICLE OPEN ACCESS

Optimization of Cassava Starch-Based Biodegradable Films With *Garcinia kola* Oil and *Cocos nucifera* L. Microfibers Using Response Surface Methodology

Yao Désiré Adjouman^{1,2}  | Amenan Aline Doh^{1,2}  | Charlemagne Nindjin^{1,2} | Dekker Matthis³ | Kouamé Alfred Kohi^{1,2} | Fabrice Achille Tetchi¹ | N'Guessan Georges Amani¹

¹Department of Food Science and Technology, Nangui ABROGOUA University 02 BP 801, Abidjan 02, Côte d'Ivoire | ²Centre Suisse de Recherches Scientifiques en Côte d'Ivoire (CSRS) 01 BP 1303, Abidjan 01, Côte d'Ivoire | ³Department of Food Quality and Design, Wageningen University, P. O. Box 17 6700AA, Wageningen, Netherlands

Correspondence: Yao Désiré Adjouman (desyadjouman@gmail.com; desire.adjouman@csrs.ci)

Received: 24 July 2025 | **Revised:** 14 February 2026 | **Accepted:** 9 March 2026

Academic Editor: Poulami Jha

Keywords: biodegradable film | cassava starch | *Cocos nucifera* L. microfibers | *Garcinia kola* oil

ABSTRACT

Starch-based films have low water resistance and poor mechanical properties. This study aimed to optimize the properties of biodegradable films based on cassava starch reinforced with *Garcinia kola* oil and *Cocos nucifera* L. microfibers. To this end, OléKanga cassava starch, *G. kola* oil, and *C. nucifera* L. microfibers were used to produce an optimized packaging film using response surface methodology (RSM). The desirability function subsequently determined an optimal formulation. The results showed that adding microfibers, and oil increased thickness by 77%, reduced water content by 43%, decreased water solubility of the film by 46%, and reduced water vapor permeability (WVP) of the starch-based films from 955×10^{-13} to 5.15×10^{-13} g·Pa⁻¹·s⁻¹·m⁻¹. In other words, there was a 99% increase in the film's water vapor barrier. However, elongation was reduced with the addition of microfibers. The addition of *G. kola* oil increased film opacity from 1.56 ± 0.05 to 4.26 ± 0.08 A·mm⁻¹ and reduced the breaking strength by 62%. An optimal film formulation was determined using the desirability function, with 4 g OléKanga cassava starch, 30% glycerol, 5% *C. nucifera* L. microfibers, 25% *G. kola* oil, and 5% lecithin. The properties of the films were improved by adding *G. kola* oil and *C. nucifera* L. microfibers to the starch matrix.

1 | Introduction

In a global context where environmental issues are gaining traction across all forums and countries are constantly reminded to do their part to combat plastic pollution, initiatives are multiplying everywhere [1]. Indeed, global production of conventional plastics from petroleum-based raw materials has increased annually, and this trend is expected to continue [1]. According to a study by the Regional Environment Agency in Upper Normandy [2], more than 100,000 marine animals (mainly turtles) die each

year, either trapped in plastic bags or after ingesting floating waste, mistaking it for prey. Today, plastic accounts for between 60% and 80% of the ocean's waste. This is why more countries are banning the use of plastic bags within their borders. In Côte d'Ivoire, particularly in the metropolitan area of Abidjan, plastic packaging waste represents roughly 30% of household solid waste, thereby exerting significant pressure on urban environmental quality [1]. However, these products take several years to degrade and often generate toxic decomposition products [1].

This is an open access article under the terms of the [Creative Commons Attribution](https://creativecommons.org/licenses/by/4.0/) License, which permits use, distribution and reproduction in any medium, provided the original work is properly cited.

Copyright © 2026 Yao Désiré Adjouman et al. *Journal of Food Processing and Preservation* published by John Wiley & Sons Ltd.

In line with this awareness, Côte d'Ivoire joined the list of countries banning plastic bags for everyday use in 2013.

Replacing petrochemical plastics with renewable raw materials is a key prospect. Natural polymers derived from renewable resources, such as native starch, chitosan, and natural fibers, are considered promising alternatives to petroleum-based polymers [3]. Among natural biopolymers, starch is regarded as the most promising raw material for producing new environmentally friendly materials, especially for packaging and disposable applications [4, 5]. Starch films are biodegradable and offer advantages such as nontoxicity, colorlessness, and tastelessness. Nevertheless, starch films have drawbacks, notably low water resistance and poor mechanical properties [6]. Several studies have focused on reinforcing starch-based films with various biological materials, such as cellulose fibers [7, 8]. According to Mittal et al. [5], the incorporation of fibers provides significant reinforcement, as reflected by markedly higher load-bearing strength and stiffness.

Coconut fiber is one of the plentiful natural fibers in tropical regions. According to FAOstat [9], roughly 124,810 tons of coconuts are produced annually in Côte d'Ivoire. However, numerous waste coconut husks inundate the vicinity of coastal city markets [10]. Coconut fiber predominantly contains cellulose, hemicellulose, and lignin. The substantial lignin content (46% by mass) in coconut fibers means they are inherently resistant and do not require specific chemical treatment. Additionally, coconut fiber also absorbs less water compared to other fibers due to its lower cellulose content [11]. Due to these attributes, coconut fiber could be used to reinforce starch-based films.

The properties of starch-based films can be further improved by formulating composite films by incorporating lipid compounds. Several studies have been conducted in that regard. Hernando et al. [12] produced composite films based on cassava starch and apple pectin with the addition of laurel oil and oleic acid. Nindjin et al. [13], on the other hand, added peanut oil and sucrose to cassava starch. They obtained strong, flexible films with low water vapor permeability (WVP).

Garcinia kola is a widespread forest species in Côte d'Ivoire. *G. kola* is a spontaneous plant with several uses. The most common are food and pharmacopoeia [14]. Recently, several studies have shown that *G. kola* seeds contain oil. Indeed, Stephen et al. [15] evaluated the nutraceutical potential of *G. kola* seed oil. In addition, the antioxidant and antimicrobial activity of *G. kola* seed oil was studied by Doh et al. [16]. This study revealed that *G. kola* oil has good antioxidant activity and that this activity depends on the oil concentration. In addition, the antimicrobial activity of *G. kola* oil was found to be positive on bacterial strains (*Staphylococcus aureus*, *Escherichia coli*, and *Salmonella typhimurium*) and fungal strains (*Candida albicans*, *Aspergillus flavus*, and *Aspergillus niger*). With its antioxidant and antimicrobial properties, *G. kola* seed oil could also be used in the food packaging industry.

This study aims to produce a biodegradable film based on cassava starch (*Manihot esculenta* Crantz) incorporated with *G. kola* oil and microfibers of the coconut mesocarp (*Cocos nucifera* L.). Considering that, it is important to study the film-forming properties regarding the different proportions of variables. Response surface methodology (RSM) was used for optimizing the composition of the packaging film.

2 | Materials and Methods

2.1 | Plant Materials

Cassava roots (*Manihot esculenta* Crantz), especially from the variety OléKanga, were used for the extraction of starch. Concerning oil, the extraction was made from the seeds of *G. kola*. The fibrous mesocarp of *C. nucifera* L. was used to obtain microfibers.

2.2 | Methods

2.2.1 | Extraction of Composite Materials From Films

2.2.1.1 | Cassava Starch Extraction (*Manihot esculenta* Crantz). The starch extraction was made according to the method described by Amani et al. [17]. Cassava tubers were peeled and cut into chips, washed twice with tap water, and ground using an electric grinder. The grind was weighed and then mixed in equal proportion with water, macerated with a spatula. The mixture was filtered through a series of sieves whose meshes are respectively 500, 250, and 100 μm in diameter to obtain the starch milk. The starch milk obtained has undergone alternating decantation and washing at least once. The product obtained was dried in an oven (Biobase model BOV-T105F, China) at 45°C for 48 h. The dried product was ground using a blender (Silver Crest, SC-9520) and weighed.

