Extraction and physical modification of Mango palmer kernel starch and its application in cookies

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Abstract

Introduction. The aim of this study was to extract, analyze and apply mango kernel starch (*Mangifera indica* L.) in cookies production.

Materials and methods. The starchy material was extracted using an aqueous medium, without chemical treatment, and cookies were produced with different amounts of modified mango seed starch. Determination of proteins, lipids, ash, and moisture was done according to AOAC standards. Thermogravimetry was performed to evaluate mass loss with heating, using TA-50 equipment. Thermal transitions and the gelatinization enthalpy of the starches were assessed using Differential Scanning Calorimetry. Scanning Electron Microscopy applied to observe the morphology of starch granules before and after modification.

Results and discussion The results of this study showed that the starch extracted from the seed kernel of Palmer mango exhibits distinct physico-chemical characteristics, with a higher content of carbohydrates and lipids compared to other starches, such as mango endocarp starch. Thermal analyses, including thermogravimetry and differential scanning calorimetry, indicated that the modified starch had greater thermal stability and higher gelatinization enthalpy, particularly in the 100% modified starch.

Scanning electron microscopy revealed differences in the morphology of the starch granules, with the modified starches showing significant changes compared to native starch, such as alterations in the shape and size of the granules. Colorimetric analysis showed that the modifications to the starch resulted in significant changes in color parameters, especially in luminosity (L*), where the modified starches exhibited a lighter color than the native starch.

These results indicate that the modifications made to Palmer mango starch lead to improvements in thermal and morphological properties, making it a potentially useful material for industrial applications.

Conclusions. The modifications made to the starch from Palmer mango seed significantly improved its thermal and morphological properties, highlighting its potential for diverse industrial applications.

Introduction

Interest in new materials derived from renewable sources, with low market value and low environmental impact, has grown significantly (Stabnikova et al., 2021). Starch is one of the raw materials with high potential for application in various fields, such as the food industry (Demiate et al., 2011; O'Leary et al., 2010), the oil industry (Cescon et al., 2018; Jing et al., 2019), the textile industry, the cosmetics industry, and the pharmaceutical industry (Guo et al., 2018). The identification of alternative sources of starch, such as the kernel of the mango, can also help to obtain new properties or contribute to the replacement of conventional sources (Garcia et al., 2020).

The *Mangifera indica* L. plant, better known as mango, is native to the forests of South and Southeast Asia, specifically India. Approximately 40 to 60% of waste is generated during mango processing, of which 12 to 15% corresponds to the peels and 15 to 20% to the seeds. It is estimated that 60% of the seed mass consists of starch (Kringel et al., 2020). Thus, mango kernels can be considered a non-traditional source of starch. The starch extracted from mango kernels is a potential food ingredient, and the process of its utilization itself makes it possible to turn waste into a valuable food product (Chowhan et al., 2020; Naqash et al., 2017; Pappen et al., 2020; Sousa et al., 2021a).

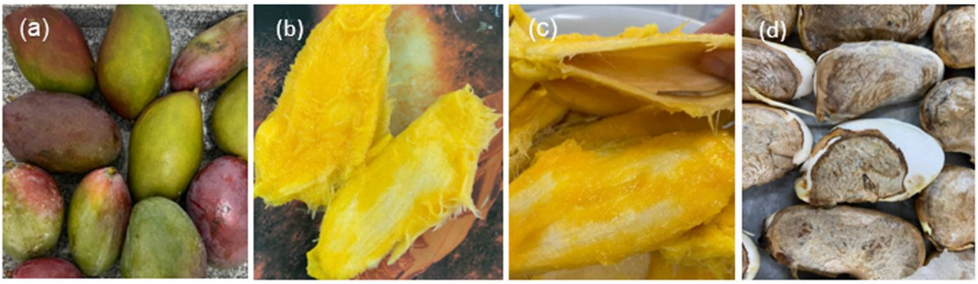

Figure 1. Morphology of the mango kernel:
a, whole mango, b and c, open core, and d, kernel

Given the ongoing challenge of developing gluten-free products, which remains a significant hurdle for the food industry (Naqash et al., 2017), there is a need for innovative solutions. Despite Brazil's substantial biscuit production, the availability of high-quality gluten-free options is limited due to difficulties in achieving sensory attributes comparable to conventional products (Pappen et al., 2020a).

