



Universidad Autónoma de Chihuahua
Dirección de Planeación y Desarrollo Institucional

**Listado de información con la que debe contar
el Reporte Técnico del Proyecto**

1. Institución u Organismo: Universidad Autónoma de Chihuahua
2. Nombre del proyecto: “Aprovechamiento Integral de Recursos Regionales para la Obtención de Productos Alimenticios y Subproductos de Valor Agregado Mediante la Aplicación de Procesos de Conservación de Alimentos y Bioprocesos”
3. No. convenio: IDCA 11468
4. Responsable del proyecto: No. Empleado 7193, No. de Folio Promep UACH-CA-122
5. Fecha: Fecha de Informe 17/Diciembre/2013, Primer trimestre
6. Periodo de vigencia del proyecto: Inicio 12/Agosto/2013 – Termino 12/Agosto/2014
7. Reporte de actividades desarrolladas en el proyecto con base en los objetivos y metas: (Descripción detallada, presentar un análisis describiendo cada una de las actividades realizadas en relación al cumplimiento de los objetivos y metas propuestos, incorporar hojas, no más de 3 cuartillas)

OBJETIVO 1: Evaluar los cambios en el contenido de antocianinas, propiedades físicas y químicas durante la nixtamalización de maíz azul en hidróxido y lactato de calcio con y sin ultrasonido.

- a. Cotización de materiales y reactivos para realizar las actividades necesarias para el cumplimiento este objetivo
- b. Caracterización fisicoquímica del maíz azul
- c. Caracterización física, química, propiedades térmicas y estructurales de grano, harina, masa y tortilla
- d. Análisis datos experimentales obtenidos a partir de la caracterización física, química, propiedades térmicas y estructurales de grano, harina, masa y tortilla
- e. Elaboración de manuscrito para publicación en revista arbitrada indizada
- f. Envío y seguimiento del manuscrito para su revisión en International Journal of Food Science and Technology
- g. Publicación del manuscrito “Structural, functional, thermal and rheological properties of nixtamalised and extruded blue maize (*Zea mays L.*) flour with different calcium sources”

OBJETIVO 2: Extraer componentes funcionales de maíz y del pericarpio para su aplicación como ingredientes en la formulación de alimentos.

- a. Cotización de materiales y reactivos para realizar las actividades necesarias para el cumplimiento este objetivo

OBJETIVO 3: Encapsulación y aplicación de pigmentos de tuna roja para el desarrollo de yogurt con colores naturales y con propiedades antioxidantes.

- a. Cotización de materiales y reactivos para realizar las actividades necesarias para el cumplimiento este objetivo
- b. Caracterización fisicoquímica de la tuna roja
- c. Caracterización física (densidad, color, índice de solubilidad, actividad de agua), química (contenido de polifenoles totales, betalainas, flavonoides y actividad antioxidante) y morfológica (microscopía) de encapsulados de tuna roja obtenidos mediante secado por aspersión
- d. Análisis datos experimentales obtenidos a partir de la caracterización física, química y morfológica de los encapsulados de tuna roja obtenidos mediante secado por aspersión
- e. Elaboración de manuscrito para publicación en revista arbitrada indizada
- f. Envío del manuscrito para publicación en el Journal of Food Science and Biotechnology

OBJETIVO 4. Evaluar subproductos de la nuez y de maíz para la separación de metales tóxicos contenidos en soluciones control con aplicaciones en efluentes contaminados.

- a. Cotización de materiales y reactivos para realizar las actividades necesarias para el cumplimiento este objetivo
- b. Escritura de anteproyecto de tesis “Remoción de cromo hexavalente en soluciones acuosas por el proceso de adsorción utilizando el ruezno (pericarpio y mesocarpio) de la nuez como material adsorbente”

8. Descripción de las metas alcanzadas durante el ejercicio del proyecto:

- a. Avances en los objetivos planteados

9. Productos:

(Listar y anexar los productos resultado de las acciones realizadas en el proyecto, ejemplo: libros, revistas, publicaciones, memorias, etc.)

- a. Publicación de 1 artículo en revista arbitrada e indizada “Structural, functional, thermal and rheological properties of nixtamalised and extruded blue maize (*Zea mays* L.) flour with different calcium sources”, DOI: 10.1111/ijfs.12340
- b. Envío del manuscrito “Effect of Soluble Fiber on the Physicochemical Properties of Cactus Pear (*Opuntia ficus indica*) Encapsulated by Spray Drying” para revisión en el Journal of Food Science and Biotechnology
- c. Presentación en congreso: Conferencia “Aplicación de Ultrasonido en la Nixtamalización”, Inst. Tecnológico de Cuauhtémoc. Cuauhtémoc, Chih., 26 de Septiembre de 2013 - 26 de Septiembre de 2013
- d. Presentación en congreso: “Effect of different calcium sources on the bioactive compounds stability of extruded and nixtamalized blue maize flour”, 2013 EFFoST Annual Meeting. Bologna, Italia, 15 de Noviembre de 2013.

10. Impacto académico: (El obtenido con la implementación del proyecto)

- a. Mejora en la producción académica-científica
- b. Mejora en el trabajo en equipo en desarrollo experimental
- c. Fortalecimiento en actividades grupales, como CA

11. Actividades de apoyo complementarias:

(Identificar, de ser el caso, actividades de apoyo no contempladas originalmente en el proyecto).

- a. Cotización de materiales y reactivos necesarios para el desarrollo experimental en el cumplimiento de los objetivos planteados

- b. Entrenamiento en el uso y manejo de equipo específico de las diferentes tecnologías aplicadas, así como en el montaje técnicas para los diferentes análisis de las muestras.

