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Physicochemical and techno-functional characterization of fibers and powders from leaves of stubble of two cultivars (MD2 and Smooth Cayenne) of pineapple (*Ananas comusus* L.)

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The aim of this work was to characterize the physicochemical and techno-functional properties of the fibers and powder obtained from the leaves of the two pineapple cultivars, MD2 (MD) and Smooth Cayenne (SC), to investigate their potential as a food ingredient in the development of new food products. Proximal chemical analysis shows that all evaluated materials have a low moisture content between 8.61 and 9.70% and a fat content between 0.06 and 1.04%, while the protein content varies between 5.32 and 8.36% and the ash content is higher in the powders than in the fibers (7.37 to 7.16 and 5.62 to 5.41, respectively). In addition, the samples have a high content of carbohydrates such as cellulose, hemicellulose, lignin, pectin, gum, wax and dietary fiber, which were identified by X-ray diffraction. The samples are acidic with a pH around 4.8. The results show that the fibrous (F) and the powder (P) materials have a swelling water capacity (SWC) between 16.14 and 26.90 mL g^{-1}, an oil retention capacity (ORC) between 2.95 and 7.26 g g^{-1}, a water absorption index between 8.20 and 13.25 g g^{-1} and a high and low water solubility index (WSI) between 0.040 and 0.060 g g^{-1}. On the other hand, the color is similar for the fibers (FMD–FSC) and slightly different for the powders (PMD–PSC). Scanning electron microscopy shows that the fibers have a micrometric thickness of 5 to 10 μm and the powders have a particle size of 5 to 50 μm. X-ray fluorescence shows that the ash powder contains important minerals for the human body, such as K, Ca, Cl, P, S, Mg, Si, and Mn. The carbohydrates have an influence on the techno-functional and pasting properties of the powder as well as on the hydrophilic properties, which are reflected in the high peak viscosity. This fact can confirm and improve the prospects for its use as an ingredient in the food industry, following a circular economy approach, since the industry considers it as waste without value.

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1. Introduction

Pineapples are one of the most produced and consumed tropical fruits in the world. As a result, their production generates large amounts of agricultural waste known as stubble. They are routinely discarded and 200 to 250 tons of stubble are generated per hectare. For this reason, it is important to look for alternatives to reuse. Mexico is one of the 10 largest producers of pineapple (*Ananas comosus* L.).[1] Cuttings propagate the plant, *i.e.*, a vegetative structure that can detach from a plant and produce a new plant. Between 2018 and 2023, the state of Veracruz, the main producing state, increased its production by 32%. As a result, the amount of agricultural waste has also increased, *i.e.*, the plants that have already produced fruit and seedlings that are not used to propagate the plant; this waste is known as pineapple stubble. It is estimated that 200 to 250 tons are produced per hectare.[2,3] This stubble must be removed to start a new cycle either by burning it or composting it in the field. However, both systems pose an environmental problem[4,5] as the stubble is an excellent substrate for the development of pests such as the stable fly (*Stomoxis calcitrans*),[6] which is why alternatives for the management of this stubble need to be developed. The current trend is to look for alternative uses for these residues to fulfill the concept of circular economy.

In this context, Mishra *et al.*[7] and Todkar & Patil[8] reported that pineapple straw contains cellulose similar to cotton fibers. The cellulose in pineapple straw could provide considerable mechanical strength so that it can be used to produce yarns with other natural or synthetic fibers, especially

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with cotton for the development of ecological natural fibers. In addition, pineapple stubble contains an average of 30–32% cellulose, 12–26% hemicellulose, 7–9% lignin and soluble monosaccharides which are a potential feedstock for fuel production.[6,9] In addition, Saini *et al.*[10] reported the production of xylo oligosaccharides (XOs) from pineapple leaves which are considered prebiotic because they regulate gastrointestinal flora, improve the absorption of nutrients, neutralize free radicals and lower cholesterol levels.[11]

In China, the leaves of the pineapple crown are traditionally used to treat gastrointestinal complaints such as indigestion, diarrhea and dysentery. This is attributed to the high content of phenolic compounds (2-methoxy-4-vinylphenol) and antioxidants, which modulate intestinal motility and reduce inflammation.[12] Hu *et al.*[13] demonstrated that consumption of pineapple leaf extract did not cause maternal toxicity or fetal teratogenic effects on appearance and skeleton in Wistar rats. However, Xie *et al.*[14] studied the effect and mechanisms of action of pineapple leaf phenol (PLP) on liver lipid metabolism in mice fed a high-fat diet. They found that PLP consumption inhibited fatty liver formation and promoted fat oxidation in liver mitochondria by carnitine palmitoyl transferase-1. De Aquino Gondim *et al.*[15] evaluated the chemical composition of the leaves of seven commercially available pineapple varieties and found that their composition in terms of threonic acid, quinic acid, malic acid, citric acid, phenylalanine, fructose-phenylalanine, hydroferuloyl-glucose, (*E*,*E*)-*N*,*N*′-diferuloilespermidina, ananaflavoside B and tricin is similar, but their mineral content is significant, and they showed through a cytotoxicity study that their consumption poses no risk. In addition, Ugbogu *et al.*[16] studied the antidiarrheal, anti-inflammatory and analgesic effects as well as the toxicity profile of the aqueous leaf extract of *Ananas comosus* (AACLE) in rats. They identified bioactive compounds such as 2-methoxy-4-vinylphenol, *n*-hexadecanoic acid, *n*-heptadecanol-1 and hexadecanoic acid methyl ester as potential anti-inflammatory agents. In addition, they concluded that the leaf extract is non-toxic and has hepatoprotective, antioxidant, anti-androgenic and hypo-cholesterolemic effects.

As mentioned above, pineapple leaves have been shown to be a source of non-cytotoxic biologically active chemical species, a source of prebiotics and an environmental problem if not disposed of properly. The aim of this work is to investigate the composition, physicochemical and techno-functional properties of the stubble of two pineapple cultivars and to determine the structural and chemical differences in order to utilize them as a potential functional, high-value food additive. The evaluation of techno-functional properties is crucial to determine how the material behaves when incorporated into food matrices. High water absorption index (WAI) and swelling capacity (SWC) indicate the potential to improve texture, juiciness and moisture retention in products such as baked goods or meat analogs, potentially extending shelf life. Oil retention capacity (ORC) indicates the ability to bind fats, which could influence mouthfeel and fat stability in emulsions and possibly modulate fat absorption during digestion. A low water solubility index (WSI) is desirable for ingredients that are intended to provide volume and texture without dissolving too much and are therefore suitable for applications such as fiber fortification in solid foods. In addition, the evaluation of proximate composition (proteins, fiber, and minerals) and physicochemical properties (pH, color, and texture) is crucial for assessing their suitability as food ingredients. These parameters determine not only their nutritional value (*e.g.* mineral content and low fat content) and safety (*e.g.* reduced water activity), but also their behavior in food matrices.