The aim of this study was to extract, analyze and utilize starch from mango (*Mangifera indica* L.) kernels in cookies production.

Materials and methods

Materials

Mangoes of the Palmer variety were obtained from local markets in the region of Ponta Grossa, PR, in January 2023. The starchy material was extracted using an aqueous medium, without chemical treatment, using an adaptation of the methodology described by (Bet et al., 2018; Borsato et al., 2022).

Proximate analysis

Proximate analysis was performed following the official AOAC methodologies for proteins (920.87), lipids (968.20), ashes (923.03), and moisture (934.01). The carbohydrate content was calculated by difference.

Physical modification

The starch granules were modified using the Vibra Cell ultrasound equipment (SONICS), model USC 1400. The process followed the methodology outlined by Bisinella et al. (2022).

Thermogravimetry

The mass loss of each sample and the temperatures of each thermal event were obtained using the TGA-50 thermal analysis system (Shimadzu, Japan) with the aid of TA- 60WS software, for which it was also possible to obtain DTG values, which are a mathematical resource for a more precise identification of the temperatures involved in the mass loss of the samples.

The sample mass ranged from 7–9 mg weighed in an open alumina crucible. The analysis conditions were: heating from 30 to 650 °C at a heating rate of 10 ºC min^{-1}, under air flow of 100 mL min^{-1}. Derivative thermogravimetric (DTG) curves (first derivative of TG curves) were calculated. Before analysis, the instrument was preliminarily calibrated with standard weight and tested with standard calcium oxalate monohydrate (Bet et al., 2018; Borsato et al., 2022).

Differential Scanning Calorimetry

The DSC curves were obtained using a DSC-Q200 (TA-Instr., USA) thermal analysis system, with the following parameters: heating rate of 10 °C min^{-1} under airflow of 50 mL min^{-1}, and samples weighing about 2.5 mg. A suspension was prepared at a 4:1 ratio (water: starch, w/w) and held for 60 minutes to equilibrate the moisture content. The aluminum crucibles were sealed, and after one hour, the curves were performed. The instrument was previously calibrated with indium (99.99% purity, T_p = 156.6°C, ΔH = 28.56 J g^{-1}) (Beninca et al., 2020; Bisinella et al., 2022).

Field Emission Gun (FEG)

The morphology and measurements of the starch granules were performed in a Vega 3 (Tescan, Chech Rep.) scanning electron microscope (SEM), under acceleration voltage of 25 kV and 1.500 x magnification. Before analysis, the samples were metallised with gold. The area of granules was calculated using Image J 1.47 for Windows software (Beninca et al., 2020).

Preparation of the biscuits

For the preparation of the biscuits, the dough was prepared using the following ingredients: corn starch, margarine, sugar, coconut milk, water, and mango kernel starch (Nunes et al., 2015). Table 1 shows the proportions of the raw materials used in the shortbread formulations. It is clear that the main differences between the formulations are the percentage of corn starch, which was replaced by mango kernel starch, and the addition of pomace flour. The pomace flour corresponds to the fraction that remained in the sieve during the starch extraction, which was then dried in an oven with air circulation at 45 ºC for 24 hours (Silva et al., 2016).

