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Physicochemical, Structural and Rheological Properties of Hawthorn Yam (*Dioscorea rotundata*) Flour

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Abstract

Hawthorn yam has been widely used for the development of food products. The objective of the present work was to investigate the physicochemical, structural, and rheological properties of hawthorn yam flour (CHYF). The identification of chemical composition by proximal and Fourier Transform Infrared Spectroscopy (FTIR) analysis; the morphological by Scanning Electron Microscope (SEM) and rheological properties by steady shear test, stress test, frequency sweep, temperature sweep, and pasting properties analysis were done. CHYF presents a high carbohydrate content (80.47 ± 1.14%), followed by the protein content (8.76 ± 0.58%), ash (3.56 ± 0.52%), and the lowest fat content (0.39 ± 0.06%), and different functional groups such as C=O, COO, N–H, and O-H. Flour particles present a particle size between 27 and 43μm, with spherical-oval morphology with a smooth surface. Flours present a non-Newtonian fluid shear-thinning adjusted to the Power Law model ($R^2 > 0.99$), and a characteristic of a typical strong gel material with a storage modulus (G') higher than loss modulus (G'') and paste temperature of 81.6 ° C with a Peak Viscosity (PV) of 750 cP. Then, it is a potential source of natural ingredients with technological properties for the food industry and the food sovereignty of many countries in the world.

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Introduction

Yams are root crops from Africa and Asia that belong to the Dioscorea family, considered a traditional indigenous crop.[1] Yams contains around 600 species with importance economically, such as *Dioscorea oppositifolia, Thunb, Dioscorea rotundata Poir, Dioscorea dumetorum, Dioscorea esculenta, Dioscorea bulbifera, Dioscorea, cayenensis and Lamk Dioscorea alata*, but only 12 species are edible.[2]

The tubers of yam present diverse compositions

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that contribute to its function, which consists mainly of starch, sugars, protein, fibers, polysaccharides, polyphenols, flavonoids, and other ingredients.[3] Moreover, fresh yam has a high moisture content with values between 50 and 80%, making it susceptible to deterioration and difficult to preserve. Yams are one of the main stable foods in tropical country,[4] and have considerable nutritional properties such as antioxidants, antidiarrheal, immune regulation, and anti-inflammatory abilities,[5] becoming popular use in the diet due to their healthy function. Traditionally, yams are usually consumed in different forms resulting from any of the processes of fermentation, drying, boiling, roasting, milling, steaming, frying, and pounding.[6] Therefore, the main product obtained from yam is the flour obtained by a dry and milled process assembling a food product for consumption that can be stored for several weeks; nevertheless, yam flour, in its native form, has limited use in the food production industry.

The process of fresh tuber transformation consists of the development of flour by peeling, slicing, blanching, and sun drying,[7] therefore there is an urgent demand to modify the yam flour to improve its high perishability. The flours are used in food systems as ingredients or processing aids and can be consumed as a food ingredient in various forms, i.e. to develop instant pounded yam. The knowledge of physical and rheological properties is needed since these are frequently incorporated in the preparation of a wide range of tropical foods and for the design, development, sensory evaluation, and evaluation of the shelf-life stability estimation of the food product; however, rheological studies of Colombian hawthorn yam flour - CHYF are limited, so this investigation can increase its potential applications in the food industry. Therefore, the objective was to investigate the physicochemical, structural, and rheological properties of Colombian hawthorn yam flour (CHYF).

Materials and Methods

Materials

Colombian Hawthorn yam tubers (2 – 4 kg) was cultivated in the Department of Bolivar - Municipality, Carmen de Bolivar, Colombia, as shown in Figure 1. Acetic acid, sodium hydroxide, and phenolphthalein were obtained from Sigma–Aldrich (St. Louis, MO, USA). Hexane and ethanol were purchased from Panreac (Barcelona, Spain).

Fig. 1. Colombian Hawthorn yam (*D. rotundata*). Source: Authors

Formulation of Yam Flour

Colombian Hawthorn yam tubers were cleaned to remove solid particles; after that were washed, peeled off, and sliced manually to a thickness of 10 mm with a manual stainless steel knife. Thin slices were air-dried in a crossflow at 50 °C for 24 h and pulverized using an IKA MF 10.2 (Germany). The obtained flour was packaged separately in transparent polypropylene bags, sealed, and stored at 5 °C.