2. Materials and methods

2.1. Materials

Two different pineapple varieties named MD2 (Fig. 1a) and Smooth Cayenne (Fig. 1b) were harvested on August 5, 2024, in the city of Juan Rodriguez Clara, Veracruz, Mexico, and processed immediately.

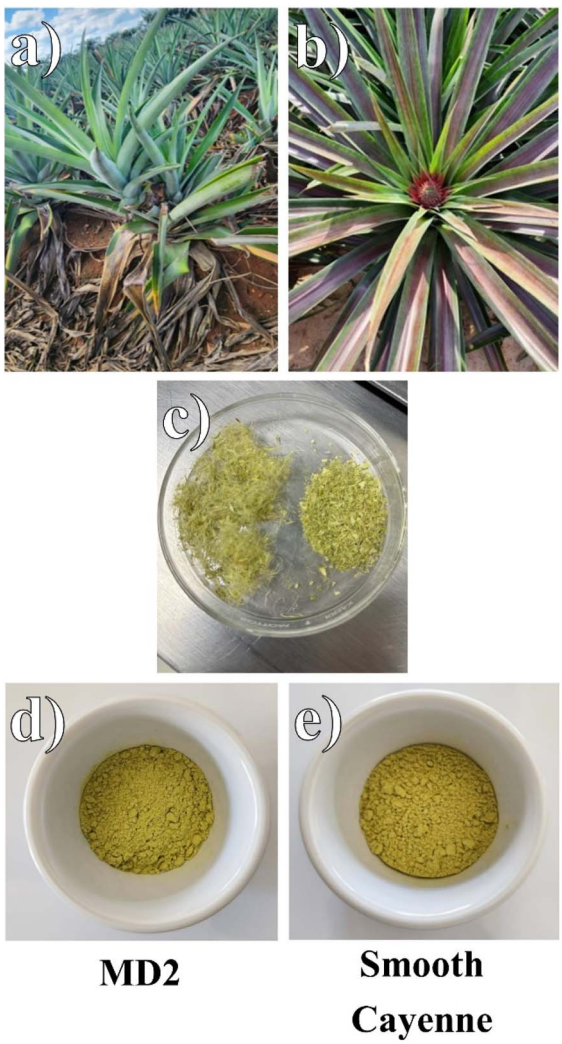

Fig. 1 (a) Pineapple plant of MD2. (b) Pineapple plant of Smooth Cayenne. (c) Fiber (left) and powder (right) after grinding the pineapple plant. (d) MD2 and (e) Smooth Cayenne powder samples.

2.2. Sample processing

After collecting the pineapple stubble leaves (seedlings/sprouts) (see Fig. 1a and b), they were washed with tap water and neutral soap and then cut into small pieces, 2 cm long, to be crushed with a Thermomix blender (USA) at speed 7 (4400 rpm for 30 s) and then dried at 50 °C for 3.5 h in an Excalibur air dryer (USA). The upper phase corresponds to the fibers (F) or fibrous materials/pineapple leaf fibers referred to as Fiber MD2 (FMD) and Fiber Smooth Cayenne (FSC) (see Fig. 1c), while the lower phase corresponds to the powder that was sieved with a mesh size of 100 (149 μm) to obtain samples referred to as Powder MD2 (PMD, Fig. 1d) and Powder Smooth Cayenne (PSC, Fig. 1e). The samples were stored in a vacuum plastic bag at room temperature and in the dark until use.

2.3. Chemical proximate analysis

The analysis of FMD, FSC, PMD and PSC samples was performed in triplicate according to the methods of the Association of Official Analytical Chemists.[17] Moisture, protein, fat and ash were assayed through methods 925.10, 954.01, 920.29 and 923.03, respectively. The protein content was calculated from $N \times 6.25$ and the carbohydrate content was determined from the difference. The pH values were determined according to Lario *et al.*[18] with modifications by direct measurement on slurry, ground samples (0.5 ± 0.05 g) with 10 mL distilled water.

2.4. Swelling water capacity (SWC)

Swelling water capacity (SWC) was evaluated according to Vilela *et al.*[19] with modifications. Each sample (0.5 ± 0.05 g) was hydrated with distilled water (35 mL) in a 50 mL Falcon tube at room temperature. The suspensions were stirred for 120 minutes at 25 °C in a centrifuge (200 rpm). After equilibration for 18 hours, the bed volume was recorded and the SWC value was calculated as the volume/dry mass (mL g^{-1}) of the sample.

2.5. Oil retention capacity (ORC)

The oil retention capacity (ORC) was determined according to the method of Mallek-Ayadi *et al.*[20] with modifications. Ground samples (1 ± 0.05 g) were mixed with cannoli oil (10 mL), vortex shaken for 3 min, centrifuged at 9000×*g* for 20 min at 4 °C and the excess supernatant was decanted. The ORC was expressed as g oil/g dry sample.

2.6. The water solubility index (WSI) and water absorption index (WAI)

The water solubility index (WSI) and the water absorption index (WAI) were determined according to the method described by Pacheco *et al.*[21] with modifications; 1 ± 0.05 g of the sample (W_1) was weighed into a 50 mL Falcon tube and 12 mL of distilled water was added if it was powder and 20 mL of distilled water if it was fiber. The whole mixture was shaken in a vortex for 3 minutes until everything was homogenized. The samples were allowed to rest for 48 hours. The suspension was centrifuged at 10 000 rpm for 30 min at 25 °C. For the fibers, the supernatant was decanted into a constant weight aluminum tube (W_3) using a Pasteur pipette, the Falcon tube was drained into the aluminum tube until no more leaching was observed, then the aluminum tube was dried at 110 °C for 24 hours. The weight of the Falcon tube with the wet fiber ("wet gel") was determined (W_2) for the powder, after centrifugation, and the supernatant from the first Falcon tube was decanted into a second Falcon tube using a Pasteur pipette, then the first Falcon tube was drained into the second Falcon tube until no more leaching was observed and the "gel" formed in the first Falcon tube was weighed (W_2). The supernatant from the second Falcon tube was allowed to rest for 1 h. Then the second Falcon tube was centrifuged at 10 000 rpm for 10 minutes, the supernatant was transferred to a constant weight aluminum container (W_3) using a Pasteur pipette, and the "gel" formed in the second Falcon tube was weighed (W_4). The aluminum containers were dried at 110 °C for 24 hours and the weight of the dry sample was recorded (W_5). The water solubility index (WSI) and water absorption index (WAI) were calculated using eqn (1) and (2), respectively.