Table 1

Biscuit formulations

Ingredients	S0	S1	S2	S3	S4
Corn starch	25	12.5	12.5	12.5	12.5
Margarine	12.5	12.5	12.5	12.5	12.5
Sugar	12.5	12.5	12.5	12.5	12.5
Coconut milk	0.1	0.1	0.1	0.1	0.1
Water	12.5	12.5	12.5	12.5	12.5
Mango kernel starch	-	10	4.68	2.5	12.5
Bagasse flour	-	2.5	7.82	10	-

S0, Control cookie with corn starch;
Cookies with corn starch replaced by:
S1, 40% bagasse flour and 10% mango kernel starch;
S2, 18.72% bagasse flour and 31.28% mango kernel starch;
S3, 10% bagasse flour and 40% mango kernel starch;
S4, 50% mango kernel starch.

Colour parameters

The MiniScan (Hunter) reflectance spectrophotometer was used to determine the colour parameters The equipment used was the portable colorimeter (Miniscan XE Plus, model 45/0-L, Hunter Associates Laboratory Inc., Reston, VA, USA) with CIE L*a*b* system.

Texture

Texture of cookies was measured with texturometer (TA.XT Plus Texture Analyser) by determining the hardness (peak breaking force) using a probe knife at a speed of 2 mm/s and distance of 20 mm through the cookies. Texture Exponent Lite software was used to evaluate the results.

Statistic

The results of the analyses were expressed as mean followed by standard deviation. Analysis of variance (Anova) and Tukey's test were used to compare the means between the samples with 95% confidence ($p < 0.05$).

Results and discussion

Proximate analysis

The results obtained for the proximate composition of native starch are presented in Table 2.

Table 2

Centesimal composition of mango kernel starch

Moisture	Ash	Lipids	Proteins	Carbohydrates
7.05±0.01	0.44±0.00	15.45±0.57	4.74±0.06	72.32±0.73

The value of carbohydrates and total fiber predominated in the proximal composition (72.32%), suggesting that the aqueous extraction allowed a good separation of the starchy fraction of the mango kernel. The starch had a high lipid content when compared to mango endocarp starch (Santos et al., 2015), which had 7.32%, and low levels of moisture (9.23%), ash (0.59%), and protein (5.30%).

Thermogravimetry

The TG/DTG curves (Figure 2) for native and modified starch allow an evaluation of the loss of mass and thermal stability of each sample during heating. Three stages of mass loss were observed for all samples, and they are associated with dehydration, decomposition, and oxidation, respectively. The derivative (DTG) was calculated to identify the temperature at which the maximum rate of loss occurs. When an inflection point in the TG curve is generated, these stages are replaced by peaks that delineate areas corresponding precisely to the changes in mass undergone by the sample.

Table 3 presents the TG/DTG results for native mango kernel starch and the 75% and 100% modified samples. Mass losses in the samples during thermal degradation occurred in three stages, as shown in Figure 3. The initial mass loss is attributed to the moisture content in the samples (Di-Medeiros et al., 2014; Ribeiro et al., 2014).

The second loss of mass, after a period of stability, corresponds to the decomposition of the main carbon chain. At this stage, there is a significant loss of mass, indicating the presence of a large number of compounds with similar thermal properties, a behavior characteristic of homopolysaccharides, to which starch belongs. In general, the second and third mass losses, which occur consecutively, are attributed to the decomposition and oxidation of organic matter to the formation of ash (Bet, 2021).

After ultrasound modification, a reduction in the thermal stability period of the starch was observed, especially when a higher vibration amplitude was applied. The reduction in stability may be associated with acoustic cavitation, which damages the structural order of the granules, facilitating the diffusion of water inside the chains and, consequently, the misalignment of hydrogen bonds (Bet, 2021).

The decomposition and oxidation of organic matter occur in consecutive stages during the second and third mass losses, as mentioned by Ribeiro et al. (2014). The lowest onset temperature of degradation was observed in the US100% starch, recording 244.65 °C, while the highest temperature was 249.55 °C for the native starch sample.

