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Dual microwave-ultrasound treatments outperform the single modifications of tef flour. In-depth analysis of physical, hydration, pasting and rheological properties

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ABSTRACT

Physical modification of native whole-grain gluten-free flours is an effective strategy to enhance their functionality and industrial applicability. The present study investigated single and dual physical modification of tef flour using microwave (MW) treatment at two moisture contents (MC) (25% and 30%), and ultrasound (US) treatment. The effects of these modifications were assessed through changes in particle morphology, hydration and pasting properties of the flours, and rheological properties of gels and doughs made with them. Both single and dual MW and US treatments significantly altered particle size distribution, with MW inducing agglomeration [D_{50} = 136 µm (MW25) and 146 µm (MW30)] and US promoting particle fragmentation [D_{50} = 100 µm (US)], resulting in improved water interactions and increased gel stability. The gels obtained from the dually modified flours exhibited improved solid-like behavior [$\tan(\delta)_1$ reduction of 8.0% (MW25-US), 13.3% (US-MW25), and 12.6% (US-MW30)] and greater resistance to deformation (λ_c increase > 200%). Dough rheology revealed that replacing 30% of corn starch with treated tef flour increased the maximum shear stress [τ_{max} up to 14.4 Pa (MW30-US)], reduced $\tan(\delta)_1$ (>20% in all dual treatments), and improved recovery capacity [10.0% (US-MW25), 15.1% (US-MW30), 19.9% (MW25-US) and 30.4% (MW30-US)], denoting an enhanced elastic behavior and higher rupture resistance in the dually treated samples compared to the control dough. Principal component analysis, explained 83.15% of total variance, confirmed that MC strongly influenced the extent of modification caused by MW treatment and demonstrated a synergistic effect of dual treatments, particularly when US was applied after MW.

1. Introduction

Cereal grains are staple foods in different cultures and represent the basis of human energy intake. It is estimated that cereal grain consumption represents more than 50% of the world daily caloric intake due to direct consumption and its incorporation as an ingredient in different food products (Awika, 2011; Landberg et al., 2019). However, most cereals are consumed as refined grains, where the nutritional fractions (i.e. bran and germ) of the grains have been removed to meet sensory expectations. An excessive intake of refined grains has been linked to adverse health outcomes, such as obesity, colon cancer, hypertension and type 2 diabetes (Landberg et al., 2019; Williams, 2012; You & Henneberg, 2016). Whole grain-based foods present a better nutritional profile due to their fiber content, vitamins, minerals, phytoestrogens, and antioxidants (Williams, 2012). In the gluten-free (GF) market, regular consumption of whole grains tends to be harder given that GF products are mostly made from starches and refined rice flour, making it a priority to promote the incorporation of more nutritious GF whole grain sources.

Tef [*Eragrostis tef* (Zucc.) Trotter] is a highly nutritious GF ancient grain native to Ethiopia. Due to their small size, tef grains are always processed into whole grain flour. Tef is rich in dietary fiber, presents an

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equilibrated balance of essential amino acids, a high content of several minerals (Ca, Fe, Mg, P, K, Na and Zn), and a wide variety of bioactive compounds (phytosterols, vitamins and phenolic compounds) (Villanueva et al., 2021; Zhu, 2018). However, the formulation and production of GF food products with organoleptic and sensory properties comparable to the gluten-containing counterparts represent a more complex challenge because of the structuring role of gluten in these products. A strategy adopted by the food industry to improve the functionality and applicability of GF sources is to modify their native characteristics using different modification approaches (genetic, enzymatic, chemical, and physical modifications) (Vela, Villanueva, Ozturk, et al., 2023). Among these different techniques, physical modifications are better perceived by consumers because they do not involve chemical or biological agents in the process (Acevedo et al., 2022). The use of microwave (MW)-assisted hydrothermal treatments and ultrasound (US) treatments has shown to generate structural and techno-functional modifications of tef flours (Calix-Rivera et al., 2023; Náthia-Neves et al., 2023; Vela, Villanueva, Li, et al., 2023; Vela, Villanueva, Ozturk, et al., 2023).

MW and US differ significantly in terms of the impact of the applied energy on the particles and the severity of the damage caused to starch and proteins. In MW treatments, heat is generated inside the treated particles due to friction between polar and ionic molecules induced by the movement caused by the microwaves, allowing faster heating and shorter processing times than those of conventional heating techniques (Kurek et al., 2024; Vicente et al., 2023). In US treatments, the modification caused to the starches and flours is linked to the cavitation phenomenon, in which the rapid generation and collapse of bubbles due to high-pressure gradients and high local velocities of liquid layers caused by the sinusoidal ultrasound waves passing through the aqueous medium create shear forces capable of fracturing particles and even breaking molecular bonds (Jambrak et al., 2010; Li et al., 2018). Previous research studies have shown that MW and US treatments, applied independently, can be used to modulate the techno-functional properties of GF flours, and lead to more consistent and resistant gels and doughs (Solaesa et al., 2021; Vela, Villanueva, & Ronda, 2023). Some recent studies have covered combined MW and US treatments in the modification of starches from chestnut (Wang et al., 2020), corn (Yılmaz & Tugrul, 2023), maize and potato (Zhou et al., 2023), however, whole grain flours, which are more complex systems, that have not been studied by dual MW-US treatments.

In the present study we hypothesized that the structural impact of MW and US treatments can be combined in sequential treatments to achieve amplified modifications of tef flour, that lead to the formation of gels and dough with a higher consistency. The modification of starch by dual MW and US treatments has potential industrial applications in the production of starch-based products with specific properties, such as high water absorption capacity and low glycemic index (Wang et al., 2020; Zhou et al., 2023).

2. Materials and methods

2.1. Materials

White tef [*Eragrostis tef* (Zucc.) Trotter] grains grown in Spain were provided by Salutef (Palencia, Spain). The grains had an EU organic bio certification (ES-ECO-016-CL, batch L0HTBSG04 DIC-19), with the following composition per 100 g: 72.4 g carbohydrates (8.0 g total dietary fiber), 13.3 g protein, and 2.1 g total fat. Tef grains were milled using an LM 3100 hammer mill (Perten Instruments, Hägersten, Sweden) with a 500 µm screen size. Untreated whole grain tef flour was used as the control. Corn starch (C*Gel™ 03401), used in the formulation of the bread dough, was supplied by Brenntag Química S.A.U. (Dos Hermanas, Seville, Spain).

2.2. Microwave (MW) treatment

The moisture content (MC) of the native whole grain tef flour was determined to be 9.87% by weight difference using an OHAUS MB120 moisture analyzer (OHAUS Europe GmbH, Nänikon, Switzerland). The flour's MC was preconditioned at 25.0 ± 0.5 g/100 g and 30.0 ± 0.5 g/100 g (wet basis) before MW treatment by spraying the necessary amount of distilled water in the flour while homogenizing using a TEDDY V5A food mixer (Varimixer, Brøndby, Denmark). After homogenization, the flours were placed in hermetic polypropylene containers and kept under refrigeration (4 ± 2 °C) for 18 h to reach moisture equilibrium in the sample. Finally, the moisture content was checked, and the samples were stored in hermetically sealed polyamide/polypropylene bags (~125 g per bag). The bags were stored at - 40 °C until use.

The tef flour (120 g) was placed in hermetically sealed borosilicate glass containers, which were horizontally coupled to a rotation device (60–70 rpm) inside the MW equipment. The MW treatment was performed for 30 min using a Sharp MW R-342 equipment (Sakai, Japan) with a power of 900 W (equivalent to 7.5 W/g flour), and frequency of 2450 MHz. An external sensor was used to track the temperature of the container wall during treatments. The treatment started at 25 °C, and the temperature increased at 13 °C/min until reaching 91 °C on the wall of the container, indicating that the temperature in the middle of the container was 100 ± 5 °C [determined in initial tests using Testoterm® temperature strips (TESTO, Barcelona, Spain) placed inside the container in constant contact with the sample]. After finishing the treatment, the containers continued rotating inside the MW equipment to cool the sample down to 30 °C, followed by drying in a Memmert ICP260 incubator (Büchenbach, Germany) at 35 °C until reaching 13% MC. The samples were sieved to $<$ 500 µm using a CISA RP200N digital electromagnetic sieve shaker (Barcelona, Spain). The MW-treated samples were stored at 4 °C in hermetically sealed polypropylene containers until further use. The samples were labelled as MW25 and MW30, corresponding to the samples preconditioned to 25% MC and 30% MC, respectively, before the MW treatment.

2.3. Ultrasound (US) treatment

The tef flour (120 g) was suspended in distilled water (300 g) (concentration of 30% w/w dry matter), and the US treatment was performed using a Hielscher UP400St ultrasonicator (Hielscher Ultrasonics GmbH, Teltow, Germany) at a constant frequency of 24 kHz and a maximum output power of 180 W (equivalent to 0.45 W/g suspension) using a 22 mm probe (providing a power density of 355 W/m^2), for 15 min with an on/off pulse per second of 80%. The temperature of the dispersion was kept constant at 10 ± 2 °C during treatment using recirculating water from a LAUDA RA12 water bath (Lauda-Königshofen, Germany), and the flour was kept suspended during treatment using a magnetic stirrer to ensure a homogeneous modification effect. After US treatment, the dispersion was frozen and freeze-dried using a Telstar Lyoquest freeze-dryer (Telstar, Terrassa, Spain). The freeze-dried flours were sieved to $<$ 500 µm and stored at room temperature until use. The ultrasonicated sample was labelled as US.

2.4. Dual treatments

Dual treatments were performed by applying one modification treatment (either microwave or ultrasound treatment) after the other, following the procedures described in Sections 2.2 and 2.3. The samples that were initially modified by MW followed by US treatment were labelled as MW25-US and MW30-US, and the samples that were modified by US prior to MW were identified as US-MW25 and US-MW30.

2.5. Particle size distribution

The granulometry of the flour samples was studied using a Mastersizer 2000 instrument (Malvern Instruments Ltd, Malvern, UK) coupled with a dry dispersion unit. The reported results were the median diameter (D_{50}) and dispersion profile [(D_{90}-D_{10})/D_{50}], as indicated by Vela, Villanueva, Ozturk, et al. (2023). The samples were measured in triplicate.

2.6. Scanning electron microscopy (SEM)

The physical appearance of the flours was analyzed using a Quantas 200FEG scanning electron microscope (FEI, Hillsboro, OR, USA) equipped with a large field detector, at an acceleration voltage of 7 keV under low vacuum mode. The flour samples were placed on aluminum stubs using conductive carbon tape and coated with a 5 nm layer of gold using an SCD-05 Leica microsystem (Wetzlar, Germany). The images were recorded at magnifications of 500× and 3000×. Illustrative micrographs were selected to depict the morphologies of the tef flours.

2.7. Hydration properties of the flours

The water absorption capacity (WAC), water absorption index (WAI), water solubility index (WSI), and swelling power (SP) of the control and modified flours were determined following the method described by Calix-Rivera et al. (2023). The hydration properties were determined in triplicate.