Determination of Proximal Composition

Moisture, ash, protein, total fat, and carbohydrates of CHYF were obtained following the methods described in the Official Methods of Analysis – AOAC.[8]

Fourier Transform-Infrared Spectroscopy (FTIR – ATR) Analysis

The characterization of flours was carried out employing Fourier transform infrared spectroscopy, Spectrum 400 model apparatus (Perkin Elmer). Operate in ATR mode to obtain the Fourier transform infrared (FTIR). A small sample was placed in the ATR, proved, and measured as they were. Spectra were obtained for a wavelength range of 500 – 4000 cm^{-1} at a resolution of 4 cm^{-1}, in transmission mode.

Morphology Analysis

The morphology of CHYF was studied by scanning electron microscopy (SEM) with an energy-dispersive X-ray spectrometer (SEM-FOT) EM-30AXn, (Coxem, Daejeron, Korea).

Rheological Properties

Rheological characterization was done in CHYF at 10 %wt. in water employing a Rheometer Haake Mars 60 (Thermo Scientific, Germany) using a Peltier system and equipped with a cone plate geometry (1°; 35 mm diameter and 0.053 mm GAP). To have the same recent past thermal and mechanical history, the temperature was fixed, and each sample was equilibrated at 600 seconds before the rheological test.

- Steady shear test: Viscous flow curves were performed from 10^{-3} at 10^{3} s^{-1} at 25 ºC.
- Stress test: A stress sweep from 10^{-3} to 10^{3} Pa at 1 Hz, was carried out to determine the linear viscoelastic regime (LVR).
- Frequency sweep: The frequency sweep test was performed from 10^{-2} to 10^{2} rad·s^{1} at 25 ° C in the linear viscoelastic range.
- Temperature sweep: Thermo-viscoelasticity properties were investigated at a ramp temperature of 20 to 80 °C, at a heating rate of 5 °C/min, and a constant frequency in the linear viscoelastic range.
- Pasting properties: The sample was heated from 25 to 95 °C with a holding time of 2 min followed by cooling to 25 °C. The heating and cooling rate was at a constant rate of 5 °C/min.

Table 1: Proximal composition of CHYF

***Dioscorea* sp flours**	**Moisture %**	**Ash %**	**CHO %**	**Fat %**	**Protein %**	**References**
Colombian Hawthorn yam flour - CHYF	4.82 ± 1.13^{b}	3.56 ± 0.52^{b}	80.47 ± 1.14^{a}	0.39 ± 0.06^{c}	8.76 ± 0.58^{c}	**
White yam flour	7.42	2.25	73.41	0.19	6.96	Yalindua *et al*., [16]
Purple yam flour	10.6	2.35	79.4	0.28	6.57	Yalindua *et al*., [16]
Bitter yam (*Dioscorea dumetorum*) flour, white variety	8.18	5.39	78.15	0.64	5.67	Oyeyinka *et al*., [9]
Bitter yam (*Dioscorea dumetorum*) flour yellow variety	7.94	5.07	77.74	0.74	6.18	Oyeyinka *et al*., [9]
Chinese Yam (*Dioscorea opposita* Thunb.) Flour. Tai Nung No.2	5.39	4.3	83.02	0.3	11.1	Hsu *et al*., [17]
Chinese Yam (*Dioscorea opposita* Thunb.) Flour, Ta Shan	4.73	4.92	82.1	0.3	10.2	Hsu *et al*., [17]
Chinese Yam (*Dioscorea opposita* Thunb.) Flour, Ming-Chien	4.32	4.68	82.2	0.29	11.3	Hsu *et al*., [17]
Chinese yam flours from expansion stage	n.d.	1.77	n.d.	1.52	10.88	Zou *et al*., [18]
Chinese yam flours from dormant stage	n.d.	1.13	n.d.	3.23	9.12	Zou *et al*.,[18]

**Data determined by authors. Mean ± standard deviation. Different letters in the same file express statistically significant differences ($p < 0.05$)

CHO: Carbohydrate

n.d.: non-determinate, this may be attributed to the decreased water loss during the drying process

Statistical Analysis

Significative treatments were determined using one-way analysis of variance ANOVA with Honestly Significant Difference (Tukey's HSD) grouping at a 95% confidence level using Statgraphics Centurion XVI. All analyses were done in triplicate.