$$\text{WSI (\%)} = W_5/W_1 \times 100 \quad (1)$$

$$\text{WAI (\%)} = (W_2 + W_4)/(W_1) \quad (2)$$

2.7. Color measurement

The CieLab coordinates (L, a^* and b^*) of the samples were read using a colorimeter (Konica Minolta CM-600D, with D65 illuminant and observer at 10°), calibrated with a standard white plane.[22] The samples were placed in clean transparent tubes before measuring the color; L means lightness and ranges from 0 (black) to 100 (white). A negative value of a^* indicated green, while a positive value indicated red color. A positive value of b^* indicates yellow and a negative value denotes blue color. Hue and chroma were calculated from the a^* and b^* according to eqn (3) and (4), respectively.

$$\text{Hue} = \text{Tan}^{-1}(b^*/a^*) \quad (3)$$

$$C = \sqrt{(a^*)^2 + (b^*)^2} \quad (4)$$

2.8. Morphological characterization: scanning electron microscopy (SEM)

SEM images were obtained from a Scanning Electron Microscope (SEM) (JSM-6060LV, JEOL Tokyo-Japan) at high vacuum. The samples were mounted on SEM aluminum stubs and then coated with gold (100) in cathodic pulverization (Edwards RV5). The analysis was done with the use of a 20 kV electron acceleration voltage, and the samples were systematically observed with 1500–7000× magnification.[23]

2.9. Mineral composition: X-ray fluorescence (XRF)

Mineral compositions were obtained from an X-ray fluorescence (XRF) spectrometer, Bruker, S2 Puma, which used 20, 40 and 50 kV. A compressed tablet was made at 8 tons with a thickness of 1.2 mm using 0.250 ± 0.05 g for each sample. Its determination is performed in triplicate.

2.10. Structural properties: X-ray diffraction (XRD)

X-ray diffraction was used to determine the crystalline phases of ash and powder samples.[24] The samples were packed in an aluminum holder. An Anton Paar XRDynamic 500 diffractometers operating at 40 kV, 50 mA and a CuK_{α}-radiation wavelength of $\lambda = 1.5406$ Å was used. Diffractograms were recorded from 4 to 60° on a scale of 2θ and a step size of 0.02°.

2.11. Vibrational analysis by Fourier transform infrared (FT-IR) spectroscopy

The principal functional groups of powder samples of pineapple leaves were determined by using Fourier Transform Infrared (FT-IR) spectroscopy, within a range of wavenumbers from 4000 to 600 cm^{-1}. These measurements were made on an IR spectrophotometer (PerkinElmer, Spectrum Two) using ATR (Attenuated Total Reflectance).

2.12. Pasting properties

The apparent viscosity of PMD and PSC was determined according to Nieves-Hernández *et al.*[23] with modifications, using an Anton Paar rheometer (MCR-102; Austria) (Anton Paar, Graz, Austria). To prepare the samples, 0.5, 1.0 and 1.5 g of each powder were dissolved in distilled water to reach a final volume of 21 mL directly in the cell. The initial temperature of the system was 50 °C and held for one minute; it was then increased to 90 °C at a heating rate of 5.3 °C min^{-1} and held for 350 s, and finally the sample was cooled to 50 °C at the same rate and held for one minute. All samples were run at a constant frequency of 193 rpm.

2.13. Statistical analysis

The results are expressed as mean ± standard error of the mean (SE) ($n = 3$) unless otherwise stated. Statistical analysis was performed using one-way analysis of variance (ANOVA) followed by Tukey's post-hoc test at a significance level of $\alpha = 0.05$. All analyses were conducted using RStudio (version 4.2.2).

3. Results and discussion

3.1. Chemical proximate analysis

Table 1 shows the chemical proximate analysis of the tested samples. The moisture content of the samples varies between 8.6–9.7% and is similar for FDM and PSC samples ($P < 0.05$). The protein content is similar in the powder samples, but in the fiber samples (FM) it is almost 30% lower which is due to the fact that the fibers are composed of cellulose, hemicellulose and lignin,[8,25] with the percentage of protein accounting for about 6.9% in the whole leaf according to the literature.[5,6,26] The fat content, on the other hand, shows a different trend, as it is higher in the fiber samples than in the powders. This behavior can be explained as follows. When dry samples are ground in a blender, the speed at which this is done causes the fibers to separate from the leaf matrix, as can be seen in Fig. 1c. This blender causes a natural separation of the fibers and the powder. It is not a complete separation, as can be seen from the behavior of fat. There are residues of fat or wax on the outside of the fibers (Fig. 2d and 3h).[27] The percentage of fat in the fiber corresponds to the value reported by Chen *et al.*[6] The carbohydrate content results from the different percentages of proximate analysis. The results of the samples showed that they are rich in carbohydrates, which are found around 75% in powder and 80% in the fibers; and they have a higher dietary fiber value, which is higher than that reported by Zainuddin *et al.*[28] in MD2 plants immediately after harvesting the fruit. These carbohydrates consist of structural (or cell wall) carbohydrates dominated by cellulose and hemicellulose and non-structural (or cell content) carbohydrates, with a total fiber content.

3.2. Techno-functional properties

The pH value of the two varieties is slightly acidic (Table 2). These are not statistical differences, but the fact that pineapple cultivation requires soils with a pH of 4.5 to 6.5 for its development.[29] Other important findings in this work are the techno-functional properties of the samples. The swelling capacity (SWC) is higher in the fiber material than in the powder, and there are no statistical differences between the FMD and FSC varieties as in the powder (PMD and PSC). The value of SWC in powders is between four and five times higher than in plant flour such as chickpea, lentil, red lentil, white bean, quinoa, amaranth and oat,[30] these values are greater in FMD and FSC. The oil retention capacity (ORC) highlights the fibrous material. It is higher in FMD than in FSC, but the powders do not differ despite the variety. The ORC is slightly higher in PMD and PSC

Table 1 The chemical proximate of pineapple leaf samples of MD2 and Smooth Cayenne[a]

Variety samples	Moisture (%)	Protein (%)	Fat (%)	Ash (%)	Carbohydrates (%)
FMD	8.61 ± 0.05^{c}	5.91 ± 0.50^{b}	0.69 ± 0.02^{b}	5.62 ± 0.05^{b}	79.16 ± 0.09^{a}
PMD	9.70 ± 0.13^{a}	7.96 ± 0.48^{a}	0.12 ± 0.47^{c}	7.37 ± 0.12^{a}	74.84 ± 0.37^{b}
FSC	9.19 ± 0.09^{b}	5.32 ± 0.21^{c}	1.04 ± 0.06^{a}	5.41 ± 0.20^{b}	79.02 ± 0.43^{a}
PSC	8.65 ± 0.03^{c}	8.39 ± 0.21^{a}	0.06 ± 0.01^{c}	7.16 ± 0.05^{a}	75.74 ± 0.28^{b}

[a] FMD: fiber of MD2; PMD: powder of MD2; FSC: fiber of Smooth Cayenne and PSC: powder of Smooth Cayenne. *Different letters are significantly different between treatments ($P < 0.05$).