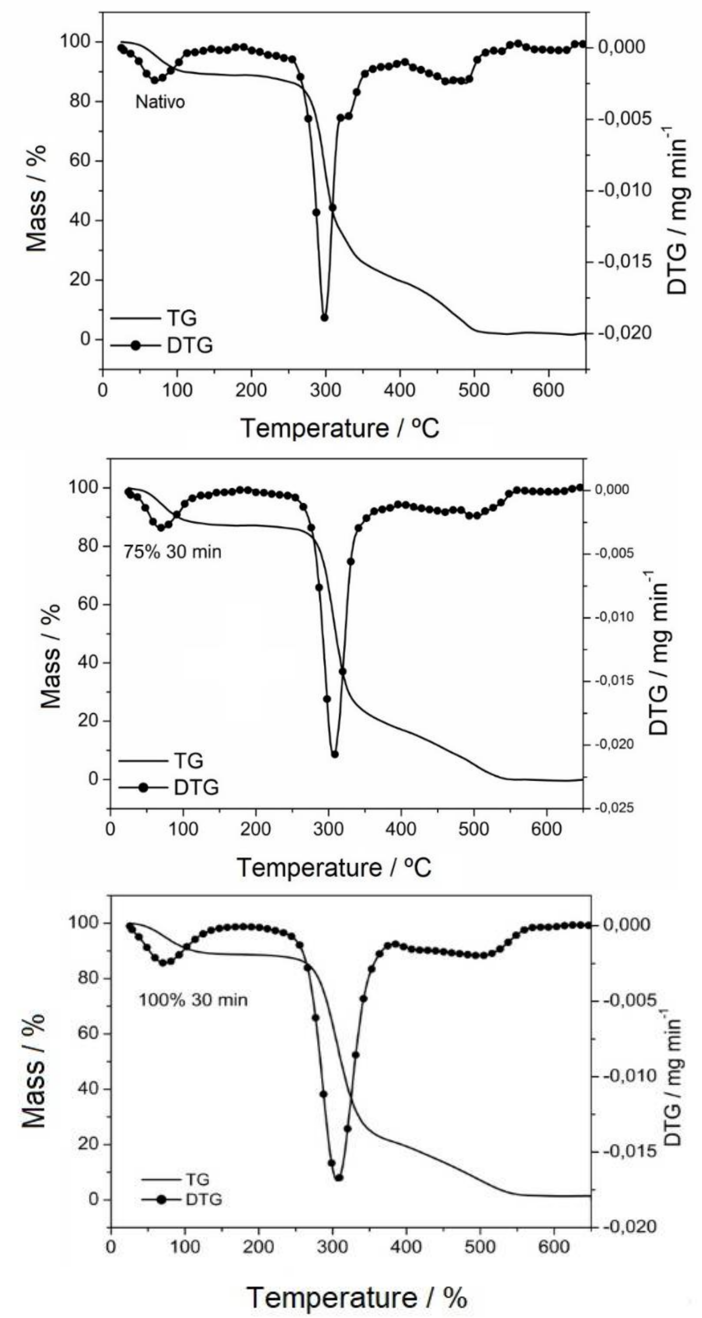

Figure 2. Thermogravimetry (TG) and Derivative Thermogravimetry (DTG) curves for the native starch, 75% modified starch, and 100% modified starch samples

Table 3

Results of TG/DTG for native and ultrasound-modified starch

Sample	Thermal event	1[a]	Stability	2[a]	3[a]
	Ti–Tf °C	30.00–131.59	131.59–249.55	249.55–376.24	376.24–534.77
Native starch	Tp °C	71.95	–	298.34	488.59
	Δm %	10.67	–	67.73	20.66
	Ti–Tf °C	30–133.37	137.37–248.85	248.85–379.93	379.93–576.56
Starch 75%	Tp °C	71.17	–	306.94	495.29
	Δm %	12.20	–	69.00	19.44
	Ti–Tf °C	30.00–133.94	133.94–244.63	244.63–382.43	382.43–567.01
Starch 100%	Tp °C	72.68	–	306.38	499.24
	Δm %	10.71	–	66.46	19.37

Δm = mass loss (%); Ti and Tf = initial and final temperatures (°C) for each stage of the thermal event; Tp = peak temperature (°C).