Results and Discussion

Proximate Composition of Chyf

The proximate composition of CHYF is reported in Table 1. The flour has a high percentage of carbohydrates (80.47 ± 1.14%), followed by the protein content (8.76 ± 0.58%), ash (3.56 ± 0.52%), and the lowest fat content (0.39 ± 0.06%) indicating that the physicochemical and material properties of the flours vary considerably among the four species.[9] The moisture content of CHYF was lower than 7%, improving the storage conditions and preventing spoilage [10,11] and longer shelf stability; the high-moisture products (>12%) usually have shorter shelf stability compared to lower moisture products (<12%).[12] Similar results were obtained by Setyawan *et al.*[13] for flour of *Dioscorea esculenta* and *Dioscorea bulbifera* with values of 5.62 and 6.72% dried by oven, by Ilesanmi *et al.*,[14] for white yam (5.50%), and consistent with the safety requirement of the Codex Alimentarius Commission for wheat flour.[15]

The proximal compositions of CHYF reveal that it could be used in food formulations as a source of carbohydrates; thus, it can be used as an alternative staple food to provide daily energy needs. Then, yams have been considered rich in resistant starch content and gluten-free, being a promising food source for people to reduce the risk of obesity, diabetes, wheat allergy, and the incidence of celiac disease.[19] In addition, the consumption of diets containing glucose and fructose is higher than the consumption of other diets.[20]

The protein content was moderate, higher than white, purple,[16] and bitter yam [9] Chinese yam ,[17] which was an alternative to improve the techno-functional properties. Then, the yams present relatively low values improving the protein digestibility.[20] Such differences between yam species may be related to their genetic origins and the geographic sources from which they were grown. Moreover, accessing these nutrients is an advantage for food sovereignty; considering the dietary seasonality is foundational to native foodways and the historical trade relationships for food products development associate the right of peoples to healthy and culturally appropriate food produced with Colombian Hawthorn yam through ecologically sound and sustainable methods.

To evaluate the functional groups of CHYF, the analysis by Fourier Transform Infrared Spectroscopy (FTIR) was done, providing unique insights into protein conformation change.[21] Then, the FTIR spectra of CHYF are shown in Figure 2. The peaks 1335–1320 are a CH deformation from ring vibration associated with cellulose and polysaccharides, the peaks between 1630–1600 are COO- asymmetric stretching, peak COO− asymmetric stretching are CH_2 bending mode, 1650–1580 are N–H bending vibration of primary amines, peaks between 1700–1600 corresponds to amide I absorption (predominantly the C=O stretching), between 1741 to 1740 correspond to C=O stretching of alkyl ester, C=O stretching of triglycerides [22] and at 3600 to 3200 cm^{-1} are smaller in the CHYF spectra, corresponding to O–H stretching attributed to moisture.[23] Then, the low infrared spectroscopy suggests relatively open-structured and highly hydrated macromolecules.

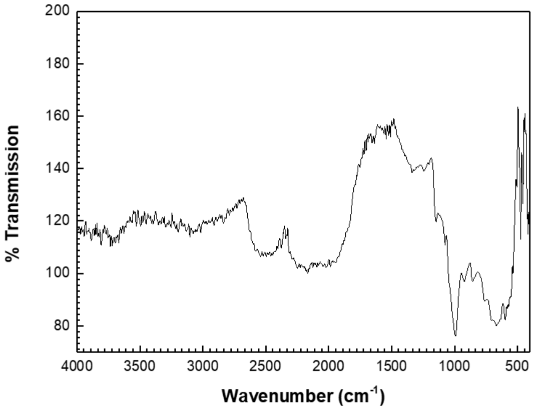

Fig. 2: FTIR spectra of Colombian Hawthorn yam flour

Morphology

Figure 2 shows images at different scales (200, 500, 1000, and 2000X) of CHYF. The micrographs displayed flour particles have spherical/oval smooth surfaces and some particles present flat faces defined with an oval morphology (Figure 3). Larger clusters (like film on the particles) are visible as cellular material (Figures 3a, 3b, and 3c), associated with the starch and protein content in flour. Möller *et al.*[24] reported similar results for the grinding of yellow peas and recognized the small round particles adhering to the surface of the starch granules as protein bodies, and Zhu *et al.*,[25] associated this cluster with the starch and fiber content in mung bean flour.