2.8. Rapid Visco Analyzer (RVA)

The pasting properties of the studied flours were determined using a Rapid Visco Analyzer 4500 (PerkinElmer Inc., Waltham, MA, USA) following the AACC International Method 76-21.02 Standard 1 (AACC International Approved Methods, 2017). The parameters obtained after the tests were pasting temperature (PT), peak viscosity (PV), trough viscosity (TV), breakdown viscosity (BV), final viscosity (FV), and setback viscosity (SV), using the TCW3 software (PerkinElmer Inc., Waltham, MA, USA). All samples were analyzed in triplicate.

2.9. Dough formulation and preparation

The dough formulation consisted of the following recipe, based on 100 g of the mixture of tef flour:corn starch (30:70) (MC of 13 g/100 g): 90 g water, 5 g sugar, 1.5 g salt, 2 g hydroxypropyl methylcellulose (HPMC), and 6 g sunflower oil. A mixture of untreated whole tef flour and corn starch (30:70) was used to prepare the control dough. The doughs containing the modified tef flours were labelled as the flour used in the formulation (MW25, MW30, US, MW25-US, MW30-US, US-MW25, and US-MW30). The doughs were prepared using Auto Bakery equipment (Funaj, China). Initially, the dry ingredients were mixed with water for 2 min, starting with a slow speed for the first minute and then increasing to a fast speed for the second minute. Afterward, oil was incorporated, and the dough mixture was further blended for 8 min at high speed. The dough was placed in closed containers and allowed to rest for 10 min before performing rheological measurements.

2.10. Dynamic-oscillatory measurements of gels and doughs

The rheological properties of the gels obtained from RVA analysis (Section 2.8) and the doughs (Section 2.9) were analyzed by dynamic oscillatory measurements using a Kinexus Pro+ (Malvern Instruments Ltd., Malvern, United Kingdom) equipped with a 40 mm parallel plate working at a 1 mm gap and constant temperature (25 °C) controlled with a Peltier KNX2002 C25P unit. The samples were left to rest for 5 min between the plates to allow relaxation before the tests started. Strain sweeps were carried out from 0.1 to 1000% strain for the gels, and from 0.01 to 500% strain for the doughs, at a constant frequency of 1 Hz to establish the linear viscoelastic region (LVR) and the maximum stress tolerated by the sample before the collapse of its structure (τ_{max}) (Vela, Villanueva, & Ronda, 2023). Frequency sweeps were performed from 10 to 1 Hz at 1% gels) and 0.05% (doughs) strain, within the LVR, and the data were fitted to the Power-law model as described by Ronda et al. (2017). Two independent doughs were prepared, and rheological measurements were performed in duplicate for each dough.

$$G'(\omega) = G'_1 \cdot \omega^a \quad \text{Eq. 1}$$

$$G''(\omega) = G''_1 \cdot \omega^b \quad \text{Eq. 2}$$

$$\tan(\delta)(\omega) = \frac{G''(\omega)}{G'(\omega)} = \left(\frac{G''}{G'}\right)_1 \cdot \omega^{b-a} = \tan(\delta)_1 \cdot \omega^c \quad \text{Eq. 3}$$

Where G'_1, G''_1, and $\tan(\delta)_1$ represent the elastic modulus (G'), viscous modulus (G'') and loss tangent [tan(δ)], respectively, at a frequency of 1 Hz. The *a*, *b*, and *c* exponents quantify the dependence degree of G', G'', and tan(δ) to the oscillation frequency (ω) (Ronda et al., 2017).

2.11. Creep-recovery measurements of doughs

Creep-recovery measurements were performed on the doughs using a Kinexus Pro+ rheometer (Malvern Instruments Ltd., Malvern, United Kingdom) with the same configuration described in Section 2.10. A constant stress of 50 Pa (outside the LVR) was applied to the samples for 60 s, followed by 180 s of recovery after the stress was removed. The data were fitted to the 4-parameter (Eqs. 4) and 3-parameter (Eq. (5)) Burgers models for the creep phase and recovery phase, respectively (Villanueva et al., 2019).

$$J_c(t) = J_{0c} + J_{1c}\left(1 - \exp\left[\frac{-t}{\lambda_{1c}}\right]\right) + \frac{t}{\mu_0} \quad \text{Eq. 4}$$

$$J_r(t) = J_{max} - J_{0r} - J_{1r}\left(1 - \exp\left[\frac{-t}{\lambda_{1r}}\right]\right) \quad \text{Eq. 5}$$

Where J(t) is the compliance at a time "t", J_0 is the instantaneous compliance, J_1 is the retarded elastic compliance, λ_1 is the retardation time, μ_0 is the steady state viscosity, and J_{max} is the maximum compliance obtained in the creep phase (Villanueva et al., 2019). The subscript "c" refers to the data corresponding to the creep phase, and the subscript "r" refers to those of the recovery phase. The recovery of the doughs was calculated as the ratio J_{steady}/J_{max} (%), where J_{steady} represents the steady-state compliance in the recovery phase (Vela, Villanueva, & Ronda, 2023). Two independent doughs were prepared, and creep-recovery tests were performed in triplicate for each dough.

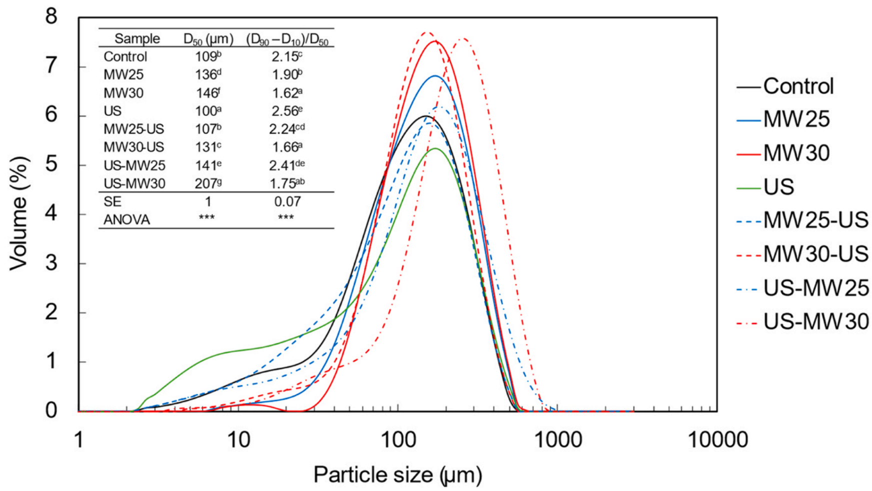
2.12. Statistical analysis

Analysis of variance (ANOVA) by Least Significant Differences (LSD) test at p-value < 0.05 and the fitting of the creep-recovery data to the 4- and 3-parameter Burger model were performed using Statgraphics Centurion XVIII software (Statgraphics Technologies Inc., The Plains, VA, USA). The principal component analysis (PCA) was performed using OriginPro 2024 (Northampton, MA, USA).

3. Results and discussion

3.1. Morphology of the tef flours

The particle size distributions of the studied tef flours are shown in Fig. 1. Granulation and particle uniformity are important factors affecting the processing performance of flours, which tend to be significantly modified by physical treatments (Vela et al., 2021). The results showed that MW treatment led to a significant increase of median



Sample	D_{50} (μm)	$(D_{90} - D_{10})/D_{50}$
Control	109^{b}	2.15^{c}
MW25	136^{d}	1.90^{b}
MW30	146^{f}	1.62^{a}
US	100^{a}	2.56^{e}
MW25-US	107^{b}	2.24^{cd}
MW30-US	131^{c}	1.66^{a}
US-MW25	141^{e}	2.41^{de}
US-MW30	207^{g}	1.75^{ab}
SE	1	0.07
ANOVA	***	***

Fig. 1. Particle size distribution of the control and treated tef flours. D_{50} = Median diameter, $(D_{90} - D_{10})/D_{50}$ = Size dispersion. SE = Pooled standard error from ANOVA. Different superscript letters in the same column represent statistically significant differences between means at $p < 0.05$. Analysis of variance and significance = *** $p < 0.001$, ** $p < 0.01$, * $p < 0.05$.

diameter (D_{50}), potentiated by the higher MC in the sample during treatment, whereas the US treatment resulted in a significant size reduction. The larger sizes obtained after MW treatment are believed to result from particle agglomeration due to protein denaturation during treatment, causing the adhesion of proteins and starch granules into larger agglomerates (Calix-Rivera et al., 2023). The agglomeration of protein bodies in MW-treated tef flours was previously identified by Náthia-Neves et al. (2023), in which an increased reduction of albumins, globulins, and prolamins was obtained with treatments at a higher MC (25% vs. 15%) and longer MW exposure (6 min vs. 1 and 2 min), indicating a greater intensity in the effect of the physical modification. The results suggest that the higher MC during the MW treatment (30% vs. 25% in the present study) allowed a greater extent of protein denaturation, which stimulated the agglomeration of particles. In the case of starch granules, Yang et al. (2017) indicated that MW treatment at 30% MC damaged the outer part of the granules and promoted granule aggregation with each other. In contrast, ultrasonication caused the fragmentation of particles due to cavitation, in which the impact of the collapsing bubbles and the consequent water jets created physical damage to the surface of the flour particles until they were fractured into smaller fragments. It was shown by Vela et al. (2021) that ultrasonication times as low as 2 min can cause a significant size reduction in rice flour. Acoustic cavitation can also have an effect at the molecular level, breaking the chains of biopolymers by disrupting covalent bonds (Jambrak et al., 2010). A significant increase in the size of tef flours after single MW treatments, as well as a reduction due to single US treatments, has been previously reported by Calix-Rivera et al. (2023) and Vela, Villanueva, Ozturk, et al. (2023), respectively. In the case of dual treatments, the results indicated a sequence-dependent effect on the median particle size of the samples. The prior modification by MW caused the agglomeration of flour particles which were then partially disaggregated by the effect of cavitation, hence the smaller D_{50} sizes in MW25-US and MW30-US compared to MW25 and MW30. In contrast, when native tef flour was initially ultrasonicated, the particles were first fragmented, leading to a higher number of small particles and a larger exposed surface. This condition facilitated protein denaturation and partial gelatinization of the starch granule surface during the subsequent microwave treatment, resulting in larger agglomerates than those produced by MW treatment of the native flour (Náthia-Neves et al., 2023; Zavareze et al., 2010). The D_{50} recorded for US-MW30 was > 90% larger than that of the control flour and > 41% larger than that of MW30.

The flour dispersion $[(D_{90} - D_{10})/D_{50}]$ was also significantly affected by the single and dual treatments (see Fig. 1). The single-treated MW flours showed a lower dispersion than the control, with greater reduction with increasing MC, whereas the US-treated sample presented the highest dispersion. The particle agglomeration caused by MW treatment had a greater impact on smaller size particles (< 80 μm), evidenced by a lower dispersion in MW25 and MW30 than the control flour. Smaller size particles have a greater surface area for specific volume than bigger particles, hence being more susceptible to agglomerate during MW treatment (Kaur & Gill, 2019). The peak corresponding to the greatest percentual volume of particles was also shifted to a bigger size in MW30 and MW25 (both to ~170 μm) compared to the control (~150 μm), while their fraction of particles between 400 μm and 1000 μm was not too different among them, resulting in significantly narrower dispersions after single MW treatments. A similar MC-dependent effect was also observed in the dual treatments; higher dispersions were observed when treated at 25% compared to their 30% MC counterpart. The dual treatment of flours at 25% MC resulted in a higher dispersion than the control sample, regardless of the order of treatment application. These results indicate that a higher MC promoted the formation of more uniform particle sizes [lower values of $(D_{90} - D_{10})/D_{50}$] owing to better conditions for agglomeration during MW.