2.2.1.2 | Extraction of *G. kola* Oil. The oil contained in the seeds of *G. kola* was extracted by maceration in hexane at room temperature according to the method described by Ungo-Kore et al. [18]. In this process, the *G. kola* seeds were crushed coarsely using a Silvercrest blender and then dried at 50°C for 48 h in an oven (Biobase model BOV-T105F, China). Approximately 1500 g of pulverized seeds was placed in a tightly sealed container with 1000 mL of n-hexane and allowed to stand at room temperature for a period of 3 days with intermittent stirring until the soluble matter was dissolved. The mixture was then filtered, the pomace (wet solid) was pressed, and the oil extract was clarified by filtration after a rest period. The oil was obtained from the mixture (oil and solvent) using a rotary evaporator at 40°C, then collected in a brown glass vial and refrigerated (4°C) pending analysis.

2.2.1.3 | Extraction of Cellulosic Microfibers From the Mesocarp of the Coconut. The fibrous mesocarp was washed with distilled water to remove contamination and impurities. Subsequently, they were dried in an oven (Biobase model BOV-T105F, China) for 72 h at 60°C. This step was followed by grinding using a blender (Silver Crest blender), then sieving was done with a 250- μm mesh sieve. This operation was followed by further grinding with a laboratory mortar. The principle of this grinding is to crush the fibers without destroying the fibrils [19]. The resulting fibers are then sieved with a 100- μm sieve to de-agglomerate them and especially to obtain fine microfibers of very small sizes.

2.3 | Design of Experiments

RSM was used for experimental design (central composite design), modeling, and analysis of the effects of specific factors

on film properties (Design Expert, version 13, Stat-Ease, Minneapolis, USA). It offers great potential for analyzing the effect of several variables on the desired responses, using linear or polynomial functions to achieve optimization. In this work, a central composite design was used to optimize packaging composition, with three factors at constant concentrations (starch, glycerol, and lecithin) and two factors at variable concentrations (microfibers and *G. kola* oil). The concentrations of starch, glycerol, and lecithin were set according to the study carried out by Adjouman et al. [20]. According to this study, concentrations of 4% starch and 30% glycerol showed good WVP and mechanical strength. The effect of the selected variables on thickness, moisture content, WVP, solubility, opacity, elongation, and breaking strength was studied. The experimental design for the RSM analysis is shown in Table 1.

2.4 | Preparation of Biodegradable Bio-Composite Films

An experimental design of 12 formulations was established by modeling with the RSM. The preparation of the gel was done by film casting in three stages according to the method established by Adjouman et al. [20]. For formulations containing microfibers, a preliminary step was necessary. The microfibers of *C. nucifera* L were mixed with 80 mL distilled water of the final mixture for 24 h with constant stirring using a mechanical stirrer (BIOBASE, Model: SK-0330-Pro, China) at 300 revolutions per minute. For the first step, 4 g of native starch powder of the cassava (variety OléKanga), glycerol (plasticizer) at 30% (on the dry basis of starch w/w) were mixed with 80 mL of distilled water of the final mixture. For films containing microfibers, starch and glycerol were added directly to the microfiber/distilled water solution prepared beforehand. The mixture obtained was heated for 20 min from 30 to 75°C using a magnetic and heated stirrer (BIOBASE, Model: MS7-H550-S, China). The second step involved mixing 20% *G. kola* oil (by weight of starch) and 5% soya lecithin by weight of oil (soya lecithin is an emulsifier that

will homogenize the oil and water) with 20 mL of the total mixture. The mixture of oil and lecithin was also heated for 20 min from 30 to 75°C with constant stirring using a magnetic and heating stirrer (BIOBASE, model: MS7-H550-S, China) at 750 rpm. The solution of *G. kola*, soy lecithin, and distilled water was homogenized at 26,000 turns for 1 min using Ultra Turrax. In the third step, the homogenized solution was mixed with the first solution and then heated from 75 to 95°C for 25 min at 750 rpm using a magnetic and heating stirrer (BIOBASE, Model: MS7-H550-S, China). On the surface of a petri dish, 20 g of the final solution obtained is poured and dried in a ventilated oven (Biobase model BOV-T105F, China) at 35°C for 24 h. The oven-dried films are removed and stored at 62% water content (ammonium nitrate [NH₄NO₃]) for 48 h in a moisture analyzer at 25°C before testing.

2.5 | Characterization of Film

2.5.1 | Determination of Film Thickness

The thickness of all films was determined using a manual Micrometer (Digital outside micrometers, MRC, China) at 10 random positions on the films.

2.5.2 | Determination of the Water Content of Films

The water content (*H*) was determined gravimetrically, according to the protocol described by Bajić et al. [21]. Rectangular samples (1 cm × 2 cm) were weighed on a precision balance (BP-B/P, BIOBASE, China) to obtain the initial mass (*M*₁) and the dry mass (*M*₂) obtained after oven drying (BOV-V125F, BIOBASE, China) at 105°C for 24 h. The water content was calculated using the following Equation (1), and the results were expressed as a percentage.

$$H(\%) = (M_1 - M_2/M_2) \times 100, \quad (1)$$

where *H* is the water content, *M*₁ is the initial mass, and *M*₂ is the dry mass.

TABLE 1 | Experimental design of the combination of variables to produce a biodegradable film.

Try	Constants			Factors	
	Starch (g)	Glycerol (% based on starch mass)	Lecithin (% based on oil mass)	Microfibers (% based on starch mass)	<i>Garcinia kola</i> oil (% based on starch mass)
1	4	30	5	0	0
2	4	30	5	0	10
3	4	30	5	0	40
4	4	30	5	5	20
5	4	30	5	5	30
6	4	30	5	5	50
7	4	30	5	10	25
8	4	30	5	15	20
9	4	30	5	15	30
10	4	30	5	15	50
11	4	30	5	20	10
12	4	30	5	20	40

2.5.3 | Determination of WVP of Films

The permeability of the film to water vapor was measured according to Kumaran [22], with some modifications. Circular film samples are mounted and sealed on the opening of a cylindrical jar containing 50 g of anhydrous calcium chloride (0% RH) to maintain a 75% relative humidity difference through the film. The assembly is placed in a desiccator at 25°C containing a saturated solution of sodium chloride (75% RH). After obtaining the equilibrium conditions (2 h later), 8 measurements (to the nearest 10–4 g) of the cut are made every 60 min and illustrated as a time-related curve. The slope of each line is calculated according to linear regression ($r > 0.99$), and water vapor transmission (WVTR) is calculated by the ratio of the slope of the straight line ($\text{g}\cdot\text{s}^{-1}$) to the transfer surface (m^2). The exposed area is 0.00246 m^2 , and the WVP ($\text{g}\cdot\text{Pa}^{-1}\cdot\text{S}^{-1}\cdot\text{m}^{-1}$) is calculated according to the Equation (2):

$$\text{WVP} = \text{WVTR} / S(R_1 - R_2)d, \quad (2)$$

where S is the vapor pressure at water saturation (Pa) at the test temperature (25°C) = 3170 Pa, R_1 is the relative humidity in the moisture analyzer, R_2 is the relative humidity in the pot, and d is the thickness of the film (m).

2.5.4 | Determination of the Water Solubility of Films

According to the method described by López et al. [23], $2 \text{ cm} \times 3 \text{ cm}$ pieces of each film were cut and stored for 7 days in a moisture analyzer containing silicate gel. The samples were weighed (to the nearest 0.0001 g; initial dry weight) using a precision balance (BP-B/P, BIOBASE, China) and placed in beakers containing 80 mL of deionized water. Samples were kept with constant stirring at 200 rpm in a stirring chamber (mechanical agitator, BIOBASE, Model: SK-0330-Pro, China) for 1 h at laboratory temperature ($\sim 25^\circ\text{C}$). However, the film parts were collected by filtration and dried again in an oven (Biobase model BOV-T105F, China) at 60°C until constant weights were obtained (final dry weight). The percentage of total solid matter (or percentage of solubility) is calculated as follows:

$$S(\%) = (P_0 - P_f / P_0) \times 100, \quad (3)$$

where S is the solubility, P_0 is the initial dry weight, and P_f is the final dry weight.

2.5.5 | Determining Film Opacity

Film opacity was measured using the method described by Amariei et al. [24], using a Shimadzu 1800 UV spectrophotometer. The films were cut into rectangular pieces of $3 \text{ cm} \times 1 \text{ cm}$ and placed inside the spectrophotometer test cell. Distilled water was used as a reference. The UV–Vis spectra were recorded at wavelengths between 200 and 600 nm. The opacity was calculated by the following Equation (4):

$$\text{Opacity} = A_{600} / d, \quad (4)$$

where A_{600} is the film absorbance at 600 nm and d is the film thickness (mm).