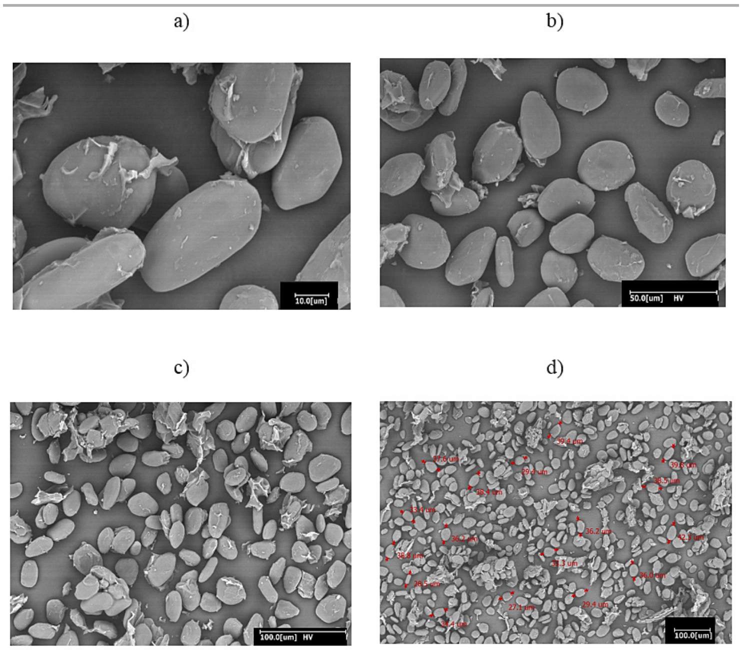
a)
b)
c)
d)

Fig. 3: Morphology of HYF. A) 2000x, b) 1000x, c)500x and d) 200x

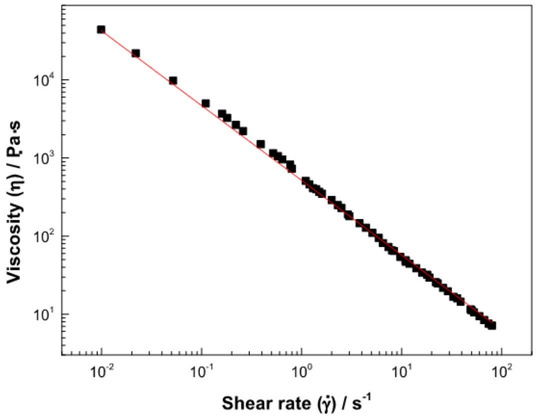

Fig. 4: Viscous flow behavior of the CHYF adjusted to the Power Law model

Furthermore, the particle size of CHYF was analyzed (Figure 3d); the deviation is in the range of 27 to 43μm (<10%). Obtained results were lower than those reported for freeze-dried potato flour (56.51 to 307.53 μm),[26] roasted Highland Barley Flour (< 150 μm),[27] and amaranth flour (380 μm).[28] The structure and morphological characteristics of the flour granules are then affected by the origin, agronomy, and processing conditions of yams prominently due to the distinct characteristics and potential applications.

Rheological Properties

The rheological properties of CHYF were evaluated in a steady and oscillatory state. Figure 4 presents the viscous flow behavior of flour, which shows a decrease in viscosity with the increase of shear rate, demonstrating a possible drop, the typical behavior of non-Newtonian fluid-type shear thinning,[29] associated with the structural collapse of the molecules as a result of the hydrodynamic effect of the forces generated. Then, the Power-law model (Equation 1) allows one to describe many fluids containing soluble solids with high molecular weight:

$$\eta=k\cdot\gamma^{n-1} \qquad ...(1)$$

where k is the consistency index and n is the flow index.

Then, the flow curves of CHYF could be adequately described by the Power law model due to the high determination coefficient ($R^2 > 0.99$), presenting a consistency index of 518.52 ± 31.79 and a flow index of 0.04 ± 0.01. Then the flow behavior index of the suspensions was lower than 1 at all applied pressures, and this corrected for shear-thinning flow behavior.