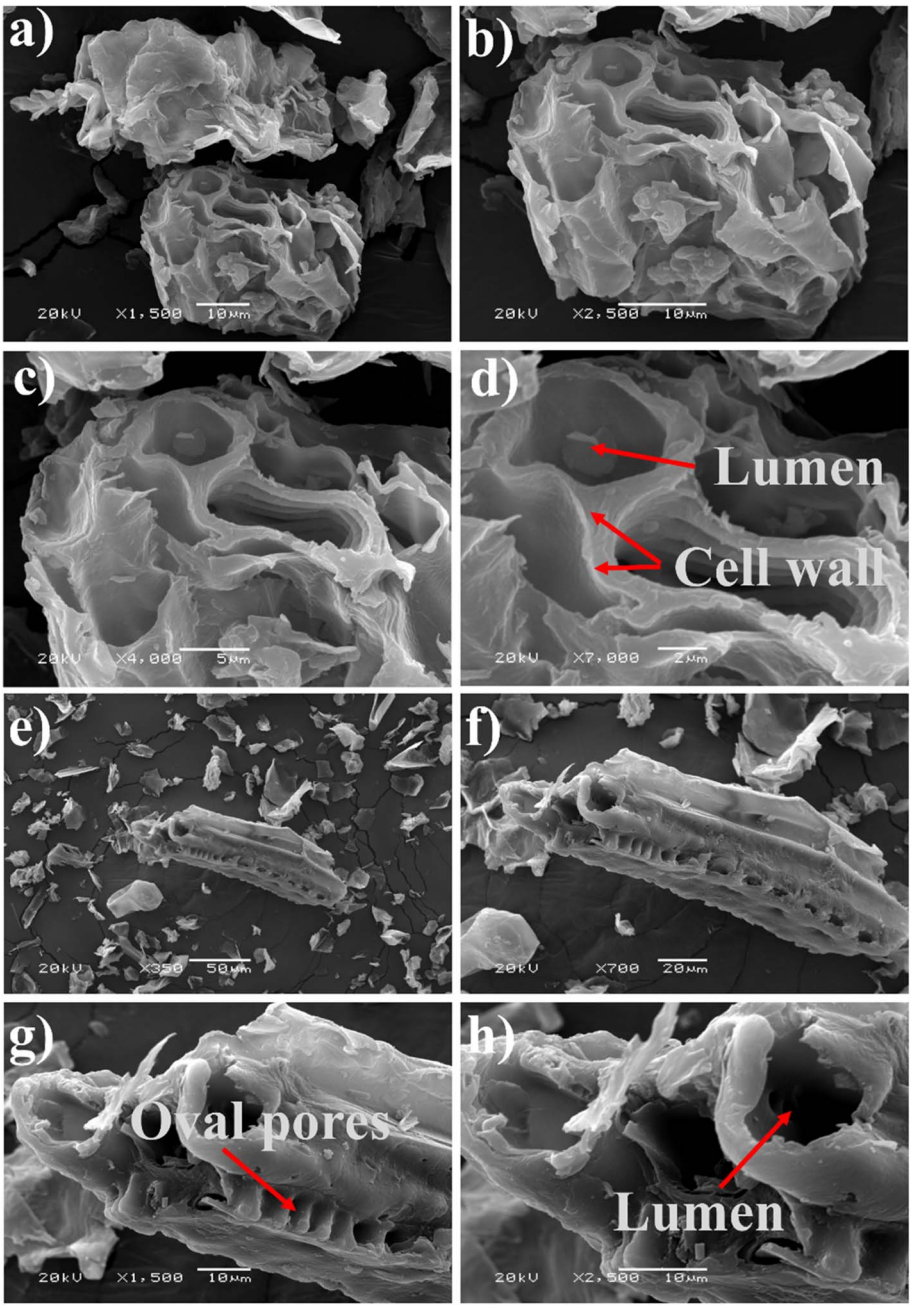

Fig. 2 Powder of MD2: (a) at 1500×, (b) at 2500×, (c) at 4000×, and (d) at 7000×. Powder of Smooth Cayenne: (e) at 350×, (f) at 700×, (g) at 1500×, and (h) at 2500×.

than in flours from fermented-lyophilized and fermented-dried lentil and quinoa.[31] The fibrous material has a better water absorption index (WAI) than the powder and they are similar despite the variety.

The WAI is related to hemicellulose, non-crystalline cellulose, lignin, surface morphology and the presence of free hydroxyl and polar groups in amorphous and crystalline surfaces, the presence of lumens and the effect of micropores (Fig. 2d, g and h), voids, crevices and holes in the fibers. The powder surface and SWC are related when the water concentration exceeds the threshold value due to the binding of water molecules to the polymer network *via* hydrogen bonds,[32] where the values of WAI and ORC are higher than those of other plant flours.[30] On the other hand, the four samples have low water solubility index (WSI). This index is a fundamental criterion for determining the quality of powder reconstitution and is important for determining product behavior in an aqueous phase,[33] which is why these samples are not recommended for products such as juices.