2.5.6 | Mechanical Properties

Tensile tests were performed using a Texture Pro CT V1.9 Build 35 according to ASTM [25] using an A/TG traction handle system. The parameters determined are breaking strength and fracture deformation (elongation).

The tested film strips ($8 \text{ cm} \times 2.5 \text{ cm}$) were cut with a scalpel for each preconditioned sample (62% relative humidity, 25°C) and mounted between the jaws of the machine. The thickness of each sample was measured at four points along its length with a manual micrometer (Digital outside micrometer, MRC, China). The initial separation between the handles was set at 50 mm. The upper part of the probe rises gradually by stretching the film at a constant speed of $20 \text{ mm}\cdot\text{min}^{-1}$ until breaking. Force/elongation curves were recorded. Breaking strength and elongation were measured at the point of breakage. For each formulation, five samples were tested to obtain greater statistical accuracy. These parameters are more prone to intra-sample variability, so more tests are required.

2.6 | Definition of Optimal Conditions Using the Desirability Function (D)

The estimation of the optimal conditions for the elaboration of the film was carried out using the simultaneous optimization method called desirability function (D) to meet the requirements of the dependent variables. Therefore, this study aimed to determine the best conditions to create biodegradable films based on cassava starch, *G. kola* oil, and *C. nucifera* L. microfiber with suitable technological properties, including low WVP and opacity as well as high tensile strength and elongation. Constraints were imposed to obtain the optimum film formulation, 25% *G. kola* oil, which is the minimum bacterial inhibitory concentration of *G. kola* oil. The optimum formulation had to contain at least 5% coconut microfibers, which represents the lowest content of cellulosic microfibers.

2.7 | Optimized Film Biodegradability

The biodegradability of the film obtained was determined by burying it in soil under normal conditions for 15 days, using the ASTM D5988-03 method. The soil used was ferrallitic clay with a moisture content of 56%. According to ISO [26], the pH of the soil must be between 6 and 8. Both of these conditions were met.

The biodegradability of a sample of films in soil was analyzed by calculating the weight loss of films buried in soil. The tests were carried out in five replicates. Each $25 \text{ mm} \times 25 \text{ mm}$ film sample was buried 100 mm below the soil surface. Before testing, the samples were weighed to obtain the initial weight (M_i) of the films. At different times (0, 3, 6, 9, 12, and 15 days), the samples were removed from the soil simultaneously and gently cleaned

with distilled water to remove impurities. They were dried in a vacuum oven at 65°C until a constant weight was obtained and weighed to obtain the final weight (M_f) of the films. The disintegration process was monitored by visual inspection. The weight loss of the sample was evaluated using the following Equation (5):

$$\Delta M (\%) = \frac{M_i - M_f}{M_i} \times 100, \quad (5)$$

where ΔM is the mass loss, M_i is the initial mass, and M_f is the final mass.

2.8 | Statistical Analysis

The physical, barrier, and optical properties of the film samples produced were measured in triplicate, while the mechanical properties of the films were tested five times. Mean values were used in the analysis. The experimental data obtained were analyzed using Design-Expert Software (Stat-Ease) to obtain analysis of variance (ANOVA), regression analysis, and factor interaction response surface plots. The linear, quadratic, and polynomial interactive effects of the process variables on the responses were analyzed, and their respective significance was assessed by the ANOVA test. The p -value (≤ 0.05) was used as a measure of the significance of the regression coefficient. Model validation and model fit were tested by assessing the adequate precision (AP) of the data. To optimize the film composition, a multicriteria approach was carried out by defining a desirability score, using weights for important attributes (elongation, WVP, fracture toughness, and opacity were considered the most important (weighting factor = 5), and solubility (weighting factor = 3) was considered slightly less important).

Each combination of experimental factors corresponded to a separate batch of films, prepared separately to ensure the reproducibility of manufacturing conditions.

3 | Results

3.1 | Appearance of Biodegradable Films

Figure 1 shows the appearance of the films of cassava starch, *G. kola* oil, and microfibers of *C. nucifera* L. The starch and

glycerol-based plastic films (Figure 1a) were homogeneous, shiny, transparent, colorless, and crack-free. On the other hand, films based on starch, glycerol, and *G. kola* oil, and microfibers of *C. nucifera* L. (Figure 1b, c) were less transparent, not shiny, colored, and without cracks. All films easily peeled off the petri dish except films with 50% *G. kola* oil.

3.2 | Film Parameters

3.2.1 | Thickness

Table 2 and Figure 2 show the influence of the addition of *G. kola* oil and *C. nucifera* L. microfibers on the thickness of cassava starch-based film. Analyses revealed that film thickness varied from 79.2 ± 0.3 to $353.76 \pm 0.56 \mu\text{m}$ with the addition of *G. kola* oil and coconut microfiber. Statistical analysis showed that film thickness increased significantly ($p < 0.05$) with microfiber concentration.

3.2.2 | Water Content

The water content shown in Table 2 increased from $51.08\% \pm 0.94\%$ in the starch and glycerol film (control film) to $29.02\% \pm 1.05\%$ after the addition of microfibers and oil. The processing of experimental data led to the development of a linear mathematical model that was significantly ($p < 0.05$) dependent on microfiber content (Figure 3).

3.3 | WVP of Films

WVP is shown in Table 2 and Figure 4. WVP decreased significantly (99%, $p < 0.05$) upon addition of 10% *G. kola* oil. Indeed, WVP fell from $954.79 \times 10^{-13} \pm 3.53 \times 10^{-12}$ to $5.15 \times 10^{-13} \pm 1.63 \times 10^{-14} \text{ g}\cdot\text{Pa}^{-1}\cdot\text{s}^{-1}\cdot\text{m}^{-1}$ after oil addition. In contrast, the WVP obtained with the addition of oil increased slightly by 17% after the incorporation of *C. nucifera* L. microfibers. However, the result obtained with the addition of the film is significantly lower (a decrease of 97%), with the WVP of the starch-based film.

3.4 | Water Solubility of Films

Results for films based on OléKanga cassava starch, *G. kola* oil, and *C. nucifera* L. microfibers, are shown in Table 2 and Figure 5.

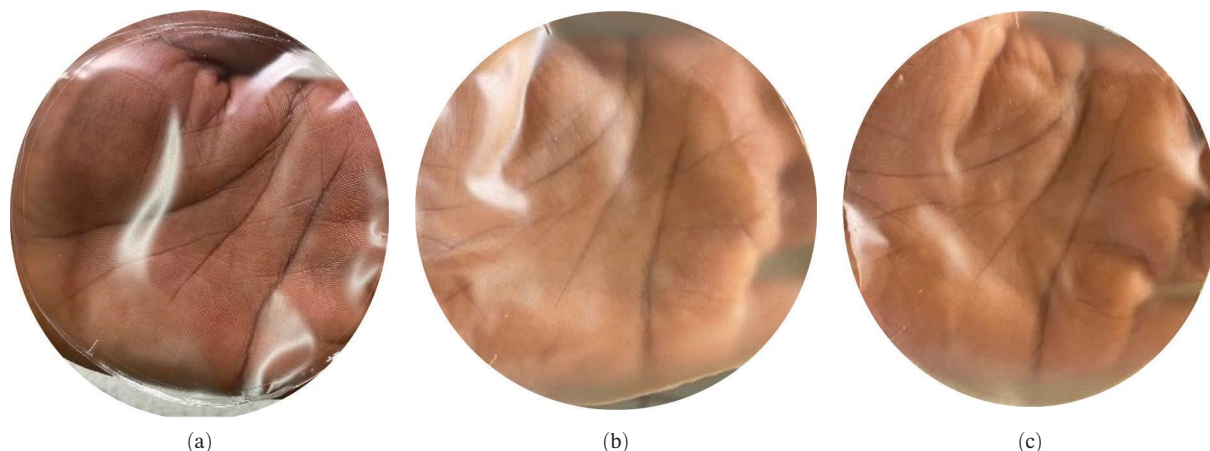


FIGURE 1 | Photograph of films based on cassava starch OléKanga and *Garcinia kola* oil and microfibers of *Cocos nucifera* L. (a) Film based on starch and glycerol. (b) Film based on starch, glycerol, and oil of *G. kola*. (c) Film based on starch, glycerol, oil of *G. kola*, and microfibers of *Cocos nucifera* L.