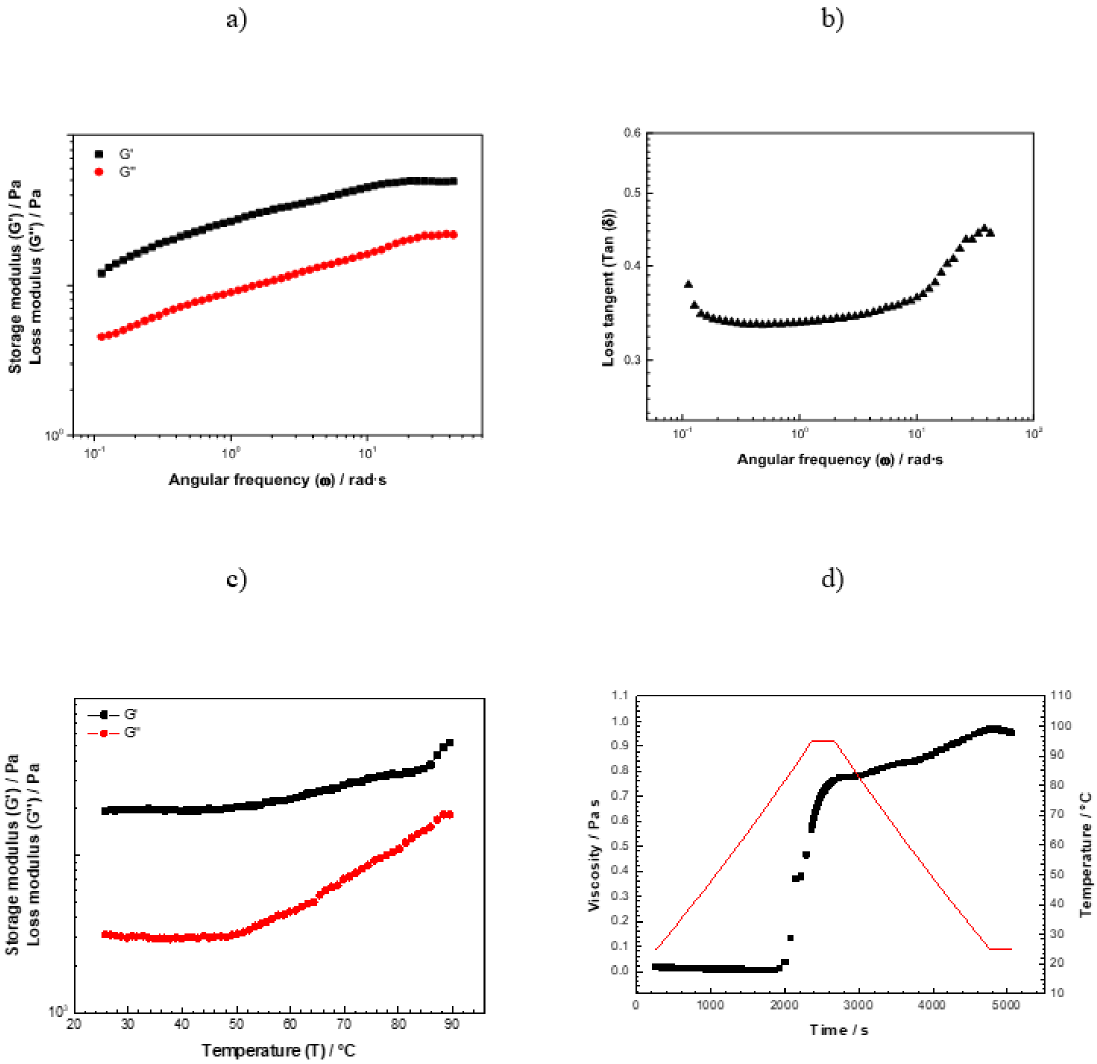

Fig5. a) Storage and loss modulus and b) Loss tangent in function of angular C) Temperature sweep of the CHYF within the linear viscoelastic range, and d) Pasting Properties of CHYF

The viscoelastic behavior of CHYF was analyzed in the linear viscoelasticity region (1 Pa). The evaluation of storage modulus (G') was higher than loss modulus (G'') and Loss Tangent (Tan δ) was done (Figure 5). G^' were higher than the loss modulus G'' with no signs of crossover in the frequency range studied exhibiting a solid-liquid behavior.

Figure 5b presents the behavior of the loss tangent (Tan δ) concerning frequency. CHYF present Tan δ lower than 0.5 indicates the relative contribution of the elastic and viscous components of flours [30]; these values are comparable with the data reported by Kong *et al.*, [31] for starch and flour of rice and Lu *et al.*,[32] for composite starch gel.

The effect of temperature on G' and G'' CHYF are shown in Figure 5c exposing the phase transitions and elasticity and allowing the selection of appropriate temperature ranges for the employee of flour for the development of food products. The flour presents a predominant solid-like behavior where G' was higher than G'' in the range of temperature studied. The viscoelastic behavior of neither did change upon heating, nevertheless, increased from 50 ºC, preserving their solid-like properties. A gel point was not observed because high temperatures reduce the intermolecular hydrogen bonding interactions, removing energized water molecules surrounding the CHYF chains.

Table 2: Pasting parameter of CHYF.