The microstructures of the studied flours at magnifications of 500 × and 3000 × are presented in Fig. 2. The control flour particles consisted of packed structures of starch granules attached to proteins and lipids, with a mostly smooth surface (Fig. 2 A2) (Calix-Rivera et al., 2023). The individual tef starch granules are polygonal in shape, with diameters of 2–6 μm (Bultosa et al., 2002). Protein bodies are about 1–2 μm in size, but seem to form a network around the compound starch granules, resulting in structures of approximately 10–30 μm (Nyakabau et al., 2013). The larger size of tef flour particles due to agglomeration in MW-treated flours can be clearly observed (Fig. 2 B1 and C1, arrows indicate the areas where particles agglomerated), and the closer magnifications show much rougher surfaces and signs of melting in the flour particles (Fig. 2 B2 and C2) compared to the control (Fig. 2 A2). The surface of the MW30 particles appeared to be more damaged than MW25, probably because of the increased effects allowed by the higher MC (as seen in D_{50}). The US-treated sample showed clear signs of particle fragmentation (Fig. 2 D1) and surface disruption, with several starch granules separated from the flour particles due to the action of cavitation (Fig. 2 D2). The characteristic polygonal shape of starch granules is indicated with arrows in Fig. 2 D2. Similar surface damage has been previously reported for US-treated flours (Vela et al., 2021; Vela, Villanueva, Li, et al., 2023). Remarkable differences were observed in the dual-treated samples, in which the effects of both MW and US can

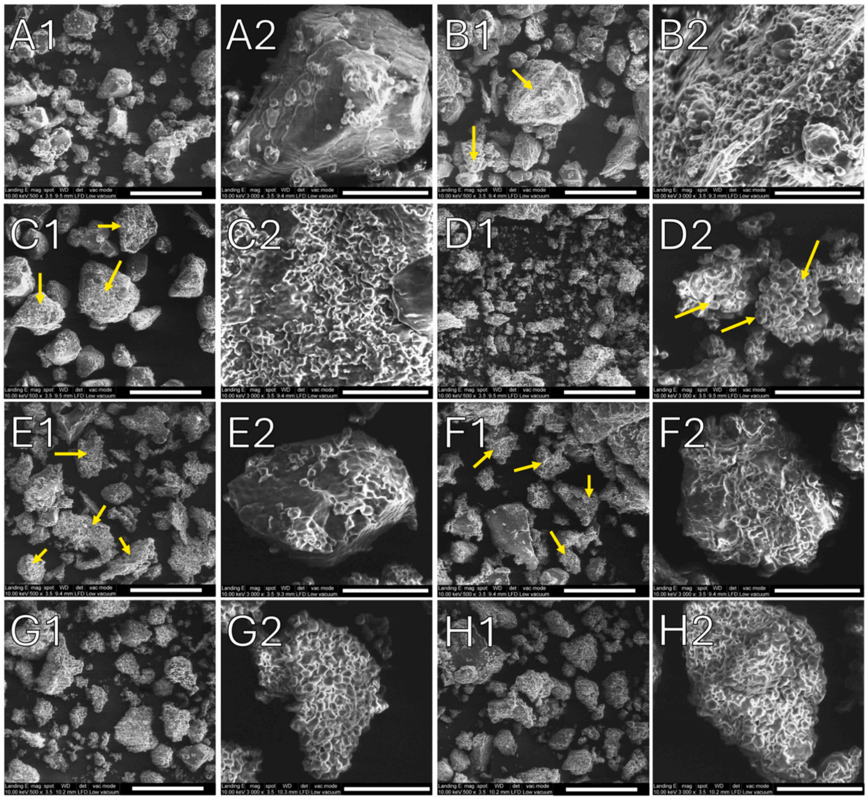


Fig. 2. Scanning electron micrographs of the studied flours. (A) Control, (B) MW25, (C) MW30, (D) US, (E) MW25-US, (F) MW30-US, (G) US-MW25, (H) US-MW30. (1) 500 ×, the white line on the bottom right corner corresponds to 200 μm, (2) 3000 ×, the white line on the bottom right corner corresponds to 40 μm.

be appreciated. In agreement with what was observed in the particle size distribution, the flours that were microwaved first formed agglomerates (as seen in MW25 and MW30). However, the subsequent cavitation caused the fragmentation of the agglomerates; thus, the resulting particles (Fig. 2 E and F) were smaller than the corresponding single-treated MW samples (Fig. 2 B and C). The signs of particle agglomeration are shown in Fig. 2, E1 and F1 with arrows. In the initially ultrasonicated samples, US-MW25 and US-MW30, it is believed that cavitation caused particle fragmentation and detachment of starch granules that were later agglomerated as a result of the MW treatment, resulting in the agglomeration of mostly broken granular structures (Fig. 2 G and H) instead of the packed particles with predominantly melted surfaces observed in MW25, MW30, US-MW25, and US-MW30. The polygonal shape of the starch granules can be clearly seen in surface of the initially ultrasonicated samples (Fig. 2, G2 and H2), compared to those ultrasonicated in the second treatment (Fig. 2, E2 and F2). When comparing the effect of the MC on the treatments, the micrographs showed that the MW treatments performed at 30% MC resulted in a greater extent of surface roughness than their counterparts treated at 25% MC.

3.2. Hydration properties

The hydration properties of the studied samples are presented in Table 1. Hydration properties in flours depend on the interaction of their constituents with water. In starch, these interactions are influenced by the amylose-to-amylopectin ratio, molecular weight, degree of branching, branch length, and conformation (Chan et al., 2010). In proteins, structural modifications and denaturation can expose polar groups,

Table 1
Hydration properties and pasting properties of the studied tef flours.

Sample	Hydration properties (dry basis)				Pasting properties					
	WAC (g/g)	WAI (g/g)	SP (g/g)	WSI (g/100 g)	PT (°C)	PV (cP)	TV (cP)	BV (cP)	FV (cP)	SV (cP)
Control	1.07^{ab}	6.4^{b}	6.8^{a}	5.7^{b}	77.9^{a}	1059^{g}	930^{g}	129^{d}	1789^{f}	859^{e}
MW25	1.36^{c}	6.7^{c}	7.3^{bc}	7.7^{cd}	88.7^{c}	875^{d}	839^{e}	36^{b}	1373^{d}	534^{d}
MW30	1.63^{d}	6.8^{cd}	7.4^{c}	10.3^{f}	94.4^{ef}	492^{a}	480^{b}	13^{a}	668^{a}	188^{a}
US	1.04^{a}	8.9^{f}	9.1^{e}	3.9^{a}	84.8^{b}	980^{f}	814^{d}	167^{e}	1690^{e}	877^{e}
MW25-US	1.16^{b}	6.6^{bc}	7.1^{b}	7.4^{c}	91.3^{d}	845^{c}	816^{d}	29^{b}	1362^{d}	546^{d}
MW30-US	1.37^{c}	6.1^{a}	6.7^{a}	8.5^{d}	95.0^{f}	495^{a}	460^{a}	35^{b}	754^{b}	294^{b}
US-MW25	1.78^{e}	7.3^{e}	7.8^{d}	8.3^{d}	90.4^{d}	947^{e}	885^{f}	63^{c}	1329^{d}	444^{c}
US-MW30	1.71^{de}	7.0^{de}	7.7^{d}	9.4^{e}	93.6^{e}	598^{b}	580^{c}	18^{a}	860^{c}	280^{b}
SE	0.03	0.1	0.3	0.1	0.3	5	4	3	22	21
ANOVA	***	***	***	***	***	***	***	***	***	***

WAC = Water absorption capacity, WAI = Water absorption index, WSI = Water solubility index, SP = Swelling power, PT = Pasting temperature, PV = Peak viscosity, TV = Trough viscosity, BV = Breakdown viscosity, FV = Final viscosity, SV = Setback viscosity. SE = Pooled standard error from ANOVA. Different superscript letters in the same column represent statistically significant differences between means at $p < 0.05$. Analysis of variance and significance = *** $p < 0.001$, ** $p < 0.01$, * $p < 0.05$.

thereby enhancing increasing water-binding affinity (Kumar et al., 2017). Water absorption capacity (WAC) reflects the maximum amount of water that flours can retain after centrifugation at room temperature. The physical modification methods led to a significant increase in WAC under almost all the studied conditions (except for US and MW25-US). In agreement with this finding, Vela et al. (2021) indicated that ultrasound treatments did not lead to significant differences in WAC of rice flours ultrasonicated for longer than 5 min. Previous ^{1}H NMR studies have shown that MW treatments affect mainly α-(1,6) glycosidic linkages, whereas US treatments preferentially fragment α-(1,4) glycosidic linkages, leading to a greater breakage of the compacted crystalline region after MW treatments (Vela, Villanueva, Li, et al., 2023; Yang et al., 2017). The higher WAC values obtained after MW treatment are therefore attributed to the enhanced starch hydrophilicity as consequence of the disruption of hydrogen bonds between amylose and amylopectin, accompanied by partial expansion of the amorphous regions (Náthia-Neves et al., 2023; Perraulta Lavanya et al., 2021). MW treatments involve rapid heating that quickly raises local temperature and intensifies the vibrational motion of water molecules within starch granules, facilitating the destruction of hydrogen bonds between starch macromolecules (Fan et al., 2013). The release of bound water molecules from within the starch granules after MW treatment results in flours with an increased capacity to interact with polar molecules. A higher WAC was observed for MW-treated samples with an initial MC of 30%, consistent with previous studies indicating that higher MC during MW treatment promoted the WAC of tef flours (Calix-Rivera et al., 2023; Náthia-Neves et al., 2023). The highest WAC values (+54% higher than the control) were obtained when MW treatment was applied after US, which could be related to the larger exposed area of the agglomerated fragments (see Fig. 2 G2 and H2), increased starch damage, and protein structure modification, all of which contribute to greater water-binding affinity (Kumar et al., 2017).