Differential Scanning Calorimetry

The differential scanning calorimetry (DSC) technique was used to study the gelatinization of starch, an endothermic process in which the ordered structure of the linear and branched starch chains passes into a disordered state, absorbing water and swelling the granules until they break. Consequently, this will lead to a change in enthalpy depending on the degree of disorder of the polymer chain. Table 4 shows the DSC results for native and modified starch at 75% and 100%, respectively.

These values were lower than those found in the literature for conventional starch sources such as corn starch (Tp: 73.2 °C) and higher than cassava starch (Tp: 69.0 °C), and were higher than the non-conventional amaranth starches (66.7–68.1 °C) mentioned by Bet et al. (2018).

When more intense ultrasound parameters are applied, damage to the starch granules is possible, which can result in changes in crystallinity. Ultrasound-induced cavitation facilitates the entry of water into the starch structure, thus accelerating the gelatinization process and reducing the enthalpy energy associated with this event.

In this study, with the modification of the starch there was a reduction in the gelatinization transition temperatures and a greater increase in the gelatinization enthalpy of the 100% modified starch, these data corroborate the study by Bisinella et al. (2022). Figure 3 shows the curves of the native and modified starches.

Table 4

Results of Differential Scanning Calorimetry for native and modified starches

Sample	T_0	T_p	T_c	ΔH_{gel}
Native	69.22±0.08	72.96±0.08	78.36±0.33	4.65±0.01
Starch 75%	68.22±0.26	72.11±0.01	77.52±0.17	4.95±0.27
Starch 100%	67.19±0.52	71.88±0.11	77.87±0.15	8.62±0.26

To, Initial temperature;
Tp, Peak temperature;
Tc, Conclusion temperature;
ΔHgel, Enthalpy of gelatinization.

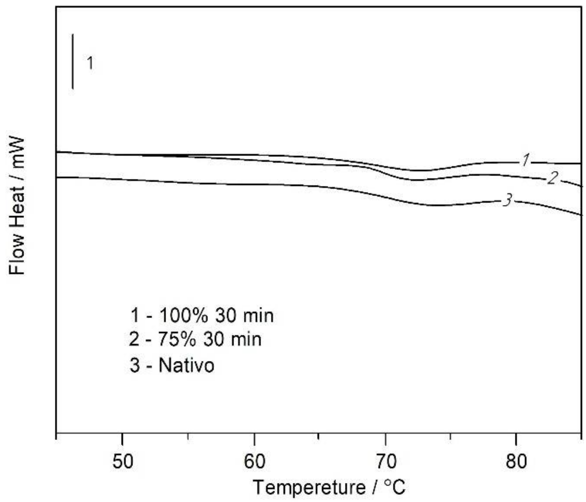

Figure 3. Differential Scanning Calorimetry curves for the native, 75%, and 100% modified starch samples

Morphology of starch granules

Using scanning electron microscopy, it was possible to check the morphology of the native and modified mango kernel starch granules (Figure 4). The starch showed elliptical granules and some spherical granules, which was expected, as observed in other studies (Espinosa-Solis et al., 2009; Santos, 2015).