TABLE 2 | Experimental responses.

	Constants				Factors		Responses						
	A (g)	Gly (%)	Lec (%)	C (%)	G (%)	Thickness (μm)	Water content (%)	WVP (1E – 13 g Pa ⁻¹ s ⁻¹ m ⁻¹)	Solubility (%)	Opacity (A·mm ⁻¹)	Elongation (%)	Resistance to breakage (MPa)	
Try 1	4	30	0	0	0	79.2 ± 0.85	51.08 ± 0.94	954.79 ± 35.34	41.45 ± 0.92	1.56 ± 0.05	39.65 ± 3.25	1.73 ± 0.07	
2	4	30	5	0	10	90.1 ± 0.79	49.52 ± 0.82	5.15 ± 0.16	33.76 ± 1.12	1.72 ± 0.02	63.79 ± 5.89	1.43 ± 0.03	
3	4	30	5	0	40	110.7 ± 0.7	45.42 ± 0.45	6.82 ± 0.24	31.88 ± 0.76	4.26 ± 0.08	45.16 ± 3.29	0.96 ± 0.06	
4	4	30	5	5	20	135.13 ± 0.74	42.36 ± 1.2	9.1 ± 0.66	32.95 ± 1.05	1.75 ± 0.02	26.77 ± 3	2.29 ± 0.1	
5	4	30	5	5	30	178.8 ± 0.65	37.31 ± 0.82	9.87 ± 1	27.4 ± 0.47	2.43 ± 0.11	52.67 ± 2.73	1.18 ± 0.14	
6	4	30	5	5	50	158.8 ± 0.62	48.35 ± 0.8	25.64 ± 1.45	25.52 ± 0.58	2.14 ± 0.03	34.05 ± 0.4	0.64 ± 0.07	
7	4	30	5	10	25	279.13 ± 0.3	40.03 ± 1.24	23.07 ± 0.9	28.44 ± 0.96	1.87 ± 0.05	43.828 ± 5.63	0.65 ± 0.05	
8	4	30	5	15	20	303.53 ± 0.5	42.75 ± 0.3	24.17 ± 0.94	26.12 ± 0.64	1.78 ± 0.03	48.876 ± 5.8	1.03 ± 0.14	
9	4	30	5	15	30	313.83 ± 1.24	41.56 ± 0.91	29.27 ± 1.72	26.03 ± 0.94	1.95 ± 0.08	18.96 ± 1.02	1.13 ± 0.14	
10	4	30	5	15	50	320.53 ± 0.38	45.01 ± 1.88	24.08 ± 0.61	27.09 ± 0.92	2.21 ± 0.04	6.53 ± 0.83	0.75 ± 0.09	
11	4	30	5	20	10	306.93 ± 1.55	38.37 ± 0.56	19.18 ± 0.5	25.05 ± 0.61	1.68 ± 0.02	16.76 ± 1.22	2.28 ± 0.3	
12	4	30	5	20	40	353.77 ± 0.56	29.02 ± 1.05	22.6 ± 0.79	26.38 ± 0.58	2.03 ± 0.02	8.72 ± 1.19	1.13 ± 0.12	

Note: The concentrations of glycerol, microfibers of *Cocos nucifera* L., and *Garcinia kola* oil are given based on the mass of starch, the concentration of lecithin is a function of the mass of *G. kola* oil. A, starch; C, microfibers of *Cocos nucifera* L.; G, *Garcinia kola* oil. Abbreviations: Gly, glycerol; Lec, lecithin.

The water solubility of the films decreased significantly ($p < 0.05$) with the addition of microfibers and *G. kola* oil to the starch matrix. Solubility depended significantly ($p < 0.05$) on microfibers and oil. Solubility ranged from $41.45\% \pm 0.92\%$ to $25.05\% \pm 0.61\%$.

3.5 | Film Opacity

Film opacity values have been presented in Table 2. Statistical analysis revealed that opacity increased significantly ($p < 0.0001$) with the addition of *G. kola* oil. This is also shown in Figure 6. The highest opacity value was obtained with the formulation containing 4g starch, 30% glycerol, 40% *G. kola* oil, and 0% microfiber. In addition, statistical analysis showed that R^2 was close to R^2 -prediction.

3.6 | Resistance to Breakage

G. kola oil and microfiber powder have an influence on the breaking strength of the films, as shown in Table 2 and Figure 7. However, according to statistical analysis and as shown in Figure 7, the relationship between the two factors and film breaking strength was found to be linear and was significantly dependent on *G. kola* oil. Indeed, the percentage of resistance decreased with the increase in the percentage of oil in films. The lowest breaking strength was observed in films with an oil concentration greater than 20%.

3.7 | Elongation

The elongation of the films produced is shown in Table 2 and Figure 8. The percentage of elongation was reduced with the addition of microfiber powder. The mathematical model revealed a linear relationship between oil, microfibers, and the elongation that was significantly ($p < 0.05$) dependent on microfibers.

3.8 | Model Validation

The adequacy of the model was verified using ANOVA, as shown in Table 3. The measures of adequacy are probability (p -value of the model), coefficient of determination R^2 , predicted R^2 , and AP. AP indicates whether the model can be used for optimization. It must be greater than 4, which is the case for all the responses. This model could therefore be used to navigate in the design space.

3.9 | Optimal Conditions for Film Preparation Using the Desirability Function

The desirability profile of the optimal conditions suggested by the RSM method is presented in Table 4. This analysis showed that the maximum overall desirability (0.611) was achieved when the optimized conditions were used (Figure 9). The optimal formulation then suggested for the film based on cassava starch, *G. kola* oil, and *C. nucifera* L. microfibers was the one with the lowest microfiber and oil limits. Thus, the optimal film had the following formulation: 4g starch, 30% glycerol, 5% *C. nucifera* L. microfibers, 25% *G. kola* oil (% relative to the mass of

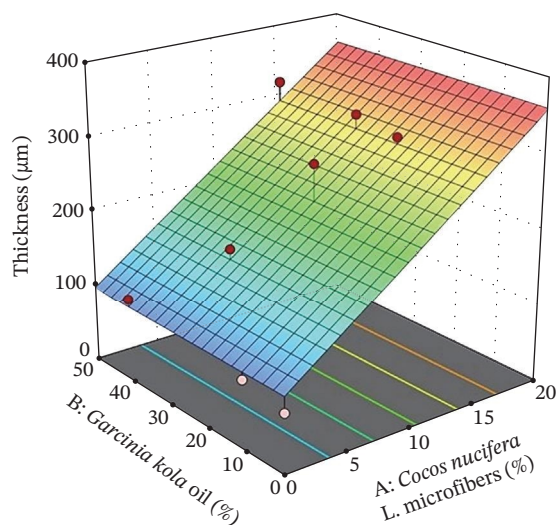


FIGURE 2 | Influence of *Cocos nucifera* L. microfibrers (factor A) and *Garcinia kola* oil (factor B) on film thickness. The colored area represents the response surface predicted by the model, the red points correspond to the experimental values, and the pink points represent the repetitions at the central point.

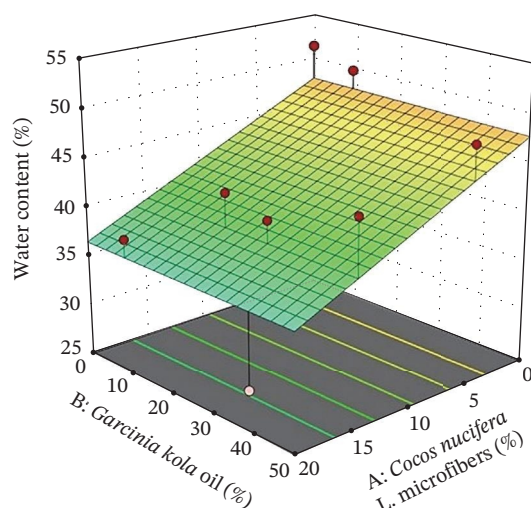


FIGURE 3 | Influence of *Cocos nucifera* L. microfibrers (factor A) and *Garcinia kola* oil (factor B) on the water content of films. The colored area represents the response surface predicted by the model, the red points correspond to the experimental values, and the pink points represent the repetition at the central point.

starch), and 5% lecithin (% relative to the mass of oil). The predicted responses are also shown in Table 4.