The water absorption index (WAI) and swelling power (SP) were mainly increased by US treatment (+39% and +34% higher than those of the control, respectively). Previous research has shown that ultrasonication leads to greater WAI and SP values due to particle fragmentation, which translates into an increased surface area (in both carbohydrates and proteins) that physically allows an improved interaction with water, as well as the breakage of intermolecular bonds of starches that result in less compact granular rearrangements (Amini et al., 2015; Kaur & Gill, 2019; Luo et al., 2008; Vela et al., 2021; Vela, Villanueva, Ozturk, et al., 2023). Single MW treatments generally showed moderate increases in both WAI and SP values. Dual treatments that applied US in the first stage (US-MW25 and US-MW30) showed WAI and SP values 9–15% higher than those of the untreated tef flour. These results align with the greater exposed surface area observed in the micrographs of US, US-MW25, and US-MW30 (Fig. 2) due to acoustic cavitation. The higher SP and WSI observed in most MW-treated flours are attributed to the preferential fragmentation of the crystalline regions in starch during the hydrothermal treatment, since it facilitated granular water intake and the leaching of amylopectin fragments out of the starch granules (Calix-Rivera et al., 2023; Lai, 2001; Villanueva et al., 2019). In the ultrasonicated sample, WSI was reduced by 35% compared to the control, which is believed to result from the prevention of amylose leaching due to the strengthening of the bonds between amylose and amylopectin or between amylopectin molecules (Zavareze & Dias, 2011). In the MW-US treatments, it is believed that the agglomerates formed by the MW treatment had a degraded fraction of amylopectin; therefore, even if the US treatment prevented amylose leaching to a certain degree, there were more flexible crystalline regions that allowed more amylose leaching than the untreated flour. This explains why the WSI values of MW25-US and MW30-US were much higher than that of US, but still lower than their corresponding single MW and US-MW counterparts. The US-MW samples showed closer WSI values to those of the single MW-treated flours, probably due to the final effect of MW treatment on the samples. The results indicate a sequence-dependent response in the hydration properties in dual treatments, likely due to the structural modifications introduced during the first processing stage, with the US-MW combination generating the greatest effects.

3.3. Pasting properties by RVA

The viscometric profiles obtained for the studied samples are presented in Fig. 3, and the pasting parameters are presented in Table 1. The treated flours showed a delay in pasting temperature (PT) and an overall reduction in the viscometric profile compared to the control. Higher PT values indicated that both single and dual treatments caused the reorientation of starch macromolecular chains, preventing the swelling of granules during heating (Acevedo et al., 2022; Marta et al., 2022). The greatest PT increases were observed in the samples involving MW treatment at 30% MC (MW30-US, MW30, and US-MW30, +13–17 °C than the control). The delay of PT is an indication of modified starch granules with enhanced thermal resistance that restricted granule swelling. Zhong et al. (2020) have related a PT delay in RVA to molecular mobility induced by MW treatments. In addition, these samples also showed the greatest reduction of viscometric profiles. It has been reported that the larger particle sizes obtained after MW treatments can contribute to a greater decrease in viscosity development in RVA measurements due to physically lower susceptibility to gelatinization after agglomerate formation (Calix-Rivera et al., 2023). The moisture-dependent effect of MW treatment on the modification of the pasting properties of flours has been reported previously (Calix-Rivera et al., 2023; Vicente et al., 2023). A higher MC during MW treatment facilitates molecular mobility in starch, allowing faster and deeper changes (Vicente et al., 2023). The higher MC enhances the efficiency of MW treatment due to the dominant role of water molecules in dielectric heating. Water increases energy absorption and promotes volumetric heating, resulting in a more homogeneous temperature distribution within the starch–protein matrix (Vicente et al., 2025). This intensified heating increases chain mobility and facilitates the disruption and reorganization of hydrogen bonding networks within starch granules.

Luo et al. (2006) have explained the viscometric profile reduction after MW treatment as consequence of an increase in the inter- and intramolecular hydrogen bonds in starch chains that prevent swelling of the granules. The sample treated by US showed the lowest viscometric profile reduction among the treated samples. A time-dependent trend in the reduction of the pasting parameters was reported by Vela et al. (2021) in the ultrasonication of rice flour, which explains the rather smooth modification obtained in the US sample after a treatment of 15 min. The lower pasting profiles after ultrasonication are consequence of the physical damage caused to starch granules, and the partial starch molecular depolymerization by cavitation (Jambrak et al., 2010; Vela, Villanueva, Li, et al., 2023). In the case of dual treatments, previous studies have reported that both US and MW can disrupt the crystalline structure of starch granules, break macromolecular chains (starch and proteins), and reduce the rigidity and integrity of the starch granules, which could result in a combined effect in the reduction of the pasting properties (Perraulta Lavanya et al., 2021; Vela, Villanueva, Li, et al., 2023; Wang et al., 2020). The reduction of the viscometric profiles was observed in the peak (PV), trough (TV), breakdown (BV), final (FV), and setback (SV) viscosities. MW30 was the sample to report the lowest values in all the pasting properties, except for TV. BV was remarkably reduced by the MW treatment, going from 129 cP in the control sample to 18 cP (US-MW30) and 13 cP (MW30). BV indicates the capacity of a starchy gel to withstand stress at high temperatures (Vela et al., 2021). The decrease in BV in all samples involving MW indicates that the modified tef flours have higher thermal stability due to the effect of the heat-moisture treatment over the starch granules, particularly when applied to flour at 30% MC. SV was also reduced by single MW treatment and dual treatments at 30% MC, with treated samples showing values up to 78% (MW30), 66% (MW30-US), and 67% (US-MW30) lower than the control. These lower SV values are indicative of a lower amylose

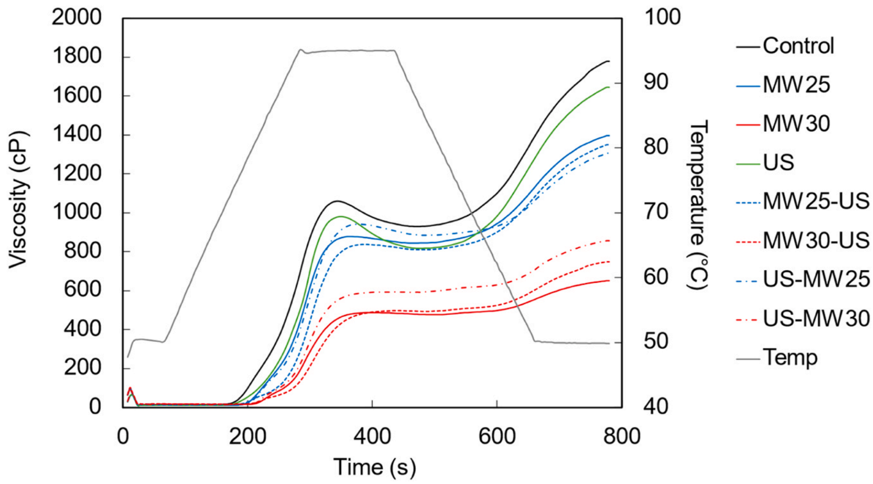


Fig. 3. Viscometric profiles of the control and modified tef flours.

retrogradation tendency, which could be related to alterations of the starch structure, such as the generation of new amylose-amylose and amylose-amylopectin interactions within the MW-treated samples (Vicente et al., 2023).

3.4. Dynamic-oscillatory measurements of gels

The rheological properties of gels made with the tef flours were studied using dynamic-oscillatory measurements. The results are presented in Table 2. The strain sweeps on the gels showed the characteristic behavior of starchy gels, with the initial LVR in which both the elastic (G') and viscous (G") moduli showed constant values until reaching the maximum stress tolerated by the gel without losing its integrity (τ_{max}) and a sudden drop in the moduli until the point where both values are equal (the cross-point), followed by a predominantly viscous behavior. Ultrasonication led to a significant increase in τ_{max} and cross-point compared to the control, while the samples involving MW treatment presented a significant reduction, which was potentiated by the higher MC. It has been reported that US treatments below 40 min avoid damaging effects on starch granules and form stronger gels with improved resistance to deformation (Vela et al., 2021). The lower τ_{max} and cross-point values obtained in the MW samples treated at 30% MC reflect an intense damage to the flour particles, which could be explained by a partial gelatinization of the starch granules, which promotes amylose leaching, and denaturation of the proteins during the MW treatment (Vicente et al., 2023). These effects interfere with the formation of a continuous and well-organized three-dimensional gel network. The resulting structures have weaker bonding zones and reduced resistance to deformation, which explains the lower τ_{max} and cross-point values. In the dual treatments, the data seem to show that the latter applied treatment had an influence on the values of τ_{max} and the cross-point.

The frequency sweeps in the gels were performed from 10 to 1 Hz at a constant strain (1%), within the LVR, to determine the dependence of G', G" and the loss tangent [tan(δ)] of the samples on the applied frequency. All samples showed higher G' values than G" at the studied frequency range, indicative of a predominantly solid/elastic behavior, characteristic of “true gels” (Kaur & Gill, 2019; Vela, Villanueva, Ozturk, et al., 2023). True gels are considered solid-like materials formed by a chemical or physical crosslinking, resulting in the development of a continuous elastic three-dimensional network structure over a wide frequency range (Calafel et al., 2025). The samples treated with MW (both in single and dual treatments) showed significantly lower G'_1 values than the control, with lower values at higher MC, while the US-treated sample showed a ~10% increase. The increased G'_1 value after ultrasonication is mainly consequence of cavitation, given that shorter polymeric chains resulting from starch macromolecule fragmentation can reassociate through hydrogen bonds and form a more elastic gel (Vela, Villanueva, Ozturk, et al., 2023). The G'_1 values obtained for the samples treated with MW at 25% MC (single and dual) were not significantly different among them and showed values closer to the control than the MW-treated samples at 30% MC. A

Table 2
Rheological properties of the gels made using the control and modified tef flours.

Sample	Gel rheological properties							
	τ_{max} (Pa)	Cross-point (Pa)	G'_1 (Pa)	*a*	G''_1 (Pa)	*b*	$\tan(\delta)_1$	*c*
Control	393^{f}	439^{e}	320^{d}	0.038^{b}	48.4^{g}	0.306^{b}	0.151^{c}	0.268^{a}
MW25	161^{d}	213^{c}	281^{c}	0.043^{b}	41.0^{e}	0.318^{bc}	0.146^{bc}	0.275^{b}
MW30	53^{a}	76^{a}	139^{a}	0.071^{c}	26.7^{ab}	0.374^{ef}	0.193^{e}	0.303^{e}
US	422^{g}	494^{f}	351^{e}	-0.009^{a}	45.1^{f}	0.286^{a}	0.128^{a}	0.296^{d}
MW25-US	214^{e}	245^{d}	279^{c}	0.042^{b}	38.7^{d}	0.335^{d}	0.139^{ab}	0.292^{d}
MW30-US	76^{b}	114^{b}	180^{b}	0.043^{b}	28.0^{b}	0.368^{e}	0.155^{c}	0.325^{f}
US-MW25	143^{c}	202^{c}	268^{c}	0.041^{b}	35.1^{c}	0.326^{cd}	0.131^{a}	0.285^{c}
US-MW30	48^{a}	66^{a}	151^{a}	0.081^{c}	25.6^{a}	0.388^{f}	0.170^{d}	0.307^{e}
SE	3	6	5	0.004	0.6	0.004	0.003	0.001
ANOVA	***	***	***	***	***	***	***	***

τ_{max} = maximum stress in the LVR. G'_1, G''_1, $\tan(\delta)_1$ = coefficients obtained from fitting the frequency sweeps data to the Power-law model, representing the elastic and viscous moduli and loss tangent of the gels, respectively. *a*, *b*, *c* = exponents obtained from fitting the frequency sweeps data to the Power-law model, which quantify the dependence of the dynamic moduli and the loss tangent, respectively, to the frequency. SE = Pooled standard error from ANOVA. Different superscript letters in the same column represent statistically significant differences between means at $p < 0.05$. Analysis of variance and significance = *** $p < 0.001$, ** $p < 0.01$, * $p < 0.05$.