Dioscorea sp flours	PV cP	TV cP	BV cP	SB cP	FV cP	PT ºC	TP Min	References
Hawthorn yam Flour - CHYF	750^{f}	620^{e}	130^{c}	330^{d}	950^{g}	81.6^{b}	30.93^{a}	**
Bitter White yam	1781.04	1355.56	384.5	902.4	2263.51	87.36	5.09	Oyeyinka *et al*., [9]
Bitter Yellow yam	1322.02	1058.23	264.4	596.5	1624.1	87.3	5.08	Oyeyinka *et al*., [9]
Chinese yam flours	3193	1560	1633	401	1961	88,10	3.93	Zou *et al*., [18]
Chinese yam flours	5135	2989	2146	1460	4449	94.49	4.4	Zou *et al*l.,[18]

PV: Peak viscosity; TV: Trough viscosity; BV: Breakdown viscosity; SV: Setback viscosity; FV: Final viscosity; PT: Pasting temperature; TP: Time peak

**Data determined by authors. CV < 0.05. Different letters in the same file express statistically significant differences (p < 0.05)

Pasting Properties

The pasting properties are important to determine the use of ingredients as a thickener and binder in the food industry and could affect the quality and end-use of CHYF. The viscosity of the gel formed is a major aspect of the decision on the use of flour in various applications. The pasting properties of CHYF at 10%w/w are shown in Figure 5d and Table 2, including peak viscosity (PV), trough viscosity (TV), breakdown viscosity (BV), setback viscosity (SB), final viscosity (FV), pasting temperature (PT) and time peak (TP). PT measures the temperature when the viscosity starts to rise and at the beginning of gelatinization; the PV indicates the maximum swelling of the flour granule before disintegration; the BV indicates the difference between the maximum and minimum viscosities at constant temperature (95 ºC) reflecting the stability of flour; the FV reflects the cold paste viscosities and the SB is indicative of the retrogradation tendency.[33]

Colombian Hawthorn yam flours showed a high PT value of 81.6 °C, associated with the presence of components, i.e., oligosaccharides, proteins, and cellulose. Other flours obtained similar results, such as yellow pea (79.3 ºC) [34] beans (80.7 – 84.1 ºC) [35] and kidney bean (89.4 – 94.9 ºC) ,[36] then small flour granules were found to exhibit greater resistance to rupture and loss of molecular order.[37] The peak temperature in the pasting temperature records the highest temperature corresponding to the peak viscosity [38]; in the case of hawthorn yam flours, the PV value is 0.75 Pa·s. BV was 0.13 Pa·s, measuring the difference between PV and the intermediate hot paste recorded during the holding stage; and it is related to gel stability, so lower values suggest that the four is more stable during cooking.[39] SB shows how the viscosity of the flour suspension paste (0.62 Pa·s,) recovered during the cooling period and is calculated by the difference between FV and TV. The SB value is commonly used to reflect the retrogradation; hawthorn yam flours present an SV of 0.33 Pa·s. associated with the development of strong or weak gels in the heating-cooling process; the highest SB resulted in the development of strong rigid gels. An increase in the FV of the flour indicates its resistivity to shearing, resulting in a rigid gel, which might be attributed to the presence of proteins and soluble sugars; then the final viscosity of CHYF was 0.95 Pa·s.

Pasting properties of CHYF present similar values to bitter white and yellow yam [9] and Chinese yam flour.[18] Then, the pasting parameters of the flours were attributed to the chemical composition and particle size that finely tuned the gel structure after the heating and cooling process.

Conclusions

Hawthorn yam flour presents a high percentage of carbohydrates and moderate by protein content, in comparison with different varieties of *Disoscorea sp*.; moreover, the chemical composition is associated with the content of polysaccharides and cellulose. The SEM images displayed flour particles with a range from 27 to 43µm, with spherical/oval morphology and a smooth surface with larger clusters as a cellular material associated with the starch and protein content in flour. Furthermore, flour at 10% wt in water presented a decrease of viscosity with the shear rate, a typical behavior of non-Newtonian flow behavior type shear thinning, with a high coefficient of correlation (R^2 > 0.99) to the Power Law model. Their viscoelastic properties evidence the structure of strong gel (G' > G'') when the pasting properties show the beginning of gelatinization at 81.6 ºC. In conclusion, Colombian Hawthorn yam flour presents considerable composition and appropriate rheological properties suitable for applications in the food industry as a thickening or gelling agent in aqueous solutions. As well as an important nutritional source for the food sovereignty of many countries where this tuber is grown.

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Conflict of Interest

The author(s) declares no conflict of interest.

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