3.10 | Film Biodegradability

Figure 10 shows the weight loss recorded during the biodegradability in soil of films made from starch, *G. kola* oil, and coconut microfibrers over a period of 15 days. The weight loss of the reinforced film ranged from $14.94\% \pm 3.70\%$ on day 3 to 100% on day 15. The results also show that the reinforced film degraded more rapidly after 9 days in the soil, with a high mass loss until it reached 100% degradation on day 15.

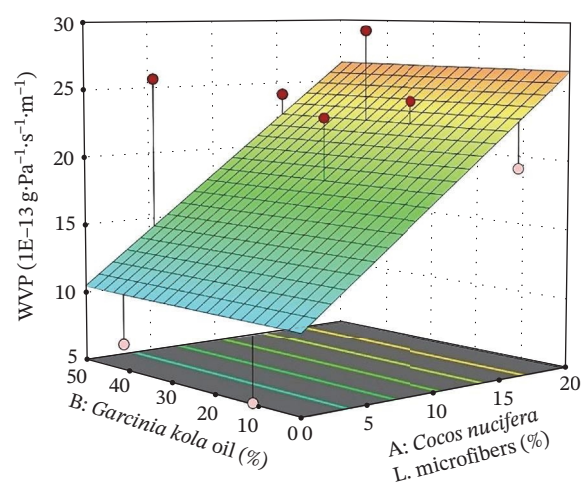


FIGURE 4 | Influence of *Cocos nucifera* L. microfibrers (factor A) and *Garcinia kola* oil (factor B) on the WVP of films. The colored area represents the response surface predicted by the model, the red points correspond to the experimental values, and the pink points represent the repetitions at the central point. WVP, water vapor permeability.

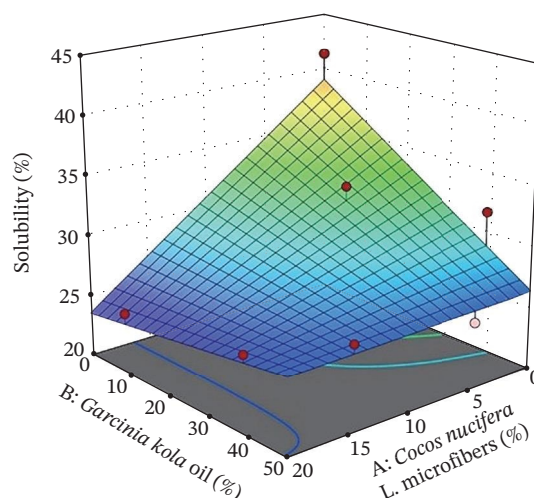


FIGURE 5 | Influence of *Cocos nucifera* L. microfibrers (factor A) and *Garcinia kola* oil (factor B) on the solubility in water of films. The colored area represents the response surface predicted by the model, the red points correspond to the experimental values, and the pink points represent the repetition at the central point.

4 | Discussion

The thickness of the biodegradable film is an important parameter because it affects the packaging material's physical properties. The variable cellulosic microfibrers from the mesocarp of *C. nucifera* L. had a significant effect on film thickness ($p < 0.05$). This result could be explained by the role of fibers in reconstructing and restructuring intermolecular chain networks, thereby increasing film thickness [27]. According to Hazrol et al. [27], this could be attributed to the presence of large filler particles in the polymer matrix, which can favor structural defects. Ibrahim et al. [28] agree with this result; they also observed that adding Palmyra fibers to corn starch led to thicker films. Edhirej et al. [29] noted that the higher the concentration of Palmyra fiber in the cassava starch film, the thicker the film. The cassava starch

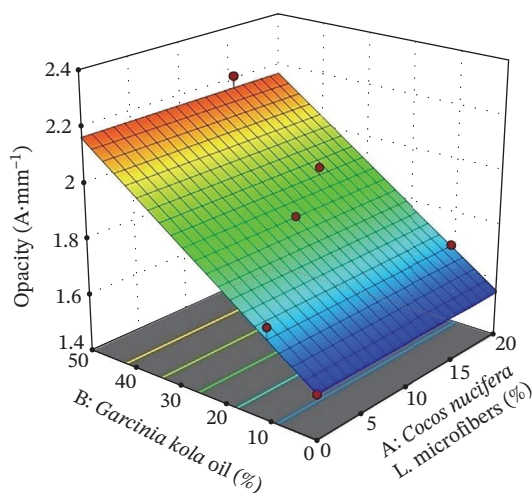


FIGURE 6 | Influence of *Cocos nucifera* L. microfibrers (factor A) and *Garcinia kola* oil (factor B) on film opacity. The colored area represents the response surface predicted by the model, while the red points correspond to the experimental values.

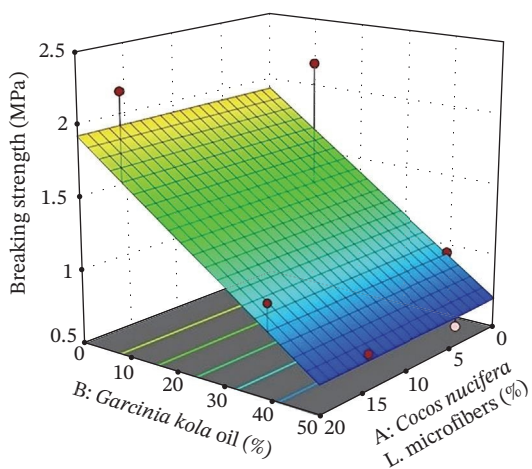


FIGURE 7 | Influence of *Cocos nucifera* L. microfibrers (factor A) and *Garcinia kola* oil (factor B) on the breaking strength of films. The colored area represents the response surface predicted by the model, the red points correspond to the experimental values, and the pink points represent the repetition at the central point.

film was the thinnest at 0.18 mm, while the film containing 8% sugar palm fiber was the thickest at 0.43 mm.

Moisture content is a critical parameter in the application of degradable films. Low water content is necessary, as high-water content can negatively affect the dimensional stability of the composite material, particularly in terms of mechanical performance, pore formation, and barrier properties [30]. In this study, the water content decreased significantly as the percentage of microfibrers increased. Enidiok et al. [31] reported in a study on the reinforcement of starch with natural fibers that, across all starch/fiber formulations, a slightly lower hygroscopicity was observed compared to the starch-only formulation. These results suggest that the presence of fiber modifies the water sensitivity of starch. According to several studies, incorporating cellulose fiber reduces moisture because cellulose has a lower affinity for water than starch [8, 32]. According to Ma et al. [32], some fibers absorb

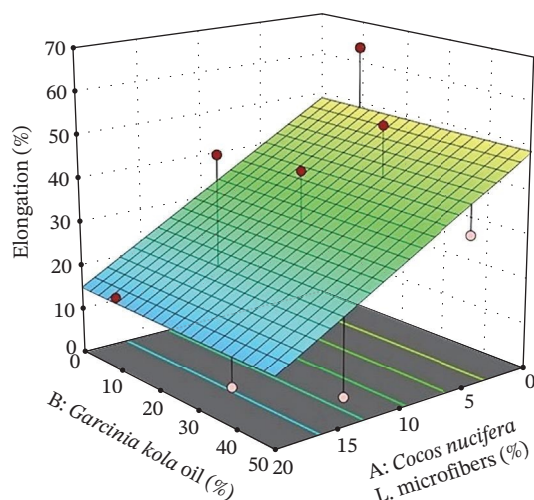


FIGURE 8 | Influence of *Cocos nucifera* L. microfibrers (factor A) and *Garcinia kola* oil (factor B) on the elongation of films. The colored area represents the response surface predicted by the model, the red points correspond to the experimental values, and the pink points represent the repetitions at the central point.

less water than others. These fibers are generally responsible for this reduction in water sensitivity in composites. For example, composites reinforced with hemp and cellulose fibers absorb less water. On the other hand, composites reinforced with cotton or wheat fibers absorb more water than starch [32]. In addition, fibers that absorb less water have a stabilizing effect on the starch polymer matrix, mainly on the amylose molecules, which undergo reduced mobility [32].