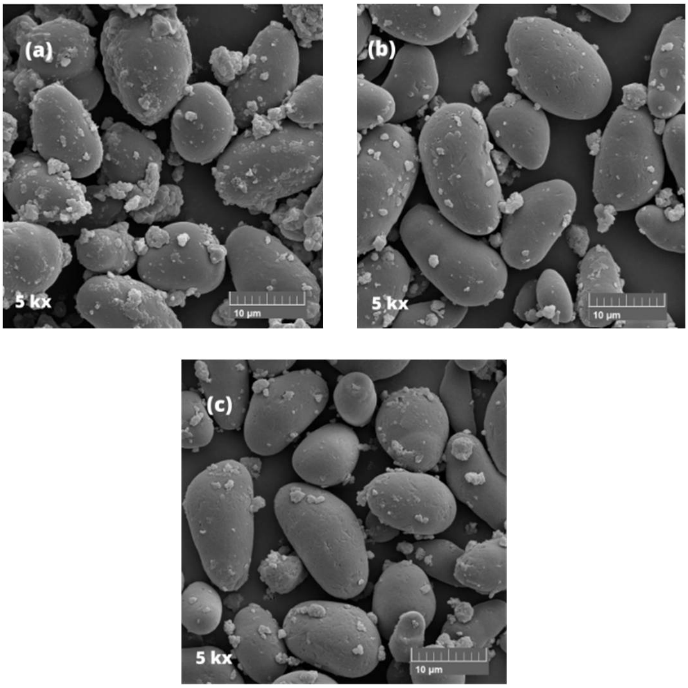

Figure 4. Microscopic images obtained by FEG for the samples: a, native starch; b, 75% modified starch; c, 100% modified starch.

Sandhu et al. (2004) reported that the diameter of mango starch is approximately 16 μm. Differences in starch morphology, hilum position and size can beattributed to different botanical origins, biosynthetic mechanisms of the amyloplast and plant physiology.

Colour parameters

The colorimetric parameters of native and modified starch are shown in Table 5.

Table 5

Colorimetric analysis of starch

Samples	L*	a*	b*
Native	61.73 ± 1.32^{c}	3.09 ± 0.12^{a}	19.54 ± 0.47^{a}
Modified starch 75%	73.71 ± 0.03^{a}	2.24 ± 0.03^{c}	16.64 ± 0.04^{b}
Modified starch 100%	69.59 ± 0.08^{b}	2.60 ± 0.04^{b}	17.06 ± 0.10^{b}
p	<0.001	<0.001	<0.001

Different letters in the same column represent significant differences according to Tukey's test (p<0.05).

The sample modified with a vibration amplitude of 75% showed greater luminosity. Compared to other studies, a* values were higher than those of starch and cassava bagasse, whose values were in the range -0.23 to 3.25, and the b* values ranged from 3.37 to 15.17 (Sandhu et al., 2004). With regard to b* values, the modified mango kernel starches did not differ significantly from each other and the highest value was found for the native starch.

Texture profile

Results of texture analysis are presented in Table 6.

Table 6
Texture analysis of the biscuits

Samples	Hardness (kgf)
S1	51.95 ± 1.36^{a}
S2	32.14 ± 3.59^{b}
S3	7.01 ± 1.98^{c}
S4	8.56 ± 0.09^{c}
S0	6.73 ± 0.32^{c}
p	<0.001

Control biscuit (S0), biscuit (S1) with 30% substitution of bagasse flour and 20% mango starch, biscuit (S2) with 45% substitution of bagasse flour and 30% mango starch, biscuit (S3) with 20% substitution of bagasse flour and 30% mango starch, and biscuit (S4) with 50% substitution of mango starch, ABC_{letras} different letters in the same column represent significant differences according to Tukey's test (p<0.05).

The highest hardness value was found in the sequilho-type biscuit formulation with a 30% substitution of bagasse flour and 20% mango starch (S1), followed by the biscuit formulation with a 45% substitution of bagasse flour and 30% mango starch (S2). The lowest values were observed when higher levels of mango starch were substituted in the biscuits (S3 and S4); however, no significant differences were found between these biscuits and the control biscuit (S0).

The hardness values obtained in the instrumental analysis ranged from 51.95 kgf to 6.73 kgf, with the lowest value for the control biscuit (S0). These values were higher compared to sour starch biscuits enriched with soluble and insoluble fibers, which ranged from 1.45 kgf to 3.90 kgf (Montenegro et al., 2008). When compared to whole biscuits made with residues from the extraction of pinhão starch, where the highest value found was 25.68 kgf and the lowest was 6.68 kgf, the results were slightly lower than the formulations (S2) and (S0) (Romko et al., 2019).