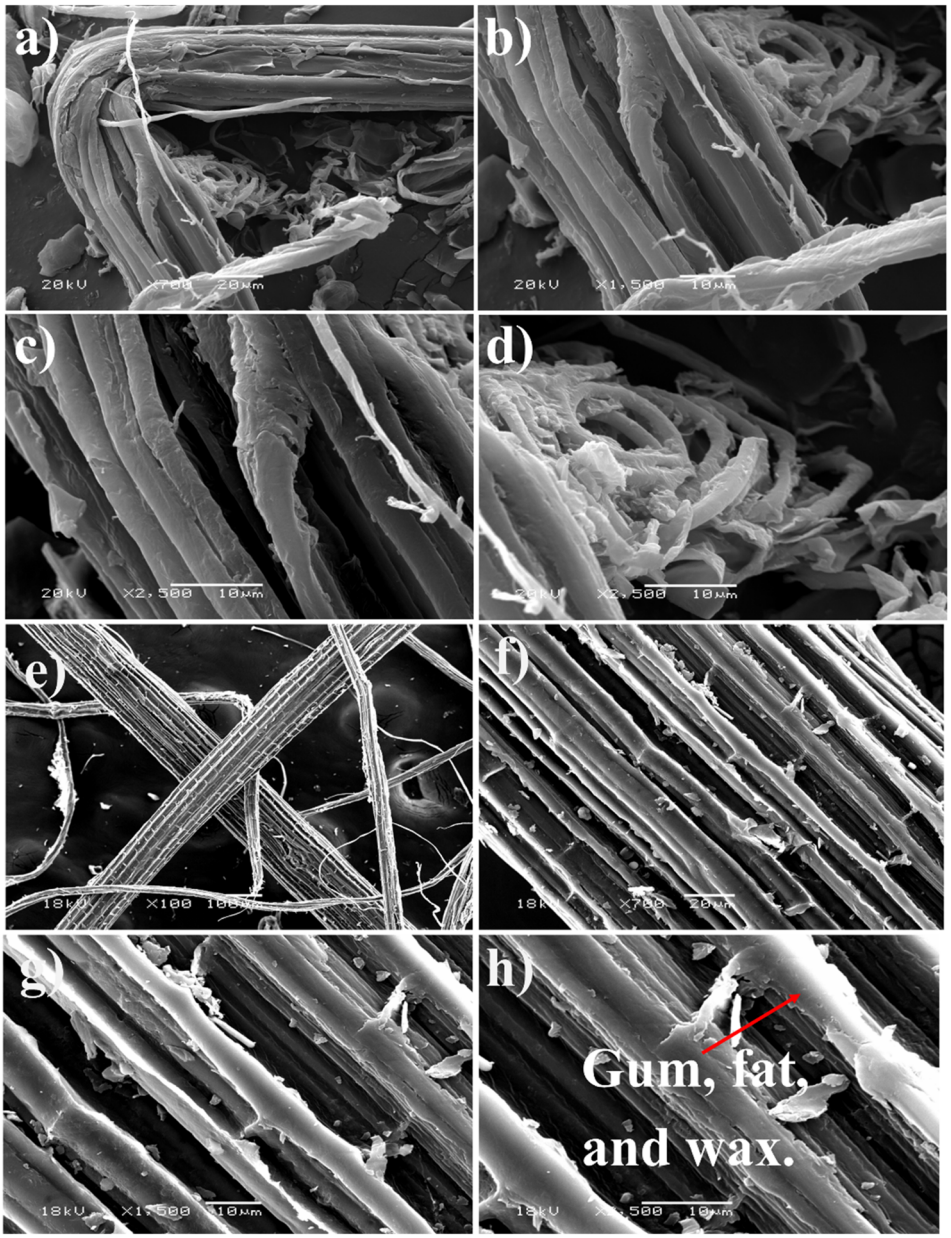

Fig. 3 Fiber of MD2: (a) at ×700 20 μm, (b) at 1500×, (c) at 2500×, and (d) at 2500×. Fiber of Smooth Cayenne: (e) at 100×, (f) at 700×, (g) at 1500×, and (h) at 2500×.

3.3. Color measurement

The color evaluation of fiber material (FMD and FSC) and powders (PMD and PSC) showed clear differences between the samples. FMD and FSC had lower values for lightness (L), greenness (a^*), yellowness (b^*) and chroma (C) compared to the powders (PMD and PSC), although FMD and FSC were not statistically different (Table 3), but it is important to note that pineapple leaves are sword-shaped, thin, inherently rigid and waxy[34] and Smooth Cayenne leaves have a spine at the edge and a purple coloration in the center of the leaf. These characteristics are not present in MD2 as it has no leaves with spines and the leaves are green. PMD was greener than PSC, which could be due to the purple hues of Smooth Cayenne. However, the chroma (C) was similar in both, and the hue was more intense in PMD than in PSC.

3.4. Morphological analysis by SEM

Fig. 2a–d shows the morphological structure of the powder of MD2 (PMD) and Fig. 2e–h shows the morphological structure of the powder of Smooth Cayenne (PSC). The powders are circular and straight and have a polygonal shape due to the lumen, the cell wall and the oval pores on the side.[35] The lumen is a cavity that is characterized when the cells lose their protoplasm and are dead and it has a functional cell wall that contributes to the

Table 2 Techno-functional properties[a]

Variety samples	pH	SWC (mL g^{-1})	ORC (g g^{-1})	WAI (g g^{-1})	WSI (g g^{-1})
FMD	4.86 ± 0.06^{a}	25.90 ± 0.96^{a}	7.26 ± 0.17^{a}	13.25 ± 0.12^{a}	0.04 ± 0.01^{b}
PMD	4.88 ± 0.05^{a}	16.62 ± 0.59^{c}	2.95 ± 0.15^{c}	8.93 ± 0.26^{c}	0.05 ± 0.01^{a}
FSC	4.86 ± 0.04^{a}	24.42 ± 0.49^{b}	6.66 ± 0.05^{b}	12.27 ± 0.32^{b}	0.04 ± 0.01^{b}
PSC	4.78 ± 0.01^{a}	16.14 ± 0.60^{c}	3.12 ± 0.11^{c}	8.20 ± 0.35^{d}	0.06 ± 0.01^{a}

[a] FMD: fiber of MD2; PMD: powder of MD2; FSC: fiber of Smooth Cayenne and PSC: powder of Smooth Cayenne. *Different letters are significantly different between treatments ($P < 0.05$).

Table 3 Color[a]

Variety samples	L	a^*	b^*	C	Hue
FMD	63.43 ± 2.09^{a}	-0.58 ± 0.50^{b}	20.45 ± 0.37^{a}	20.47 ± 0.39^{a}	131.58 ± 39.17^{b}
PMD	68.85 ± 0.53^{b}	-2.59 ± 0.18^{c}	25.74 ± 0.30^{b}	25.88 ± 0.28^{b}	95.75 ± 0.46^{b}
FSC	65.66 ± 1.72^{a}	1.01 ± 0.37^{a}	20.47 ± 0.43^{a}	20.51 ± 0.42^{a}	87.09 ± 1.12^{a}
PSC	69.95 ± 0.64^{b}	-1.05 ± 0.44^{b}	25.81 ± 0.07^{b}	25.84 ± 0.06^{b}	92.33 ± 0.98^{b}

[a] FMD: fiber of MD2; PMD: powder of MD2; FSC: fiber of Smooth Cayenne and PSC: powder of Smooth Cayenne. *Different letters are significantly different between treatments ($P < 0.05$).

mechanical strength of the plants.[35] These cavities in the powders may play an important role in the transportation of water and nutrients in the plants, which is related to the techno-functional properties.[36] In addition, the round shape (Fig. 2b) and straight shape (Fig. 2f) of the powders can be associated with the cells of the epidermis in the complete leaves of pineapple, as the epidermis is composed of two to three layers of thin-walled cells with elliptical, polygonal, elongated columnar or tapered shape[37] and it is important to remember that the powders obtained after grinding correspond to all parts of the leaves, except for the fibers (FSC and FMD).