The high-WVP of the starch and glycerol film is mainly due to the high hydrophilic character of starches in general [20, 28]. However, the results showed that the water vapor barrier properties were significantly improved by the addition of microfibrers. Sienkiewicz et al. [8] reported that adding fibers decreased the WVP of starch-based films. Indeed, the WVP behavior of films depends on the simultaneous effect of water diffusivity in the polymer matrix and the water solubility coefficient in the film [8]. The decrease in WVP could also be attributed to the formation of a rigid crystalline structure and the dispersion of microfibrers in the polymer matrix, which impedes the permeability path of vapor molecules [31]. Furthermore, according to Sienkiewicz et al. [8], the increase in intermolecular bonding between the starch matrix and the cellulose fibers caused by the addition of microfibrers could reduce the WVP. Indeed, the increase in intermolecular bonding between the starch matrix and the cellulose fibers reduces or eliminates the mobilization of the matrix chains. The results obtained agree with those of Ibrahim et al. [28], who observed a 96.55% improvement in the WVP of the film after adding Palmyra fibers to corn starch. The water solubility of the film is an important property for assessing its applicability in foods and may also determine or promote its biodegradability.

Film solubility decreased significantly with the incorporation of *G. kola* oil and microfiber powders from coconut mesocarp. According to Enidiok et al. [31], the insolubility or low water solubility of films is of critical importance for product integrity, moisture barrier properties, and shelf life. The decrease in film solubility after adding *G. kola* oil could be due to the fact that the

TABLE 3 | Statistical analysis.

Responses	Model	p-Value model	Sign terms	R^2	R^2 -pre-dict	Adequate precision	Equation
Thickness (μm)	Linear	<0.0001	C	0.93	0.90	22.30	$Y = 100.3 + 13.0 \times C$
Water content (%)	Linear	0.0121	C	0.48	0.23	5.90	$Y = 47.6 - 0.55 \times C$
WVP ($1\text{E} - 13 \text{ g Pa}^{-1} \text{ s}^{-1} \text{ m}^{-1}$)	Linear	0.0226	C	0.50	0.21	5.50	$Y = 10.5 + 0.80 \times C$
Solubility (%)	Interaction of the two factors	0.0007	C, $C \times G$	0.87	0.61	12.60	$Y = 39.1 - 0.78 \times C - 0.25 \times G + 0.015 \times C \times G$
Opacity	Linear	<0.0001	G	0.98	0.96	41.10	$Y = 1.56 + 0.012 \times G$
Elongation (%)	Linear	0.0099	C	0.50	0.32	6.10	$Y = 49.4 - 1.70 \times C$
Breaking strength (MPa)	Linear	0.0123	G	0.48	0.32	7.0	$Y = 1.935 - 0.025 \times G$

Note: C, microfibers of *Cocos nucifera* L.; G, *Garcinia kola* oil.

TABLE 4 | Simultaneous optimization parameters and the overall desirability.

Constant	Factor					Response						
A	Gly	Lec	C	G	Thickness	Water content	WVP	Solubility	Opacity	Elongation	Resistance to break age	Desirability
(g)	(%)	(%)	(%)	(%)	(μm)	(%)	($1\text{E} - 13 \text{ g Pa}^{-1} \text{ s}^{-1} \text{ m}^{-1}$)	(%)	($\text{A} \cdot \text{mm}^{-1}$)	(%)	(MPa)	
4	30	5	5	25	165.14	44.86	14.51	30.87	1.86	40.88	1.32	0.611 Selected

Note: The concentrations of glycerol, microfibers of *Cocos nucifera* L., and *Garcinia kola* oil are given based on the mass of starch; the concentration of lecithin is a function of the mass of *G. kola* oil. A, starch; C, microfibers of *Cocos nucifera* L.; G, *Garcinia kola* oil.

Abbreviations: Gly, glycerol; Lec, lecithin.

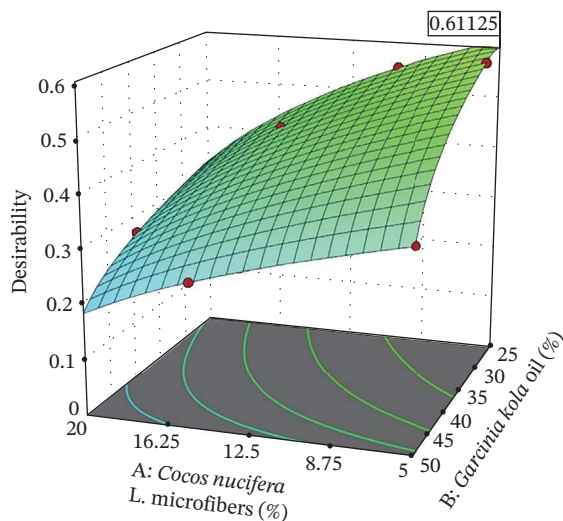


FIGURE 9 | Desirability function showing the combined effect of *Cocos nucifera* L. microfibers (factor A) and *Garcinia kola* oil (factor B). The colored surface represents the desirability predicted by the optimization model, while the red points correspond to the experimental combinations.

oil is a hydrophobic substance. According to Galus and Kadzińska [33], adding a hydrophobic substance to a film formulation produces films that are highly resistant to water. On the

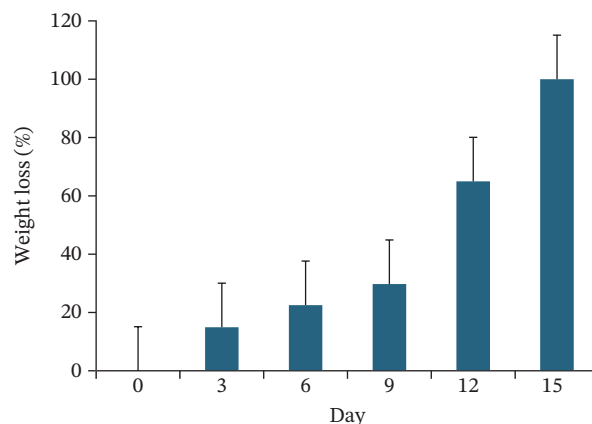


FIGURE 10 | Film weight loss.

other hand, films containing oil have a more irregular microstructure in which the oil droplets are dispersed, which affects the hydrophobicity of the film. Similar results were obtained by Filho et al. [34] after incorporating *Hymenaea stigonocarpa* and *Hymenaea courbaril* oil into biofilms made from arrowroot starch. In their study, the solubility of starch-based films decreased from 51% to 35% and 34%, respectively, for films containing *Hymenaea stigonocarpa* and *Hymenaea courbaril* oil. The addition of microfibers also significantly reduced the water solubility of the films. This could be due to a more rigid starch structure after adding microfibers [8].

The results of this study showed that the opacity of the films increased with increasing concentration of *G. kola* oil. These results are consistent with those of Galus and Kadzińska [33], who observed an increase in opacity values from 0.52 to 4.6 A·mm⁻¹ for edible films based on milk proteins with increasing rapeseed oil content. The increase in film opacity as a function of increasing lipid content has been widely reported in the literature [33]. Enidiok et al. [31] pointed out that the presence of a dispersed and immiscible phase promotes opacity, depending on the differences in refractive index, concentration, and particle size of the dispersed phase.

The elongation of the film at break is related to the flexibility and elongation capacity of the materials. However, in this study, the addition of microfibers tended to reduce this property of the films. In addition, as the microfiber load increases, the elongation of composite films decreases. This suggests that the resulting films have become stiffer and less flexible and have become stronger materials [28]. The reduction in film elongation as the fiber load increases is thought to be due to cellulose, which reconstructs the composite structure by strengthening intermolecular bonds in the plasticized starch matrix. This reconstruction of the starch network improves stiffness and hardness, therefore, decreases flexibility by eliminating chain mobility [8].

Breaking strength refers to the maximum stress that a film can withstand when stretched before breaking. In this study, the breaking strength of the films ranged from 0.65 to 2.29 MPa. The values obtained were lower than those for the breaking strength of low-density polyethylene-based films, which ranged from 9 to 23 MPa [35, 36]. This result could be due to the incorporation of an oil replacing the strong polymer–polymer interaction with a weak polymer–oil interaction, which would reduce the interfacial adhesion between the two phases and break the continuity of the polymer network [37]. Aisyah et al. [38] also reported that vegetable oil can reduce the breaking stress. On the other hand, the tensile strength of a corn starch film decreases with increasing nutmeg oil concentration. Similarly, Bhasney et al. [37] reported that the tensile strength of PLA film decreased with the addition of coconut oil.