Since both sequilho-type biscuits and pinhão do not contain gluten, the starch present, along with their higher fiber content, contributes to water absorption, leading to a consequent reduction in hardness values (Romko et al., 2019).

Colour parameters of the biscuits

Colour parameters of the biscuits are shown in Table 7.

Table 7

Colorimetry analysis of the biscuits

Formulations	L*	a*	b*
S1	52.56 ± 1.55^{c}	6.39 ± 0.02^{b}	25.26 ± 1.05^{ab}
S2	53.05 ± 1.07^{c}	7.28 ± 0.19^{a}	29.88 ± 0.45^{a}
S3	48.27 ± 0.55^{d}	6.03 ± 0.05^{b}	21.80 ± 0.04^{bc}
S4	56.56 ± 0.49^{b}	5.07 ± 0.07^{c}	23.59 ± 4.54^{bc}
S0	91.62 ± 0.05^{a}	0.05 ± 0.05^{d}	18.79 ± 0.41^{c}
p	<0.001	<0.001	<0.001

S0, Control biscuit;
S1, Biscuit with 30% substitution of bagasse flour and 20% mango starch;
S2, Biscuit with 45% substitution of bagasse flour and 30% mango starch;
S3, Biscuit with 20% substitution of bagasse flour and 30% mango starch;
S4, Biscuit with 50% substitution of mango starch.

The biscuits showed lower luminosity values compared to gluten-free "Biju" biscuits, which ranged from 67.99 to 72.85 (Ressutte et al., 2018), with the control biscuit (S0) having the highest luminosity. According to Esteller et al. (2005), lower L* luminosity values indicate less light reflectance, resulting in products with a darker coloration. All samples differed from each other, with the darkest coloration observed in the S3 formulation (Figure 5).

The S2 formulation exhibited the highest a* value (shift towards red), indicating a darker biscuit crust compared to gluten-free "Biju" biscuits, which ranged from 1.09 to 2.24 (Ressutte et al., 2018). In terms of the b* value, the S2 formulation was lower compared to biscuits made with pupunha flour, which ranged from 55.14 to 59.18 (Santos, 2022). The biscuit with 45% substitution of bagasse flour and 30% mango starch (S3) showed the highest b* value, indicating a stronger yellow hue.

In the samples subjected to baking, the breakdown of starch upon heating and swelling due to water exposure may have triggered the Maillard reaction, leading to a tendency for enzymatic browning and caramelization (Gama et al., 2010; Lohinova and Petrusha, 2023).

There is a noticeable color difference among the biscuits. The control biscuit (S0) exhibits a lighter color compared to the other biscuits (S1, S2, S3, S4), which are darker due to the addition of mango almond starch and bagasse flour. Additionally, darker granules can be observed in the biscuits (b, c, d), which contain the added mango almond bagasse flour.

In contrast, the S4 biscuit (e) shows no granules and has a lighter color compared to biscuits S1, S2, and S3 (b, c, d), as its formulation included 50% mango almond starch. Table 6 shows the results obtained from the centesimal analyses of the S0, S1, S2, S3 and S4 cookies.

Figure 5. Baked sequilho-type biscuits:
S0, Control biscuit;
S1, biscuit with 30% substitution of bagasse flour and 20% mango starch;
S2, biscuit with 45%; substitution of bagasse flour and 30% mango starch;
S3, biscuit with 20% substitution of bagasse flour and 30% mango starch;
S4, biscuit with 50% substitution of mango starch.

Physico-chemical characteristic of the cookies are shown in Table 8.