In addition, Fig. 3 shows the fibers (FMD and FSC) corresponding to a bundle of elementary fibers,[35] which run in parallel bundles along the length of the pineapple leaf' and are distributed across the width.[38] In addition, the fiber bundles in natural and whole leaves of pineapple are concentrated in the central part and basal area.[37] However, the fibers contain residues of gum, fat and wax (Fig. 3a and e), which are often removed to be used in the materials industry.[27,32] Nevertheless, these fibers are not analyzed in the following results, as it might be difficult to add them to a food matrix due to their fibrous structure.

3.5. Ash mineral composition: XRF

A very important parameter in this work is the amount of minerals in the leaf of the pineapple in ash. The value of ash is higher in this work than in other reports.[25] Ash is the total inorganic major and minor minerals, which may be present in crystalline or amorphous phases. The mineral content determined by X-ray fluorescence is shown in Fig. 4a. These minerals are important for the potential use of pineapple leaves. K, Ca, Cl, P, S, Mg, Si, and Mn are the most important minerals, while Fe, Sr, Br, and Al are the less important minerals in these leaves. The content of K, Ca, Mg, and Mn is higher in PMD than in PSC, while this is different for Cl, P, Si, and Fe. Sr, S, and Br were similar in both powders, although Al was not detected in PMD.

P, K, Ca, and Mg are considered essential nutrients in pineapple cultivation and are supplied by chemical fertilizers.[39] K is important in the human body as it is essential for the osmolarity of cells, the transmission of nerve stimuli, the regulation of heart and muscle function and other parts of the body, while P is crucial for maintaining kidney function and elimination of waste products in the liver. It is also important for the production of stomach acid, which is essential for the absorption of other minerals and vitamins such as B12.[40] Mg plays a role in numerous processes, such as the regulation of Mg cell function, activation of enzymes, binding to organic substances (proteins, nucleic acids and nucleotides), involvement in the synthesis and replication of RNA and DNA and variety of metabolic processes such as oxidative phosphorylation and muscle contact, which is why a daily Mg intake of 300–400 mg for men and 270–310 mg for women is recommended.[41] For its part, Ca not only has benefits for bone health, but is also associated with lowering blood pressure, preventing hypertensive pregnancy symptoms, preventing colorectal adenomas and reducing low-density lipoprotein.[42] A daily calcium intake of 1000 mg per day for men and 1000–2000 mg per day for women is recommended.[43] On the other hand, P is present in the human body in the form of free ions $H_2PO_4^-$ and HPO_4^{2-} which are associated with various functional roles, such as acid–base balance, storage and energy transfer through metabolism and activation of proteins during phosphorylation.[43] In addition, P is an essential component of bone tissue followed by Ca.[44] In diet, the Ca and P are fundamental for good human nutrition as the intake of calcium and phosphorus in a 1–2 : 1 (Ca : P) ratio[44–46] is crucial for bone health. In this work, the intake Ca/P = 1.90 was higher in PMD than in PSC with an intake of Ca/P = 1.29.

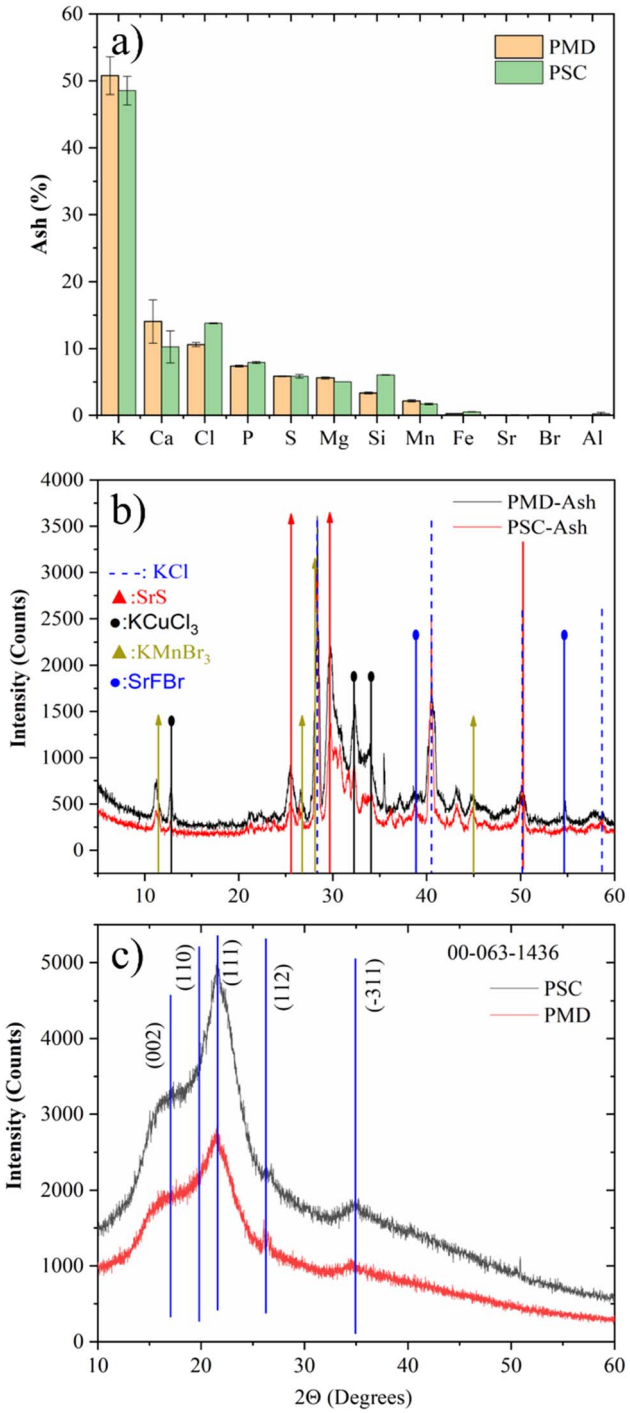

Fig. 4 (a) Concentration of minerals in ash powder of MD2 (PMD) and Smooth Cayenne (PSC). (b) X-ray diffraction patterns of the PMD-Ash (MD2 – ash powder) and PSC-Ash samples: (Cayena Lisa – ash powder). (c) X-ray diffraction patterns of PMD (MD2 powder) and PSC (Smooth Cayenne powder) samples.