Statistical analysis showed that the coefficient of determination R^2 for water content, WVP, elongation, and tensile strength ranged between 0.48 and 0.50. In such cases, the appropriate accuracy test that measures the signal-to-noise ratio can serve as an indicator of the model's ability to effectively distinguish between different design points [39]. The AP of all responses was greater than 4. AP is a statistical test that compares the range of predicted values at design points to the mean prediction error. In practice, a ratio greater than 4 is generally considered indicative of adequate model discrimination, indicating that the model can reliably navigate the design space and produce well-informed predictions. The optimum point obtained with the desirability function to define the film based on cassava starch, *G. kola* oil, and *C. nucifera* L. microfibers was 0.611. With this desirability, the optimum formulation obtained is acceptable. Indeed, according to Bagheri et al. [40], the acceptance scale of the desirability function ranges from 0 (totally undesirable response) to 1 (totally desired response) and corresponds to the interpretation of desirability: very good (1.0–0.8); good (0.8–0.63); average (0.63–0.37); poor

(0.37–0.2); and very poor (0.2–0.2). Moreover, according to Hamadache [41], the desirability of a solution with a value of 0.6 indicates satisfactory overall performance. In addition, according to Cho et al. [42], a desirability value greater than 0.6 is preferable, and a value greater than 0.9 is desirable.

Research into the biodegradability properties of films is important for their application as food packaging. In this study, films based on OléKanga cassava starch enriched with *G. kola* oil and *C. nucifera* L. microfibers were 100% degraded after two weeks. This demonstrates the good biodegradability of the films produced. This result can be attributed to increased microorganism activity during prolonged burial, which increases the weight loss of the material [43]. According to Ilyas et al. [44], microorganisms modify the polymer matrix through a metabolic or enzymatic process that breaks down the high average molecular weight polymer matrix into smaller polymers with a lower average molecular weight. As a result, this process leads to the decomposition of the material in the environment, and the completed biodegradation process is called mineralization. According to Tai et al. [45], the nature and extent of a polymer's biodegradation also depend on the environment (temperature, pH, humidity, and nutrition) as well as the properties of the polymer. The results of this study are consistent with those of Ghizdareanu et al. [46].

5 | Conclusion

The RSM has been successfully applied to the formulation of biodegradable films by linking the composition of the film-forming solution to the physical, chemical, optical, and mechanical properties of the final material. An optimal film formulation was found with 4 g cassava starch, 30% Glycerol, 5% *C. nucifera* L. microfibers, 25% *G. kola* oil (% relative to starch mass), and 5% lecithin (% relative to oil mass) by the desirability function. The results concluded that the addition of *G. kola* oil and *C. nucifera* L. microfibers strengthens cassava starch-based films. These films can potentially be used in the food packaging industry.

Author Contributions

Material preparation and data collection were performed by Amenan Aline Doh and Yao Désiré Adjouman. Data analysis was performed by Dekker Matthis and Amenan Aline Doh. The first draft of the manuscript was written by Amenan Aline Doh. All authors contributed to the study conception and design.

Acknowledgments

The authors would like to acknowledge the funding from the Organisation for the Prohibition of Chemical Weapons (OPCW) and the International Foundation for Science (IFS) for jointly supporting/funding the research project.

Funding

This project was supported by the Organisation for the Prohibition of Chemical Weapons (OPCW) and the International Foundation for Science (IFS) for jointly supporting/funding the research project (Grant 113_E_039855), 2020.

Disclosure

All authors have read and approved the final manuscript and provided comments on earlier versions of the manuscript.

Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

References

1. N. A. Angeline and L. L. Clement, "Communication Persuasive et Attitudes des Populations Urbaines Face à l'Interdiction des Sachets Plastiques en Côte d'Ivoire," *Journal Africain de Communication Scientifique et Technologique* 36 (2016): 4845–4860.
2. I. B. Basumatary, A. Mukherjee, V. Katiyar, and S. Kumar, "Biopolymer-Based Nanocomposite Films and Coatings: Recent Advances in Shelf-Life Improvement of Fruits and Vegetables," *Critical Reviews in Food Science and Nutrition* 62, no. 7 (2022): 1912–1935.
3. J. Guo, S. Dong, M. Ye, et al., "Effects of Hydroxypropyl Methylcellulose on Physicochemical Properties and Microstructure of K-Carrageenan Film," *Foods* 11, no. 19 (2022): 30–23.
4. Z. M. Adilah, B. Jamilah, and Z. N. Hanani, "Functional and Antioxidant Properties of Protein-Based Films Incorporated With Mango Kernel Extract for Active Packaging," *Food Hydrocolloids* 74 (2018): 207–218.
5. M. Mittal, R. Chaudhary, K. Phutela, M. Airon, and R. C. Singh, "Modeling and Performance Optimization of Starch-Based Biocomposite Films Using Response Surface Methodology," *Journal of Applied Research and Technology* 20, no. 4 (2022): 430–447.
6. M. O. Sobeih, S. Sawalha, R. Hamed, F. Ali, and M. P. Kim, "Starch-Derived Bioplastics: Pioneering Sustainable Solutions for Industrial Use," *Materials* 18, no. 8 (2025): 1762.
7. R. Ramesh, H. Palanivel, S. V. Prabhu, B. Z. Tizazu, and A. A. Woldeesemayat, "Process Development for Edible Film Preparation Using Avocado Seed Starch: Response Surface Modeling and Analysis for Water-Vapor Permeability," *Advances in Materials Science and Engineering* 2021 (2021): 7859658.
8. N. Sienkiewicz, M. Dominic, and J. Parameswaranpillai, "Natural Fillers as Potential Modifying Agents for Epoxy Composition: A Review," *Polymers* 14, no. 2 (2022): 265.
9. FAOstat, "Food and Agriculture Organisation of the United Nations Statistical Database," in *Statistical Division*, (FAO, 2022), <http://www.fao.org/statistics/en/>.
10. I. Kong, J. B. Shang, and K. Y. Tshai, "Study of Properties of Coconut Fibre Reinforced Poly (Vinyl Alcohol) as Biodegradable Composites," *ARP Journal of Engineering and Applied Sciences* 11 (2016): 135–143.
11. E. P. M. Koffi, K. B. Koua, P. Gbaha, and S. Touré, "Utilisation de la Fibre de Coco Comme Isolant Thermique Dans un Chauffe-eau Solaire Thermosiphon," *Scientific African* 13 (2017): 275–286.
12. H. Hernando, Marpongahtun, E. Julianti, et al., "Impact of Glycerol on Oil Palm Trunk Starch Bioplastics Enhanced With Citric-Acid Epoxidized Palm Oil Oligomers," *Case Studies in Chemical and Environmental Engineering* 10 (2024): 100839.
13. C. Nindjin, M. Beyrer, and G. N. Amani, "Effects of Sucrose and Vegetable Oil on Properties of Native Cassava (*Manihot esculenta* CRANTZ) Starch-Based Edible Films," *African Journal of Food, Agriculture, Nutrition and Development* 15, no. 69 (2015): 9905–9921.
14. N. M. T. Kouamé, C. B. Aké, A. Mangaré, and K. N. 'Guessan, "Analyse de l'Intérêt Socio-Économique des Graines de, *Garcinia kola*, Heckel (Clusiaceae) Dans la Commune de Koumassi (Abidjan), Côte d'Ivoire," *International Journal of Biological and Chemical Sciences* 10, no. 6 (2016): 2587–2595.
15. F. Stephen, A. Awoyinka, O. Oluchi, et al., "Evaluation of the Nutraceutical Potential of, *Garcinia kola* Seed Oil," *Journal of Pharmacognosy and Phytochemistry* 6 (2007): 1894–1901.
16. A. A. Doh, Y. D. Adjouman, C. Nindjin, et al., "Antioxidant Activity and Efficacy of *Garcinia kola* (Bitter Kola) Oil on Pathogenic and Alteration Microorganisms of Attiékké, *Heliyon* 9, no. 11 (2023): e21152.
17. N. G. Amani, F. Aboua, D. Gnakri, and A. Kamenan, "Étude des Propriétés Physico-Chimiques de l'Amidon de Taro (*Xanthosoma sagittifolium*)," *Industries alimentaires et agricoles* 110 (1993): 136–142.
18. H. Y. Ungo-Kore, Y. K. E. Ibrahim, and B. A. Tytler, "Anti-Dermatophytic Activity of Hexane Extracts of, *Azadirachta indica* A. Juss," *African Journal of Microbiology Research* 13 (2019): 421–429.
19. M. Rokbi, H. Osmani, A. Imad, and N. Benseddiq, "Effect of Chemical Treatment on Flexure Properties of Natural Fibre-Reinforced Polyester Composite," *Procedia Engineering* 10 (2011): 2092–2097.
20. Y. D. Adjouman, C. Nindjin, F. A. Tetchi, A.-C. Dalcq, N. G. Amani, and M. Sindic, "Water Vapor Permeability of Edible Films Based on Improved Cassava (*Manihot esculenta* Crantz) Native Starches," *The International Journal of Food Processing Technology* 8 (2017): 665.
21. M. Bajić, H. Jalšovec, A. Travan, U. Novak, and B. Likozar, "Chitosan-Based Films With Incorporated Supercritical CO₂ Hop Extract: Structural, Physicochemical, and Antibacterial Properties," *Carbohydrate Polymers* 219 (2019): 261–268.
22. M. K. Kumaran, "Interlaboratory Comparison of the ASTM Standard Test Methods for Water Vapor Transmission of Materials (E. 96 95)," *Journal of Testing and Evaluation* 26, no. 2 (1998): 83–88.
23. O. V. López, M. A. García, and N. E. Zaritzky, "Film Forming Capacity of Chemically Modified Corn Starches," *Carbohydrate Polymers* 73, no. 4 (2008): 573–581.
24. S. Amariei, F. Ursachi, and A. Petraru, "Development of New Biodegradable Agar-Alginate Membranes for Food Packaging," *Membranes* 12, no. 6 (2022): 576.
25. ASTM, "Standard Test Method for Tensile Properties of Thin Plastic Sheeting," *The Journal of ASTM International* (2002): 162–170.
26. ISO 17556, *Determination of the Ultimate Aerobic Biodegradability of Plastic Materials in Soil by Measuring the Oxygen Demand in a Respirometer or the Amount of Carbon Dioxide Evolved* (ISO, 2019).
27. M. D. Hazrol, S. M. Sapuan, E. S. Zainudin, N. I. A. Wahab, and R. A. Ilyas, "Effect of Kenaf Fibre as Reinforcing Fillers in Corn Starch-Based Biocomposite Film," *Polymers* 14, no. 8 (2022): 1590.
28. M. I. J. Ibrahim, S. M. Sapuan, E. S. Zainudin, and M. Y. M. Zuhri, "Preparation and Characterization of Cornhusk/Sugar Palm Fibre Reinforced Cornstarch-Based Hybrid Composites," *Journal of Materials Research and Technology* 9, no. 1 (2020): 200–211.
29. A. Edhirej, S. M. Sapuan, M. Jawaid, and N. I. Zahari, "Cassava/Sugar Palm Fiber Reinforced Cassava Starch Hybrid Composites: Physical, Thermal and Structural Properties," *International Journal of Biological Macromolecules* 101 (2017): 75–83.
30. R. Y. Aguirre-Loredo, A. I. Rodríguez-Hernández, E. Morales-Sánchez, C. A. Gómez-Aldapa, and G. Velazquez, "Effect of Equilibrium Moisture Content on Barrier, Mechanical and Thermal Properties of Chitosan Films," *Food Chemistry* 196 (2016): 560–566.
31. E. S. Enidiok, D. O. Anakor, G. O. Erifeta, P. Thanikaivelan, and O. M. Oluba, "Development and Characterization of Chia Oil-Activated Ginger Starch-Feather Keratin Biocomposite for Prolonged Post-Harvest