moisture-dependent effect of MW treatment on G'_1 of buckwheat gels has been previously reported by Vicente et al. (2023), in which the highest MC (30%) led to a ~52% reduction compared to the control. G' in the tef gels did not show a high dependence on frequency (*a*), which was only significantly modified by US, MW30, and US-MW30. The G''_1 values showed that all treatments led to a significant reduction in this parameter, especially when MW treatments were performed at 30% MC, and that there was a higher dependence on frequency (*b*) than that observed for G'. The lower G''_1 values after the modification of the flours could be due to the straightening of amylose molecules outside the starch granules during the treatments, resulting in a reduction of the viscous component in the gels (Vela, Villanueva, Ozturk, et al., 2023). The treatments resulted in a significant reduction (US, MW25-US, and US-MW25) and increase (MW30 and US-MW30) in $\tan(\delta)_1$ compared to the control, with all treated samples showing a significant increase in the dependence of this parameter on the applied frequency (*c*). Lower $\tan(\delta)_1$ values are indicative of a higher solid-like behavior in the gel, which appears to be caused by ultrasonication. Ultrasonication is believed to cause structural disorganization in flour particles due to cavitation, which allows the reassociation of polymeric chains to a more solid structure when the gel is formed (Monroy et al., 2018). The higher $\tan(\delta)_1$ values obtained for MW30 and US-MW30 could be explained by the partial gelatinization of starch during MW treatment due to the high moisture content of the sample which results in a less rigid gel (Vicente et al., 2023). Excessive molecular mobility under high-moisture MW conditions may disrupt the balance between disentanglement and structural integrity, leading to partial granule damage and impaired formation of ordered bonding zones in the gel network (Zhong et al., 2020).

3.5. Dynamic-oscillatory measurements of doughs

In the case of the doughs, tef flour (both control and treated) represented 30% of the flour-starch mixture used as the basis of the formulation. The data obtained from the dynamic-oscillatory measurements of the doughs are presented in Table 3, and the behavior of some selected samples (control, MW30-US, and US-MW30) is illustrated in Fig. 4 (all curves are presented in Supplementary Fig. 1). The strain sweeps showed a significant increase in τ_{max} and cross-point in all doughs incorporating treated flours. Unlike starchy gels, where excessive molecular mobility may weaken the continuous network, in dough systems the incorporation of structurally modified flours acts as a reinforcing filler within a complex hydrated matrix. The improvement in dough resistance to stress could be caused by cross-linking between starch chains in the amylose portion during treatments, which promote stronger particle–particle and particle–water interactions, resulting in enhanced resistance to deformation (Vicente et al., 2023). The highest increases were observed in the dual-treated samples, mainly in the MW-US-treated samples, in which MW30-US showed a 4.2- and 9.2-fold increase in τ_{max} and the cross-point, respectively, compared to the control dough. It was previously reported that MW treatments can have a structuring impact on GF doughs, which has led to an increase in τ_{max} and the cross-point of doughs containing 30% and 50% MW-treated rice flour (Villanueva et al., 2019), and 25%, 37.5%, 50%, and 75% MW-treated quinoa flour (Vicente et al., 2024). The positive correlation between MC and WAC of tef flours after MW treatments has been indicated to potentially improve the consistency of doughs since MW treatment at higher MC enhances chain mobility and promotes partial reorganization of starch macromolecules, increasing the availability of hydrophilic regions (Náthia-Neves et al., 2023). Upon dough formation, these modified components exhibit improved water binding and interfacial interactions, contributing to a more cohesive and structured dough network. In GF doughs made with ultrasonically treated flours, Vela, Villanueva and Ronda (2023) reported that the incorporation of 30% sonicated rice flour did not significantly alter τ_{max} and the cross-point. In the present study, the US treatment resulted in a significant increase in τ_{max} and the cross-point, however it was the lowest increase among treated flours.

In the frequency sweeps, the results showed that G'_1 and G''_1 were increased by the treatments, resulting in a significant reduction in $\tan(\delta)_1$. The dual-treated samples showed greater reductions in $\tan(\delta)_1$: US-MW25 (~21%) < US-MW30 and MW25-US (~24%) < MW30-US (~32%), compared with the control, indicating a more solid-like behavior than the doughs containing flours treated with a single treatment. Previous studies have reported that the incorporation of MW-treated flours can lead to more stable dough structures, particularly treatments at higher MC (Vicente et al., 2024; Villanueva et al., 2019). Yang et al. (2017) reported that MW treatment of waxy maize starch at 160 W/g and 30% MC increased the proportion of longer B-type amylopectin chains at the expense of A chains. Longer B-type chains act as inter-cluster connectors in amylopectin, which can enhance network connectivity and elastic response [higher G'_1 and lower $\tan(\delta)_1$]. This effect does not imply the synthesis of new long chains, but rather a shift in chain-length distribution toward longer chains, likely due to a reduction in short A chains or an increased proportion of B chains associated with stronger inter-cluster interactions (Yang et al., 2019). In dough system, this enhanced inter-cluster connectivity promotes the formation of a network in which starch-rich domains contribute to elastic load-bearing structures, resulting in higher G'_1 values and lower $\tan(\delta)_1$. The reduction in $\tan(\delta)_1$ observed in doughs containing

Table 3
Rheological properties of the doughs made incorporating the control and modified tef flours.

Sample	Dough rheological properties							
	τ_{max} (Pa)	Cross-point (Pa)	G'_1 (Pa)	*a*	G''_1 (Pa)	*b*	$\tan(\delta)_1$	*c*
Control	3.4^{a}	20^{a}	912^{a}	0.315^{d}	553^{a}	0.297^{d}	0.606^{d}	-0.018^{ab}
MW25	6.4^{c}	40^{b}	1223^{b}	0.286^{c}	691^{bc}	0.259^{bc}	0.565^{c}	-0.028^{a}
MW30	6.4^{c}	48^{b}	1546^{c}	0.274^{c}	842^{d}	0.247^{b}	0.547^{c}	-0.027^{a}
US	5.4^{b}	41^{b}	1113^{b}	0.294^{cd}	628^{ab}	0.278^{cd}	0.565^{c}	-0.016^{b}
MW25-US	10.4^{e}	115^{d}	1754^{c}	0.232^{ab}	802^{cd}	0.240^{ab}	0.460^{b}	0.007^{c}
MW30-US	14.4^{f}	184^{e}	2942^{e}	0.210^{a}	1216^{e}	0.221^{a}	0.413^{a}	0.010^{c}
US-MW25	8.0^{d}	76^{c}	1598^{c}	0.245^{b}	767^{cd}	0.246^{b}	0.481^{b}	0.001^{c}
US-MW30	9.9^{e}	116^{d}	2401^{d}	0.232^{ab}	1106^{e}	0.239^{ab}	0.461^{b}	0.006^{c}
SE	0.3	3	78	0.009	39	0.008	0.008	0.003
ANOVA	***	***	***	***	***	**	***	***

τ_{max} = maximum stress in the LVR. G'_1, G''_1, $\tan(\delta)_1$ = coefficients obtained from fitting the frequency sweeps data to the Power-law model, representing the elastic and viscous moduli and loss tangent of the doughs, respectively. *a*, *b*, *c* = exponents obtained from fitting the frequency sweeps data to the Power-law model, which quantify the dependence of the dynamic moduli and the loss tangent, respectively, to the frequency. SE = Pooled standard error from ANOVA. Different superscript letters in the same column represent statistically significant differences between means at $p < 0.05$. Analysis of variance and significance = *** $p < 0.001$, ** $p < 0.01$, * $p < 0.05$.

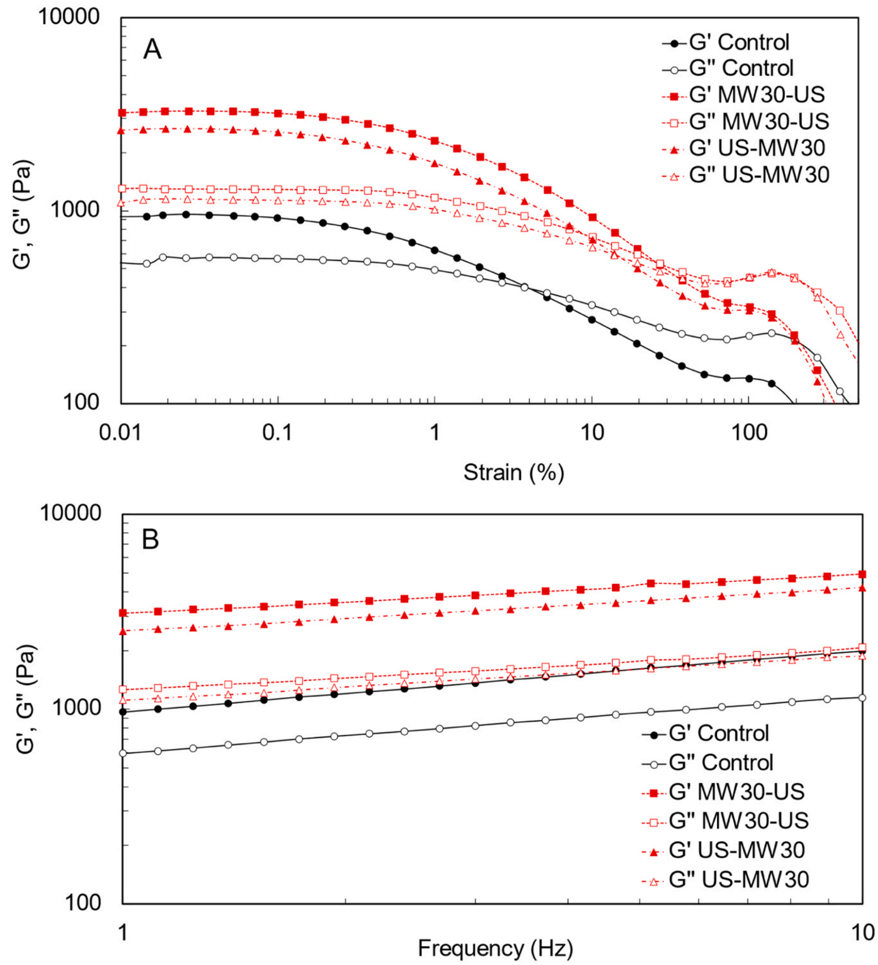


Fig. 4. Rheological properties of the control dough and doughs with dual-treatments MW30-US and US-MW30. (A) The elastic (G') and viscous (G") moduli in the strain sweep and (B) the G' and G" moduli in the frequency sweep.