3.6. Structural properties: XRD

Fig. 4b and c shows the X-ray patterns of ash from the powder of MD2 and PSC. After calcination at 550 °C, the ash is composed of minerals in amorphous and crystalline phases. This figure shows the crystalline phases in both ashes. With the help of the free PDF-4 software and the mineral content determined by X-ray fluorescence, at least 5 crystalline compounds could be identified: KCl with PDF # 00-041-1476, SRS with PDF # 00-008-0489, KCuCl3 with PDF # 00-020-0874, KMnBr3 with PDF # 00-027-0411, and SrFBr with PDF # 00-034-0658. Table 4 shows the 2θ coordinates of the patterns identified in the sample. These crystalline minerals match those identified in Fig. 4b. KCl represents patterns with greater intensity, which is due to the importance of potassium fertilizers in this crop. The K cation is of fundamental importance as it promotes the increase of soluble solids, titratable acidity and vitamin C content in the pineapple fruit. It also regulates the osmotic potential of the cells and activates enzymes involved in respiration, photosynthesis, regulation of metabolic processes and opening of the stomata, making the fruit more resistant to attacks by pathogens.[47] On the other hand, the X-ray diffraction patterns of PSC and PMD are shown in Fig. 4c. Both patterns correspond to cellulose II with low crystallinity. The continuous lines show the identification of the crystal structure of this cellulose with PF 00-063-1436. As expected, the powders are mainly composed of cellulose. These results are consistent with the reported X-ray diffraction profiles.[48]

3.7. Vibrational analysis: FT-IR

FT-IR spectroscopy allowed us to investigate the chemical structure by identifying the functional groups of PSC and PMD samples (Fig. 5). However, an interesting observation is that the intensity of the bands of PMD is stronger than that of PSC. In addition, 15 bands of some functional groups in the biomass components are identified in Table 5. The band at 3330 cm^{-1} is characteristic of cellulose and hemicellulose (–OH, binding vibration).[38] The bands at 1372, 1318, 1242, 1152, 897 and 770 cm^{-1} show the bending and stretching vibrations of cellulose, the bands at 1730, 1242 and 1039 cm^{-1} show the stretching vibrations of hemicellulose and the bands at 3330, 1598 and 897 cm^{-1} are associated with lignin (Table 5). These spectra show the characteristic absorption bands associated with cellulose, hemicellulose and lignin fractions, indicating their presence in the samples. In addition, in the leaves of Pérola pineapple crown, Braga *et al.*[49] described the 4000–2995 cm^{-1} region as the axial strain of the O–H hydrogen bond and the 896 cm^{-1} band as the movement of atoms C5 and C6 in cellulose. In addition, Chung *et al.*[50] identified bands at 2849 cm^{-1} originating from impurities such as waxes and pectins. Therefore, the O–H, CH_2–, and COOH groups could be the main binding sites of the powders and these give them their techno-functional properties.[51]

3.8. Pasting properties of pineapple powders

The pasting profile of a sample is influenced by intrinsic and extrinsic parameters: intrinsic parameters such as the chemical composition and extrinsic parameters such as the particle size, the method by which the sample was obtained, and others. Fig. 6 shows the properties of the pasting profile of PSC and PMD with different proportions (1.5/20.5, 2.0/19 and 2.5/18.5 g mL^{-1}) in

aqueous suspensions, as well as the thermal profile (*x*-axis). It can be seen that the viscosity increases when a larger amount of sample is used, as the powders reduce the free water in the sample and thus increase the viscosity. PMD 1.5 has a lower peak viscosity than PSC 1.5 when a larger amount sample of powder is used. This could be related to the fact that PSC has a higher proportion of cellulose, hemicellulose, lignin, pectin and wax than PMD, which gives them more hydrophilic properties[27] as the (–OH) group of these compounds (cellulose and hemicellulose) increases hydrophilicity by forming hydrogen bonds and increasing swelling capacity. For this reason, Fig. 6 shows a slowdown in viscosity between 0 and 800 s and an increase between 900 and 1100 s, which can be explained by the fact that powders need a certain time to fully hydrate and can delay water adsorption and swelling.[63] In addition, the increase in temperature affects the weakening of hydrogen bonds by breaking them, causing the viscosity to decrease and the powders to require more time to hydrate and become stable. This pasting profile shows that when these fibers are used as an additive, for example in baking, the workability of the dough can be improved at a concentration of more than 2.5%. And, of course, the product increases its insoluble content. The increase in heating is also

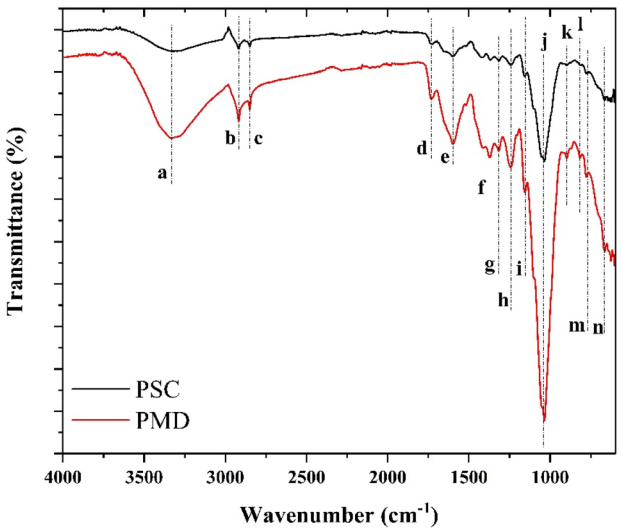

Fig. 5 FT-IR spectra of PMD (MD2 powder) and PSC (Smooth Cayenne powder) samples.

Table 4 Crystalline phases found in the powder of MD2 (PMD) and powder of Smooth Cayenne (PSC)