Table 8

Physico-chemical characteristic of the cookies

Cookies	Moisture	Proteins	Ash	Lipids	Carbohydrates
S0	3.78±0.02	0.39±0.08	0.06±0.03	7.95±1.06	87.81±1.16
S1	7.49±0.57	1.69±0.02	0.39±0.08	11.06±1.34	79.35±0.7
S2	7.16±0.66	2.57±0.30	0.48±0.01	15.27±0.64	74.82±0.02
S3	3.57±0.001	1.58±0.06	0.08±0.07	11.07±0.76	83.69±0.76
S4	6.66±2.27	1.58±0.007	0.09±0.01	11.63±0.07	79.95±2.29

Control cookie with corn starch (S0); and cookies with corn starch replaced by: (S1) 40% bagasse flour and 10% mango kernel starch; (S2) 18.72% bagasse flour and 31.28% mango kernel starch; (S3) 10% bagasse flour and 40% mango kernel starch; and (S4) 50% mango kernel starch.

It was observed that cookie S3 had the lowest percentage of moisture, while cookie S1 had the highest. After adding bagasse and mango kernel starch, there was an increase in protein, ash and lipid content.

When the cookies formulated in this study are compared to sequilla-type cookies formulated with amaranth, corn and rice flour (Pappen et al., 2020b), the cookies with mango kernel starch had a higher carbohydrate content in all formulations. In addition, they had lower lipid and protein content when compared to sequilla-type cookies with flour from the residual pequi pulp cake, which registered values between 14.72 and 18.54%; and from 5.37 to 9.18%, respectively (Sousa et al., 2021b).

The highest hardness (51.95 ± 1.36) kgf was found for cookie S1, with 40% bagasse flour and 20% mango kernel starch, a combination that increased the cookie's compressive strength. Cookie S2 (18.72% bagasse flour and 31.28% mango kernel starch) also showed high hardness (32.14 ± 3.59 kgf), although significantly lower than S1.

On the other hand, in formulations S3 and S4, the addition of mango kernel starch resulted in cookie hardness values (7.01 ± 1.98 kgf and 8.56 ± 0.09 kgf, respectively) with no significant difference compared to the control cookie containing corn starch (6.73 ± 0.32) kgf. This suggests that increasing the proportion of mango starch contributes to a softer texture in the cookies.

When compared with wholemeal cookies made with the residue from the extraction of pine nut starch, which showed hardness values of 6.68 kgf to 25.68 kgf it can be seen that the S2 and S0 formulations in this study are similar or slightly higher (Romko et al., 2019).

The absence of gluten in sequilhos and pine nut cookies, together with the higher fiber content, influences water absorption and the reduction of hardness values (Ronko et al., 2020). This shows that the texture of cookies can be significantly adjusted by modifying the proportions of mango pomace flour and mango starch, offering variations that can meet the different texture preferences of consumers.

Conclusions

The starch from the Palmer mango seed (*Mangifera indica* L.) has excellent nutritional characteristics and is an important source of energy, being of great interest to the food industry. The modified starch exhibited morphology with elliptical and some spherical granules.

Through thermal analysis, it was observed that thermal stability allows application in baked bakery products. The development of "sequilhos" type cookies using mango seed kernel starch proved to be feasible, showing a proximate composition that confirms its nutritional value as a source of protein, carbohydrates, and lipids, making it suitable for use in formulating new food products. It is worth noting that in formulations with a 50% substitution of mango starch, the cookies had a lighter color compared to those containing bagasse in their formulation.

The development of biscuits using mango seed starch proved to be viable, presenting a proximal composition that enhances its nutritional value as a source of proteins, carbohydrates and lipids, making it suitable for use in the formulation of new food products. It is worth mentioning that in the formulations with 50% replacement of mango starch, the cookies were lighter in color compared to those that contained pomace in their formulation and were softer. The effect of replacing corn starch with mango seed starch in the production of cookies seems very promising and could be the subject of new research and future studies, to add value to waste obtained during mango processing.

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