US-treated flours has been attributed to the polymeric fragmentation of starch macromolecules induced by cavitation, resulting in shorter linear chains capable of reassociating and forming a more stable structure (Monroy et al., 2018; Qin et al., 2022). The lower $\tan(\delta)_1$ values obtained with the dual treatments suggest a synergistic structuring effect of combining MW and US. The greater modification caused by dual treatments to flour components improve their interaction with water, as reflected in the WAC and SP parameters, and facilitates intermolecular connectivity, thereby increasing dough consistency. This synergistic effect likely arises from the complementary mechanisms of MW-induced molecular rearrangement and US-induced chain fragmentation, which together increase the number of interaction sites and promote a more

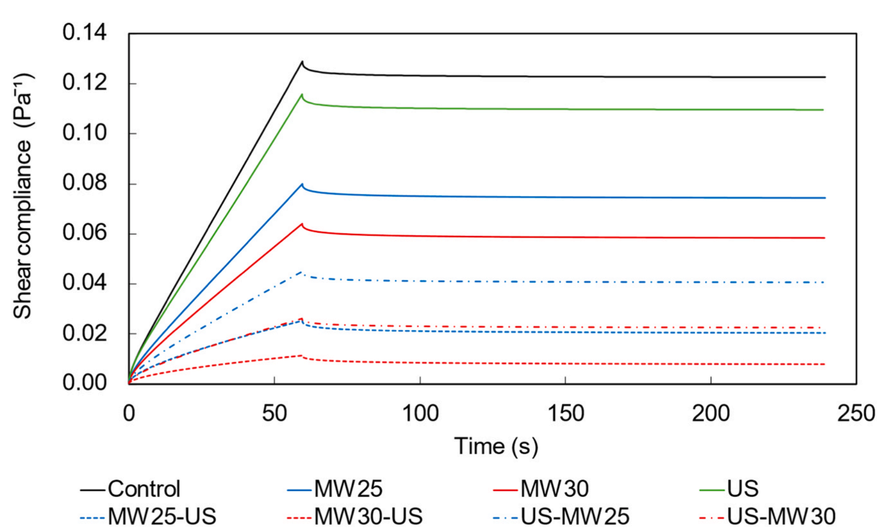


Fig. 5. Creep-recovery tests performed on doughs containing the studied flours.

interconnected and mechanically stable dough network. The results obtained for *a* and *b* showed that the incorporation of treated tef flours led to a lower dependence of both G′ and G" on the applied frequency, denoting more stable moduli. The values of *c* were negative in the control dough and in the doughs containing single-treated flours, but became positive in the doughs made with dual-treated flours. Furthermore, the *c* values in the dual-treated doughs were closer to 0 than those of the control and the single-treated MW and US samples, indicating improved stability in these doughs.

3.6. Creep-recovery measurements of dough

The curves obtained from the creep-recovery tests are presented in Fig. 5, and the results obtained from fitting the data to the 4-parameter (creep phase) and 3-parameter (recovery phase) Burges models are presented in Table 4. The creep phase was performed at 50 Pa, outside of the LVR (OLVR), because stresses outside the maximum stress withstand by the doughs are closer to those applied during bread making (Villanueva et al., 2021). The curves obtained with all the doughs showed the viscoelastic behavior characteristic of GF doughs (Vela, Villanueva, & Ronda, 2023; Vicente et al., 2024). The creep-recovery curves OLVR showed a predominantly viscous component, with an almost direct proportionality between compliance and time in the creep phase and an almost horizontal response in the recovery phase (Villanueva et al., 2019). The incorporation of treated flours into the doughs resulted in lower compliance curves, particularly in those containing dual-treated flours, indicating less deformable doughs. The instantaneous compliance in both the creep and recovery phases (J_{0c} and J_{0r}) are related to the energy of elastic stretching of the bonds in viscoelastic materials when stress is applied, which vanishes immediately after its removal, while the retarded elastic compliances (J_{1c} and J_{1r}) relate to the disruption and conversion of the bonds (Witczak et al., 2012). The use of physically modified tef flours in the doughs did not have a remarkable effect on J_{0c} and J_{1c}. It only led to a significant reduction in doughs containing flours treated with dual treatments at the highest MC (MW30-US and US-MW30). A reduction in J_{0c} and J_{1c} has been previously reported in doughs containing MW-treated quinoa flour, indicating a lower deformation for the same applied stress (Vicente et al., 2024). However, in the recovery phase, the use of treated tef flours led to a significant reduction in J_{0r} and J_{1r}, except for J_{0r} in the dough containing the US-treated sample. The highest reductions in J_{0r} and J_{1r} were observed for MW30-US (~55% and ~42%, respectively) and US-MW30 (~54% and ~44%, respectively), consistent with the behavior observed in the creep phase. These results are in agreement with the higher consistency of the MW30-US and US-MW30 doughs, as reflected by their higher G'_1 and G''_1 values and lower $\tan(\delta)_1$. This effect is believed to be derived from the magnified effect of the MW treatment because of the high MC, and potentiated by the particle and macromolecular fragmentation due to the US treatment. These results suggest the presence of stronger and more stable bonding zones within the modified dough matrix (Zhong et al., 2020). The lower values of the instantaneous and retarded elastic compliances indicate that the doughs containing single- and dual-treated flours would expand less under the pressure of fermentation gas during proofing and baking (Villanueva et al., 2021).

The retardation time (λ) characterizes the response of the dough to the instantaneous application of a constant stress (Ronda et al., 2017). The retardation times in the creep phase (λ_c) and recovery phase (λ_r) were significantly increased in almost all doughs containing treated flours, except for the US-treated tef sample. The maximum increases were determined for the doughs containing MW-US-treated flours, representing an increase of > 300% (λ_c) and > 80% (λ_r) compared to the control. A higher retardation time indicates a longer time required to obtain the viscoelastic deformation of the dough, indicating a greater resistance to deformation (Villanueva et al., 2019). The increase in retardation time is likely related to the increase in swelling capacity and overall interaction with water of the treated tef flours, since hydration capacity can change the consistency of the doughs by shaping the interconnectivity of starch and hydrocolloids (Witczak et al., 2012). This behavior can be attributed to the plasticizing effect of water during MW treatment, which enhances molecular mobility and promotes partial rearrangement of starch chains. Upon dough formation, these reorganized structures exhibit enhanced hydration-driven swelling and stronger water-mediated interactions, which slow down deformation processes and increase the characteristic response times of the system (Vicente et al., 2025). Consistently, higher retardation times have also been reported in GF doughs with the incorporation of MW-treated (Villanueva et al., 2019) and US-treated rice flours (Vela, Villanueva, & Ronda, 2023) compared to their counterparts made with untreated flours. All treatments significantly increased the steady-state viscosity (μ_0) of the doughs compared to the control, except for the US treatment, which did not show a significant difference to the control. The single MW treatments moderately increased μ_0, by ~60% above the control value, with no significant difference between 25% and 30% MC. In contrast, dual treatments produced the strongest effects, particularly MW30-US, which increased μ_0 to 7637 Pa•s, (~1600% higher than the control dough), characteristic of a viscoelastic network in which starch-rich domains and fragmented polymeric chains are strongly interconnected through hydrogen bonding. The substantial increase in μ_0 is consistent with the reductions observed in compliance parameters and the enhanced elastic response identified in dynamic-oscillatory measurements, collectively confirming the creation of a robust cross-linked dough structure (Villanueva et al., 2021). Ronda et al. (2014) reported that a proper consistency, with high enough G′ and G" moduli and viscosity, μ_0, helps to retain the carbon dioxide produced during bread fermentation. However, doughs with excessively low J_0

Table 4
Creep-recovery parameters of the doughs obtained after fitting the data to the 4- and 3-parameter Burgers-models.

Sample	J_{0c} (10^{-5} Pa^{-1})	J_{1c} (10^{-5} Pa^{-1})	λ_c (s)	μ_0 (Pa•s)	J_{0r} (10^{-5} Pa^{-1})	J_{1r} (10^{-5} Pa^{-1})	λ_r (s)	Recovery (%)
Control	95^{bc}	550^{c}	1.5^{a}	487^{a}	194^{e}	418^{e}	9.9^{a}	4.9^{a}
MW25	88^{b}	501^{c}	2.6^{b}	788^{b}	163^{d}	389^{d}	13.7^{b}	7.0^{b}
MW30	87^{b}	499^{c}	3.3^{c}	994^{b}	152^{c}	388^{d}	14.9^{c}	8.5^{c}
US	106^{c}	544^{c}	1.4^{a}	581^{a}	186^{e}	393^{d}	10.1^{a}	5.5^{a}
MW25-US	91^{b}	515^{c}	6.3^{f}	3128^{d}	131^{b}	345^{c}	17.9^{d}	19.9^{f}
MW30-US	57^{a}	316^{a}	6.6^{f}	7637^{e}	88^{a}	243^{a}	20.7^{e}	30.4^{g}
US-MW25	96^{bc}	520^{c}	4.6^{d}	1554^{c}	128^{b}	301^{b}	13.5^{b}	10.0^{d}
US-MW30	62^{a}	372^{b}	5.4^{e}	3363^{d}	89^{a}	235^{a}	17.7^{d}	15.1^{e}
SE	4	18	0.2	120	4	8	0.3	0.4
p-value	***	***	***	***	***	***	***	***

The subindex "c" refers to parameters determined in the creep phase, and "r" refers to parameters corresponding to the recovery phase. J_0 = Instantaneous compliance, J_1 = retarded elastic compliance, λ = retardation time, μ_0 = steady-state viscosity. SE = Pooled standard error from ANOVA. Different superscript letters in the same column represent statistically significant differences between means at $p < 0.05$. Analysis of variance and significance = *** $p < 0.001$, ** $p < 0.01$, * $p < 0.05$.

and J_1 compliances could restrict dough expansion and lead to a lower development in breads. These results are consistent with those of Villanueva et al. (2019) and Vicente et al. (2024), who reported an increase in μ_0 measured OLVR of doughs made with MW-treated rice flour and buckwheat flour, respectively, both at 30% MC.

The recovery capacity of the doughs after the creep-recovery tests ranged between 5.5% and 30.4% when using treated tef flours, compared to 4.9% for the control dough (Table 4). This parameter relates the contribution of the elastic deformation to the total deformation and indicates the ability of the dough to return to its original state after the applied stress is removed (Villanueva et al., 2019). The increase was particularly high when using flours with dual treatments. The results indicate that the order of application of treatments and the MC level have a significant effect on the flour. The MW30-US dough, which exhibited significantly greater elastic behavior compared to the control, showed the highest recovery capacity. Since elasticity reflects the degree of bonding between the structural elements of the dough, this increase could indicate less deformation or breakage of the network structure when using MW-US-treated flours (Skendi et al., 2009), which could be promoted by higher moisture content. Mechanistically, the initial MW treatment likely increases molecular mobility by disrupting hydrogen bonds within amorphous and crystalline starch regions, while the subsequent US treatment promotes macromolecular fragmentation and surface activation. This sequential modification enhances intermolecular connectivity during dough formation, resulting in a more elastic and structurally resilient network (Solaesa et al., 2021; Vela, Villanueva, Li, et al., 2023).