Preservation of Tomato Fruits," *Carbohydrate Polymer Technologies and Applications* 7 (2024): 100464.

32. X. Ma, J. Yu, and J. F. Kennedy, "Studies on the Properties of Natural Fibres-Reinforced Thermoplastic Starch Composites," *Carbohydrate Polymers* 62, no. 1 (2005): 19–24.

33. S. Galus and J. Kadzińska, "Moisture Sensitivity, Optical, Mechanical and Structural Properties of Whey Protein-Based Edible Films Incorporated With Rapeseed Oil," *Food Technology and Biotechnology* 54, no. 1 (2016): 78–89.

34. A. M. Filho, M. V. A. Ventura, R. Favareto, C. F. S. Castro, M. B. Teixeira, and F. A. L. Soares, "Incorporation of Oil From *Hymenaea stigonocarpa* and *Hymenaea courbaril* Into Biofilms Made From Arrowroot Starch: Physicochemical, Biodegradability and Antifungal Activity," *Journal of Agricultural Science* 14, no. 4 (2022): 156.

35. K. Chala, "Elaboration et Caractérisation d'un Matériau à Base de Polyéthylène Basse Densité Recyclé et Chargé par le Talc," Doctoral Dissertation (2016).

36. C. Yacine and B. Bilal, "Effets du Vieillissement Thermique sur les Caractéristiques du Polyéthylène Basse Densité (PEBD)," (Université Mouloud Mammeri Doctoral Dissertation, 2018).

37. S. M. Bhasney, R. Patwa, A. Kumar, and V. Katiyar, "Plasticizing Effect of Coconut Oil on Morphological, Mechanical, Thermal, Rheological, Barrier, and Optical Properties of Poly (Lactic Acid): A Promising Candidate for Food Packaging," *Journal of Applied Polymer Science* 134, no. 41 (2017): 45390.

38. Y. Aisyah, L. P. Irwanda, S. Haryani, and N. Safriani, "Characterization of Corn Starch-Based Edible Film Incorporated With Nutmeg Oil Nanoemulsion," in *IOP Conference Series: Materials Science and Engineering*, 352, (IOP Publishing, 2018): 012050.

39. N. R. Draper and H. Smith, "Fitting a Straight Line by Least Squares," in *Applied Regression Analysis*, (John Wiley & Sons, 1998): 15–46.

40. V. Bagheri, B. Ghanbarzadeh, A. Ayaseh, et al., "The Optimization of Physico-Mechanical Properties of Bionanocomposite Films Based on Gluten/Carboxymethyl Cellulose/Cellulose Nanofiber Using Response Surface Methodology," *Polymer Testing* 78 (2019): 105989.

41. K. Hamadache, "Optimisation du Substrat de Culture pour *Quercus suber* L. Avec un Plan de Mélange et la Fonction de Désirabilité," *Annales de la Recherche Forestière en Algérie* 14 (2024): 67–76.

42. H. J. Cho, D. W. Lee, N. Marasini, et al., "Optimization of Self-Microemulsifying Drug Delivery System for Telmisartan Using Box–Behnken Design and Desirability Function," *Journal of Pharmacy and Pharmacology* 65, no. 10 (2013): 1440–1450.

43. Z. H. Kamaruddin, R. Jumaidin, R. A. Ilyas, M. Z. Selamat, R. H. Alamjuri, and F. A. M. Yusof, "Biocomposite of Cassava Starch-Cymbopogon Citratus Fibre: Mechanical, Thermal and Biodegradation Properties," *Polymers* 14, no. 3 (2022): 514.

44. R. A. Ilyas, S. M. Sapuan, R. Ibrahim, et al., "Thermal, Biodegradability and Water Barrier Properties of Bio-Nanocomposites Based on Plasticised Sugar Palm Starch and Nanofibrillated Celluloses from Sugar Palm Fibres," *Journal of Biobased Materials and Bioenergy* 14 (2020): 234–248.

45. N. L. Tai, R. Adhikari, R. Shanks, and B. Adhikari, "Aerobic Biodegradation of Starch–polyurethane Flexible Films Under Soil Burial Condition: Changes in Physical Structure and Chemical Composition," *International Biodeterioration & Biodegradation* 145 (2019): 104793.

46. A. I. Ghizdareanu, D. Pasarin, A. Banu, A. Ionita, C. E. Enascuta, and A. Vlaicu, "Accelerated Shelf-Life and Stability Testing of Hydrolyzed Corn Starch Films," *Polymers* 15, no. 4 (2023): 889.