ICCD card (PDF)			Samples					
2θ	d (A°)	(hkl)	2θ	d (A°)	(hkl)	2θ	d (A°)	(hkl)
(00-041-1476) KCl			**Ash-PMD[a]**			**Ash-PSC[a]**		
28.345	3.146	(2 0 0)	28.366	3.146	(2 0 0)	28.349	3.148	(2 0 0)
40.507	2.225	(2 2 0)	40.591	2.222	(2 2 0)	40.503	2.227	(2 2 0)
50.169	1.816	(2 2 2)	50.134	1.819	(2 2 2)	50.115	1.820	(2 2 2)
58.640	1.573	(4 0 0)	58.681	1.024	(4 0 0)	58.665	1.573	(4 0 0)
(00-008-0489) SrS			**Ash-PMD[a]**			**Ash-PSC[a]**		
25.584	3.479	(1 1 1)	25.534	3.488	(1 1 1)	25.544	3.487	(1 1 1)
29.685	3.007	(2 0 0)	29.707	3.007	(2 0 0)	29.693	3.008	(2 0 0)
50.255	1.814	(3 1 1)	50.232	1.816	(3 1 1)	50.246	1.815	(3 1 1)
(00-020-0874) KCuCl3			**Ash-PMD[a]**			**Ash-PSC[a]**		
12.838	6.890	(0 2 0)	12.801	6.915	(0 2 0)	12.814	6.908	(0 2 0)
32.267	2.772	(1 0 2)	32.291	2.772	(1 0 2)	32.313	2.779	(1 0 2)
34.075	2.629	(0 5 1)	34.063	2.631	(0 5 1)	34.093	2.629	(0 5 1)
(00-027-0411) $KMnBr_3$			**Ash-PMD[a]**			**Ash-PSC[a]**		
11.453	7.720	(0 2 0)	11.425	7.744	(0 2 0)	11.437	7.736	(0 2 0)
26.749	3.330	(1 2 1)	26.724	3.335	(1 2 1)	26.785	3.328	(1 2 1)
28.126	3.170	(0 3 1)	28.114	3.173	(0 3 1)	28.239	3.160	(0 3 1)
44.973	2.014	(0 0 2)	44.942	2.016	(0 3 1)	44.999	2.014	(0 3 1)
(00-034-0658) SrFBr			**Ash-PMD[a]**			**Ash-PSC[a]**		
38.852	2.316	(1 1 2)	38.822	2.317	(1 1 2)	38.820	2.3197	(1 1 2)
54.651	1.678	(2 1 2)	54.619	1.679	(2 1 2)	NA	NA	NA
(00-063-143) cellulose II			**PMD[a]**			**PSC[a]**		
17.059	5.193	(0 0 2)	17.069	5.194	(0 0 2)	17.136	5.174	(0 0 2)
19.808	4.478	(1 1 0)	19.793	4.485	(1 1 0)	19.854	4.471	(1 1 0)
21.591	4.112	(1 1 1)	21.590	4.115	(1 1 1)	21.582	4.117	(1 1 1)
26.255	3.391	(1 1 2)	26.265	3.392	(1 1 2)	26.257	3.393	(1 1 2)
34.912	2.567	(3−1 1)	34.889	2.571	(−3 1 1)	34.911	2.569	(−3 1 1)

[a] This word (phases found in PMD and PSC).

Table 5 Assignments of IR bands for PSC and PMD samples

Functional group		Vibrational type	Wavenumber (cm^{-1})[a]	Wavenumber (cm^{-1}) (reference)	
A	O–H	–OH, bond vibrational strain	3330	3326 3330	52 and 53
B	Alkyl, aliphatic, aromatic	The axial deformation of the methylene group	2917	2917–2848	49
C	$–CH_2–$	$–CH_2$-symmetric stretching	2849	2849	50
D	R–COO–R C═O	Ester Hemicellulose acetyl and uranic ester group	1730	1730 1732	38 and 54
E	C–O	C O stretch in conjugated (lignin)	1598	1593–1609	55
F	C–H	C–H (cellulose)	1372	1375	56
G	CH_2	CH_2 wagging (cellulose)	1318	1315 1318	56
H	–COO C–O–C	Hemicellulose Cellulose chain and stretching of lignin aryl group	1242	1242 1239–1243	38 and 53
I	C–O–C, β-1,4 glycosidic linkage of cellulose	Cellulose Polysaccharides	1152	1159	38 and 55
J	C–O–C C═O	C–O–C stretching of glycosidic linkages of the xylan ester	1039	1034	57 and 58
K	C–O–C	Movements of the atoms C5 and C6 from cellulose β-glycosidic linkage of cellulose	897	896 895–897	49 and 59
L	Aromatic C–H deformation vibration of lignin	Lignin (conypheryl alcohol or guaiacyl units)	817	817	60 and 61
M	CH_2	CH_2 vibration in cellulose I_α	770	770	55
N	C–OH	C–OH bond	664	667	49 and 62

[a] This work.

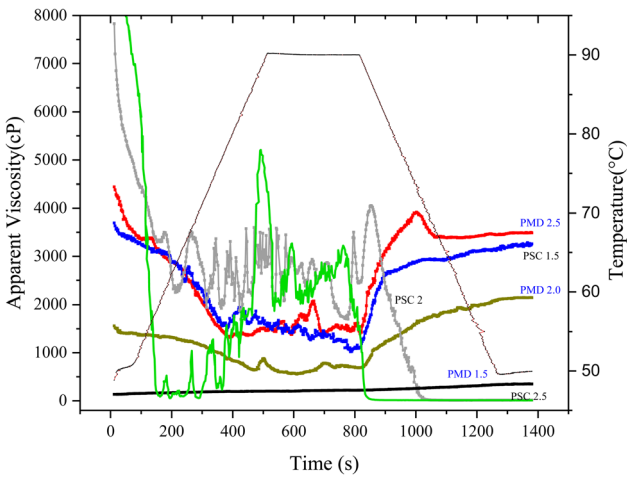

Fig. 6 Apparent viscosity of PMD (1.5, 2, and 2.5 w/w) powder of MD2 and PSC (1.5, 2, and 2.5 w/w) powder of Smooth Cayenne.

related to the presence of starch in these samples. The high values of the final viscosity of the slurries show that they behave like a pudding.

4. Conclusions

The minerals contained in the leaves of both pineapple varieties are K, CL, Ca, P, S, Mg, Si, Mn, Fe, Sr and Br, which are involved in a variety of processes in the human body. The carbohydrates consist mainly of cellulose, hemicellulose, lignin, pectin and wax, which can be detected by FT-IR vibrational analysis and X-ray diffraction, with bands corresponding to these functional groups and crystalline phases. These components impart techno-functional properties to the product. In addition, the high SWC and ORC values of fibrous materials (FMD and FSC) compared to powders (PSC and PMD) is due to the fact that FMD and FSC have gum on the surface that makes them more hydrophilic, while the powders have a polygonal shape with voids called lumina that fill and do not allow further water molecules to penetrate. On the other hand, the result of the pasting profile is related to these carbohydrates, as the powders need a certain time to fully hydrate and can delay water absorption and swelling. All results show that pineapple leaf powders represent a new ingredient with nutritional benefits, such as increasing protein content, carbohydrate content in the daily diet and essential mineral content in the formulation of new food products. The powders could be used in bakery products, pasta and dough (to make tortillas) due to the higher WAI and SWE, as these properties affect the extension of shelf life and textural properties, as water is retained and also the oil retention capacity shows that these powders could be involved in the retention of fats in the digestive system. This shows that the powders have a high potential as food ingredients. Future work could focus on food fortification and evaluating the digestive properties of these powders.

Author contributions

Jennifer P. Silva-Cardenas: formal analysis, methodology, writing – review & editing, writing – original draft. Ma. Estela Vazquez-Barrios: conceptualization, methodology, software, writing – review & editing. Luis F. Zubieta-Otero: supervision, resources, writing – review & editing. Mario E. Rodriguez-Garcia: supervision, resources, writing – review & editing.

Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Supplementary information is available. See DOI: https://doi.org/10.1039/d5fb00263j.

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