3.7. Principal component analysis

Principal component analysis (PCA) was employed to visually represent the correlation between the properties studied and the effect of the treatments over the tef flour (see Fig. 6, and the PCA loadings in Supplementary Table 1). The statistical analysis explained 83.15% of the total variation, of which 68.03% corresponded to the first principal component (PC1) and 15.12% to the second principal component (PC2). All the properties studied were included in the analysis, where the flour- and gel-related properties are shown in black in Fig. 6A, and the dough-related properties are depicted in blue. Most of the flour- and gel-related properties appeared in the upper-right quadrant (+PC1, +PC2), while the dough-related properties were distributed almost evenly between the upper-left (-PC1, +PC2) and lower-right (+PC1, -PC2) quadrants (Fig. 6A). This distribution of parameters resulted in the PCA score plot presented in Fig. 6B. The extent of modification achieved in terms of the Euclidean distances between each modified sample and the control was US < MW25 < US-MW25 < MW25-US < MW30 < US-MW30 < MW30-US. These results indicate that MC was the most influential factor, and that dual treatments had a greater effect than single treatments, particularly when MW was applied prior to US.

Samples grouping was distinct along the PCs: MW treatment tended to drive the samples towards negative PC1, directly associated with the increase in D_{50}, WAC, WSI, and $\tan(\delta)_{1\text{-gel}}$, and the lower values of pasting properties (PV, SV, FV, BV, and TV) and gel rheological properties ($G'_{1\text{-gel}}$, $G''_{1\text{-gel}}$, $\tau_{max\text{-gel}}$, and Cross-point$_{gel}$). US treatment tended to cluster the samples on the positive side of PC2, associated closely with the flour techno-functional properties, WAI, SP, and the values of gel rheological properties ($G'_{1\text{-gel}}$, $G''_{1\text{-gel}}$, $\tau_{max\text{-gel}}$, and Cross-point$_{gel}$), indicating that changes in particle size [lower D_{50} and higher $(D_{90} - D_{10})/D_{50}$] due to acoustic cavitation played an important role in the extent of modification caused by ultrasonication. The dual treatments were positioned following both behaviors (+PC2 due to US, and -PC1 due to MW) with a greater influence by the latter applied treatment. The contribution of MC during MW treatment can be seen as a displacement in PC1, with higher MC moving the plots towards -PC1 compared to the control. When comparing the effect of the physical modifications of the flour on their applications (gels and doughs), the PCA showed that the gelatinization behavior and the gel rheological properties were closer aligned with the US treatment, while the effect of MW treatment was closer to some flour-related properties [PT, WSI, WAC, D_{50}] and gel properties [a_{gel}, b_{gel} and $\tan(\delta)_{1\text{-gel}}$]. The dual treatments were directly aligned to several dough rheological properties [$G'_{1\text{-dough}}$, $G''_{1\text{-dough}}$, c_{dough}, $\tau_{max\text{-dough}}$, Cross-point$_{dough}$, μ_0, λ_c, λ_r, Recovery]. These results confirmed the significant influence that the order of modifications applied, and the MC of the sample during MW treatment have on the extent of modification achieved in the samples, in which the dual treatments enhanced the doughs' dynamic-oscillatory parameters, emphasizing the structuring effect achieved in doughs by the application of dual treatments combining MW and US.

4. Conclusion

Single and dual US and MW treatments induced distinct and controllable structural modifications in tef flour, affecting particle morphology, hydration properties and rheological behavior. US primarily promoted particle fragmentation, whereas MW induced particle agglomeration, with dual treatments combining both effects. The dual treatments exhibited a synergistic effect on the extent of modification achieved, which was strongly governed by the order of application and the moisture content during MW treatment. The higher MC during MW treatment resulted in a greater degree of modification of the tef flour,

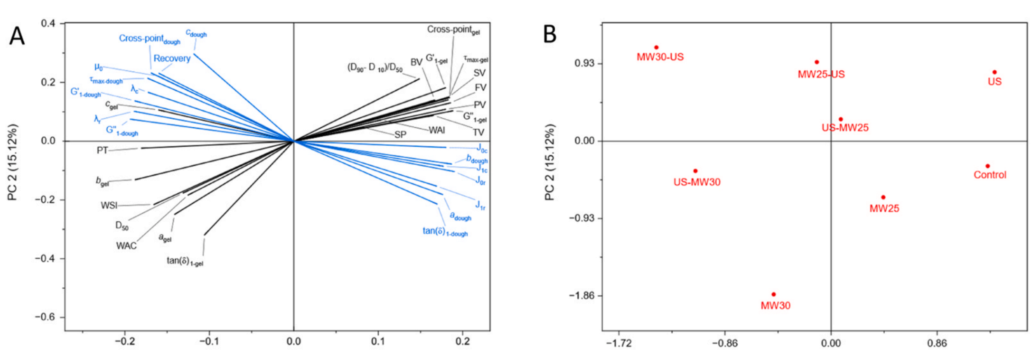


Fig. 6. Principal component analysis (PCA). (A) Parameters studied. The flour and gel-related properties are shown in black, and the dough-related properties are shown in blue. (B) The sample scores. D_{50} = median diameter, $(D_{90} - D_{10})/D_{50}$ = size dispersion, WAC = water absorption capacity, WAI = water absorption index, WSI = water solubility index, SP = swelling power, PT = pasting temperature, PV = peak viscosity, TV = trough viscosity, BV = breakdown viscosity, FV = final viscosity, SV = setback viscosity. τ_{max} = maximum stress in the LVR. G'_1, G''_1, $\tan(\delta)_1$ = the elastic and viscous moduli and loss tangent, respectively. a, b, c = exponents obtained from the Power-law model. J_0 = Instantaneous compliance, J_1 = retarded elastic compliance, λ = retardation time, μ_0 = steady-state viscosity. The subindex "c" refers to parameters of the creep phase, and "r" refers to parameters of the recovery phase.

resulting in modified flours with improved water interaction, which led to gels with increased stability and lower resistance to deformation, and doughs with increased τ_{max} and cross-point, reduced $\tan(\delta)_1$ and improved recovery capacity. The sequence-dependent response observed in dual treatments suggests that the impact of MW on starch structures creates a matrix more susceptible to mechanical disruption by US treatment, whereas initial particle fragmentation by US limits the extent of further MW-induced reorganization. While these findings support a structure-function relationship driven by the order of modification applied, future research should focus on understanding the molecular mechanisms explaining the contributions of flour components in the modifications by sequential physical treatments.

From a scientific perspective, this study provides new insight into how dual physical modifications can be strategically applied to tailor the functionality of gluten-free whole-grain flours, extending current understanding beyond single physical treatments. From an industrial point of view, the ability to enhance dough strength and elastic recovery through exclusively physical, additive-free modifications offers a promising alternative for improving the quality and processability of GF products while maintaining clean-label formulations.

CRediT authorship contribution statement

Caleb S. Calix-Rivera: Writing – original draft, Visualization, Methodology, Formal analysis, Data curation, Conceptualization. **Marina Villanueva:** Writing – review & editing, Visualization, Validation, Supervision, Project administration, Methodology, Investigation, Data curation, Conceptualization. **Felicidad Ronda:** Writing – review & editing, Visualization, Validation, Supervision, Resources, Project administration, Funding acquisition, Conceptualization. **Maricela E. Ordoñez:** Methodology, Investigation, Formal analysis. **Antonio J. Vela:** Writing – review & editing, Visualization, Validation, Supervision, Methodology, Investigation, Data curation, Conceptualization.

Declaration of Competing 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.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.foostr.2026.100520.

Data availability

Data will be made available on request.

References

AACC International Approved Methods. (2017). *Method 76-21.02. General pasting method for wheat or rye flour of starch using the rapid visco analyser* (11th ed.). Cereals & Grains Association. https://doi.org/10.1094/aaccintmethod-76-21.01

Acevedo, B. A., Villanueva, M., Chaves, M. G., Avanza, M. V., & Ronda, F. (2022). Modification of structural and physicochemical properties of cowpea (*Vigna unguiculata*) starch by hydrothermal and ultrasound treatments. *Food Hydrocolloids, 124*(A), Article 107266. https://doi.org/10.1016/j.foodhyd.2021.107266

Amini, A. M., Razavi, S. M. A., & Mortazavi, S. A. (2015). Morphological, physicochemical, and viscoelastic properties of sonicated corn starch. *Carbohydrate Polymers, 122*, 282–292. https://doi.org/10.1016/j.carbpol.2015.01.020

Awika, J. M. (2011). Major cereal grains production and use around the world. In , *ACS symposium series: 1089. Advances in cereal science: Implications to food processing and health promotion* (pp. 1–13). https://doi.org/10.1021/bk-2011-1089.ch001

Bultosa, G., Hall, A. N., & Taylor, J. R. N. (2002). Physico-chemical characterization of grain tef [*Eragrostis tef* (Zucc.) Trotter] starch. *Starch/Staerke, 54*, 461–468. https://doi.org/10.1002/1521-379X(200210)54:10<461::AID-STAR461>3.0.CO;2-U

Calafel, M. I., Criado-Gonzalez, M., Aguirresarobe, R., Fernández, M., & Mijangos, C. (2025). From rheological concepts to additive manufacturing assessment of hydrogel-based materials for advanced bioprinting applications. *Materials Advances, 6*, 4566–4597. https://doi.org/10.1039/D5MA00019J

Calix-Rivera, C. S., Villanueva, M., Náthia-Neves, G., & Ronda, F. (2023). Changes on techno-functional, thermal, rheological, and microstructural properties of tef flours induced by microwave radiation—Development of new improved gluten-free ingredients. *Foods, 12*(6), 1345. https://doi.org/10.3390/foods12061345

Chan, H. T., Bhat, R., & Karim, A. A. (2010). Effects of sodium dodecyl sulphate and sonication treatment on physicochemical properties of starch. *Food Chemistry, 120* (3), 703–709. https://doi.org/10.1016/j.foodchem.2009.10.066

Fan, D., Ma, S., Wang, L., Zhao, H., Zhao, J., Zhang, H., & Chen, W. (2013). ^{1}H NMR studies of starch-water interactions during microwave heating. *Carbohydrate Polymers, 97*(2), 406–412. https://doi.org/10.1016/j.carbpol.2013.05.021

Jambrak, A. R., Herceg, Z., Šubarić, D., Babić, J., Brnčić, M., Brnčić, S. R., Bosiljkov, T., Čvek, D., Tripalo, B., & Gelo, J. (2010). Ultrasound effect on physical properties of corn starch. *Carbohydrate Polymers, 79*(1), 91–100. https://doi.org/10.1016/j.carbpol.2009.07.051

Kaur, H., & Gill, B. S. (2019). Effect of high-intensity ultrasound treatment on nutritional, rheological and structural properties of starches obtained from different cereals. *International Journal of Biological Macromolecules, 126*, 367–375. https://doi.org/10.1016/j.ijbiomac.2018.12.149

Kumar, V., Sharma, H. K., & Singh, K. (2017). Effect of precooking on drying kinetics of taro (*Colocasia esculenta*) slices and quality of its flours. *Food Bioscience, 20*, 178–186. https://doi.org/10.1016/j.fbio.2017.10.003

Lai, H. (2001). Effects of hydrothermal treatment on the physicochemical properties of pregelatinized rice flour. *Food Chemistry, 72*(4), 455–463. https://doi.org/10.1016/S0308-8146(00)00261-2

Landberg, R., Hanhineva, K., Tuohy, K., Garcia-Aloy, M., Biskup, I., Llorach, R., Yin, X., Brennan, L., & Kolehmainen, M. (2019). Biomarkers of cereal food intake. *Genes and Nutrition, 14*, 28. https://doi.org/10.1186/s12263-019-0651-9

Li, M., Li, J., & Zhu, C. (2018). Effect of ultrasound pretreatment on enzymolysis and physicochemical properties of corn starch. *International Journal of Biological Macromolecules, 111*, 848–856. https://doi.org/10.1016/j.ijbiomac.2017.12.156

Luo, Z., Fu, X., He, X., Luo, F., Gao, Q., & Yu, S. (2008). Effect of ultrasonic treatment on the physicochemical properties of maize starches differing in amylose content. *Starch/Staerke, 60*(11), 646–653. https://doi.org/10.1002/star.200800014

Luo, Z., He, X., Fu, X., Luo, F., & Gao, Q. (2006). Effect of microwave radiation on the physicochemical properties of normal maize, waxy maize and amylomaize V starches. *Starch/Staerke, 58*(9), 468–474. https://doi.org/10.1002/star.200600498

Marta, H., Cahyana, Y., Bintang, S., Soeherman, G. P., & Djali, M. (2022). Physicochemical and pasting properties of corn starch as affected by hydrothermal modification by various methods. *International Journal of Food Properties, 25*(1), 792–812. https://doi.org/10.1080/10942912.2022.2064490

Monroy, Y., Rivero, S., & García, M. A. (2018). Microstructural and techno-functional properties of cassava starch modified by ultrasound. *Ultrasonics Sonochemistry, 42*, 795–804. https://doi.org/10.1016/j.ultsonch.2017.12.048

Náthia-Neves, G., Calix-Rivera, C. S., Villanueva, M., & Ronda, F. (2023). Microwave radiation induces modifications in the protein fractions of tef flours and modulates their derived techno-functional properties. *International Journal of Biological Macromolecules, 253*, Article 126908. https://doi.org/10.1016/j.ijbiomac.2023.126908

Nyakabau, T., Wokadala, O. C., & Emmambux, M. N. (2013). Effect of steeping additives on tef starch extraction and its quality. *Starch/Staerke, 65*, 738–746. https://doi.org/10.1002/star.201200241

Perraulta Lavanya, J., Gowthamraj, G., & Sangeetha, N. (2021). Effect of heat moisture treatment on the physicochemical, functional, and antioxidant characteristics of white sorghum (*Sorghum bicolor* (L.) grains and flour. *Journal of Food Processing and Preservation, 45*, Article e16017. https://doi.org/10.1111/jfpp.16017

Qin, W., Xi, H., Wang, A., Gong, X., Chen, Z., He, Y., Wang, L., Liu, L., Wang, F., & Tong, L. (2022). Ultrasound treatment enhanced semidry-milled rice flour properties and gluten-free rice bread quality. *Molecules, 27*(17), 5403. https://doi.org/10.3390/molecules27175403

Kurek, M. A., Wojtasik-Kalinowska, I., Marcinkowska-Lesiak, M., Onopiuk, A., Szpicer, A., Kultys, E., & Zalewska, M. (2024). Effect of microwaves on food carbohydrates. In *Microwave processing of foods: Challenges, advances and prospects* (pp. 221–249). Springer Nature. https://doi.org/10.1007/978-3-031-51613-9_12.

Ronda, F., Villanueva, M., & Collar, C. (2014). Influence of acidification on dough viscoelasticity of gluten-free rice starch-based dough matrices enriched with exogenous protein. *LWT - Food Science and Technology, 59*, 12–20. https://doi.org/10.1016/j.lwt.2014.05.052

Ronda, F., Pérez-Quirce, S., & Villanueva, M. (2017). Rheological properties of gluten-free bread doughs: Relationship with bread quality. In *Advances in food rheology and* *its applications* (pp. 297–334). Elsevier Inc. https://doi.org/10.1016/B978-0-08-100431-9.00012-7.

Skendi, A., Papageorgiou, M., & Biliaderis, C. G. (2009). Effect of barley β-glucan molecular size and level on wheat dough rheological properties. *Journal of Food Engineering, 91*(4), 594–601. https://doi.org/10.1016/j.jfoodeng.2008.10.009

Solaesa, Á. G., Villanueva, M., Muñoz, J. M., & Ronda, F. (2021). Dry-heat treatment vs. heat-moisture treatment assisted by microwave radiation: Techno-functional and rheological modifications of rice flour. *LWT, 141*, Article 110851. https://doi.org/10.1016/j.lwt.2021.110851

Vela, A. J., Villanueva, M., Li, C., Hamaker, B., & Ronda, F. (2023a). Ultrasound treatments of tef [*Eragrostis tef* (Zucc.) Trotter] flour rupture starch α-(1,4) bonds and fragment amylose with modification of gelatinization properties. *LWT, 174*, Article 114463. https://doi.org/10.1016/j.lwt.2023.114463

Vela, A. J., Villanueva, M., Ozturk, O. K., Hamaker, B., & Ronda, F. (2023b). Modification of the microstructure of tef [*Eragrostis tef* (Zucc.) Trotter] flour ultrasonicated at different temperatures. Impact on its techno-functional and rheological properties. *Current Research in Food Science, 6*, Article 100456. https://doi.org/10.1016/j.crfs.2023.100456

Vela, A. J., Villanueva, M., & Ronda, F. (2023c). Physical modification caused by acoustic cavitation improves rice flour bread-making performance. *LWT, 183*, Article 114950. https://doi.org/10.1016/j.lwt.2023.114950

Vela, A. J., Villanueva, M., Solaesa, Á. G., & Ronda, F. (2021). Impact of high-intensity ultrasound waves on structural, functional, thermal and rheological properties of rice flour and its biopolymers structural features. *Food Hydrocolloids, 113*, Article 106480. https://doi.org/10.1016/j.foodhyd.2020.106480

Vicente, A., Villanueva, M., Caballero, P. A., Lazaridou, A., Biliaderis, C. G., & Ronda, F. (2024). Microwave treatment enhances the physical and sensory quality of quinoa-enriched gluten-free bread. *Food Hydrocolloids, 155*, Article 110244. https://doi.org/10.1016/j.foodhyd.2024.110244

Vicente, A., Villanueva, M., Caballero, P. A., Muñoz, J. M., & Ronda, F. (2023). Buckwheat grains treated with microwave radiation: Impact on the techno-functional, thermal, structural, and rheological properties of flour. *Food Hydrocolloids, 137*, Article 108328. https://doi.org/10.1016/j.foodhyd.2022.108328

Vicente, A., Villanueva, M., Muñoz, J. M., Caballero, P. A., & Ronda, F. (2025). The role of microwave absorption capacity and water mobility in the microwave treatment of grain vs. flour: Impact on the treated flour characteristics. *Food Hydrocolloids, 159*, Article 110680. https://doi.org/10.1016/j.foodhyd.2024.110680

Villanueva, M., Abebe, W., Collar, C., & Ronda, F. (2021). Tef [*Eragrostis tef* (Zucc.) Trotter] variety determines viscoelastic and thermal properties of gluten-free dough and bread quality. *LWT, 135*, Article 110065. https://doi.org/10.1016/j.lwt.2020.110065

Villanueva, M., Harasym, J., Muñoz, J. M., & Ronda, F. (2019). Rice flour physically modified by microwave radiation improves viscoelastic behavior of doughs and its bread-making performance. *Food Hydrocolloids, 90*, 472–481. https://doi.org/10.1016/j.foodhyd.2018.12.048

Wang, M., Wu, Y., Liu, Y., & Ouyang, J. (2020). Effect of ultrasonic and microwave dual-treatment on the physicochemical properties of chestnut starch. *Polymers, 12*(8), 1718. https://doi.org/10.3390/POLYM12081718

Williams, P. G. (2012). Evaluation of the evidence between consumption of refined grains and health outcomes. *Nutrition Reviews, 70*(2), 80–99. https://doi.org/10.1111/j.1753-4887.2011.00452.x

Witczak, M., Juszczak, L., Ziobro, R., & Korus, J. (2012). Influence of modified starches on properties of gluten-free dough and bread. Part I: Rheological and thermal properties of gluten-free dough. *Food Hydrocolloids, 28*(2), 353–360. https://doi.org/10.1016/j.foodhyd.2012.01.009

Yang, Q., Lu, X., Chen, Y., Lou, Z., & iao, Z. (2019). Fine structure, crystalline and physicochemical properties of waxy corn starch treated by ultrasound irradiation. *Ultrasonics - Sonochemistry, 51*, 350–358. https://doi.org/10.1016/j.ultsonch.2018.09.001

Yang, Q., Qi, L., Luo, Z., Kong, X., Xiao, Z., Wang, P., & Peng, X. (2017). Effect of microwave irradiation on internal molecular structure and physical properties of waxy maize starch. *Food Hydrocolloids, 69*, 473–482. https://doi.org/10.1016/j.foodhyd.2017.03.011

Yılmaz, A., & Tugrul, N. (2023). Effect of ultrasound-microwave and microwave-ultrasound treatment on physicochemical properties of corn starch. *Ultrasonics Sonochemistry, 98*, Article 106516. https://doi.org/10.1016/j.ultsonch.2023.106516

You, W., & Henneberg, M. (2016). Cereal crops are not created equal: Wheat consumption associated with obesity prevalence globally and regionally. *AIMS Public Health, 3*(2), 313–328. https://doi.org/10.3934/publichealth.2016.2.313

Zavareze, E. D. R., & Dias, A. R. G. (2011). Impact of heat-moisture treatment and annealing in starches: A review. *Carbohydrate Polymers, 83*(2), 317–328. https://doi.org/10.1016/j.carbpol.2010.08.064

Zavareze, E. D. R., Storck, C. R., de Castro, L. A. S., Schirmer, M. A., & Dias, A. R. G. (2010). Effect of heat-moisture treatment on rice starch of varying amylose content. *Food Chemistry, 121*(2), 358–365. https://doi.org/10.1016/j.foodchem.2009.12.036

Zhong, Y., Xiang, X., Zhao, J., Wang, X., Chen, R., Xu, J., Luo, S., Wu, J., & Liu, C. (2020). Microwave pretreatment promotes the annealing modification of rice starch. *Food Chemistry, 304*, Article 125432. https://doi.org/10.1016/j.foodchem.2019.125432

Zhou, Y., Wang, M., Wang, L., Liu, L., Wu, Y., & Ouyang, J. (2023). Comparison of the effect of ultrasound and microwave on the functional properties and in vitro digestibility of normal maize starch and potato starch. *Journal of Food Process Engineering, 46*(2), Article e14222. https://doi.org/10.1111/jfpe.14222

Zhu, F. (2018). Chemical composition and food uses of teff (*Eragrostis tef*). *Food Chemistry, 239*, 402–415. https://doi.org/10.1016/j.foodchem.2017.06.101