12. Comentarios adicionales:

Los productos generados hasta la fecha no incluyen la leyenda *“Proyecto realizado con financiamiento de la Secretaría de Educación Pública-Subsecretaría de Educación Superior-Dirección General de Educación Superior Universitaria”* debido a que dichos productos se obtuvieron antes de recibir la información sobre los requisitos para presentación de productos.

PRODUCTOS ACADÉMICOS

PUBLICACIÓN DE ARTÍCULOS
ARBITRADOS INDEXADOS

Original article

Structural, functional, thermal and rheological properties of nixtamalised and extruded blue maize (*Zea mays* L.) flour with different calcium sources

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Summary The effects of different concentrations of calcium hydroxide [Ca(OH)₂] and calcium lactate [C₆H₁₀O₆Ca] on the functional and physical properties of extruded (EF) and nixtamalised (NF) blue maize flours were evaluated. Calcium source and concentration showed no significant effects on the EF expansion index. The water absorption index (WAI) of EF decreased as the concentrations of both calcium sources increased, and NF with C₆H₁₀O₆Ca had the lowest WAI. The thermal and pasting properties of NFs were higher than those of EF. NF with C₆H₁₀O₆Ca showed the highest final viscosity (FinV), indicating less damage to the starch granules, and this was correlated with microscopic analysis. In contrast, the FinV of EFs was significantly affected by calcium source and concentration. Extrusion with 0.3% and nixtamalisation at 2.95% of C₆H₁₀O₆Ca yielded high WAI value and the best rheological properties in maize flour, respectively. These results suggest the use of C₆H₁₀O₆Ca in extrusion or nixtamalisation to produce blue maize flours for tortilla or snacks with antioxidants.

Keywords Blue maize, calcium hydroxide, calcium lactate, extrusion, nixtamalisation, rheological properties.

Introduction

Pigmented maize has received increased attention in the last decade because of the consumer demand for healthy nixtamalised corn products such as tortillas and tacos. Anthocyanins give colour to the grains and are found mainly in the pericarp and/or the aleurone layer (Salinas-Moreno *et al.*, 1999). They have nutraceutical properties with possible health benefits (Lee *et al.*, 2005). During nixtamalisation, the maize is cooked and steeped in calcium hydroxide [Ca(OH)₂] solution to obtain nixtamal. The pericarp is softened and removed, which allows calcium and water to diffuse into the grain, causing physicochemical changes (Steffe & Singh, 1980) in starch, protein and germ; these changes affect the organoleptic properties of

masa (dough), flour, tortillas and other nixtamalised maize products. Blue maize nixtamalisation has the drawback that with the removal of pericarp, anthocyanin and polyphenolic derivat compounds are also lost by leaching and instability at high pH values (Pflugfelder *et al.*, 1988; Cortés-Gómez *et al.*, 2006), resulting in the varying colours of nixtamalised products. On the other hand, the extrusion cooking process has been used to manufacture instant maize flour (Mensah-Agyapong & Horner, 1992; Gómez Aldapa *et al.*, 1996). During this process, the Ca(OH)₂ addition is necessary to provoke the physicochemical changes in the starch to obtain flour, masa and tortillas with similar characteristics to nixtamalised maize flour, besides that lime represents a calcium source to improve the nutritional products value.

The effects of lime during nixtamalisation and extrusion on the thermal, rheological and structural

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characteristics of blue maize flour have been reported (Cortés-Gómez *et al.*, 2005; Del Pozo-Insfran *et al.*, 2007; Aguayo-Rojas *et al.*, 2012). However, the use of other calcium sources that generate less alkaline solutions for processing blue maize has not reported. Previous studies have shown that less alkaline conditions decrease the average molecular weight of starch, without affecting the structural characteristics of the product (Kervinen *et al.*, 1984; Pan *et al.*, 1998). The structural characteristics of maize products have been related to calcium–starch interaction during gelatinisation studies and processing (Tovar *et al.*, 2004; Cortés-Gómez *et al.*, 2005). Robles *et al.* (1988) and Méndez *et al.* (2006) found that the calcium–starch interaction after nixtamalisation causes an increase in the gelatinisation temperature and enthalpy of starch. The changes that are produced in the starch granules may be evaluated through rheological, functional and textural properties of the final products (Robles *et al.*, 1988; Mondragón *et al.*, 2004; Méndez *et al.*, 2006). The effect of alkalinity and calcium concentration on the physicochemical and functional properties on the extruded and nixtamalised flour has been evaluated in previous studies (Zazueta-Morales *et al.*, 2002; Mora-Rochín *et al.*, 2010; Aguayo-Rojas *et al.*, 2012). Although $\text{Ca}(\text{OH})_2$ has been used traditionally, other combined or individual compounds, with or without calcium, have been studied in nixtamalisation (Robles *et al.*, 1988; Maya *et al.*, 2010). Ruiz-Gutiérrez *et al.* (2011) reported that white maize nixtamalisation with calcium lactate [$\text{C}_6\text{H}_{10}\text{O}_6\text{Ca}$] resulted in tortillas with good colour and texture. The use of $\text{C}_6\text{H}_{10}\text{O}_6\text{Ca}$ for nixtamalisation or extrusion in blue maize has not been reported, and it can represent an advantage because it produces near-neutral pH values, which would minimise the degradation of antioxidants (Brouillard, 1982; Rein, 2005) and less damage in the starch improving the physical and chemical properties of masa, tortilla and related products. The novel contribution of this investigation allows the researcher to evaluate the effect of the calcium lactate on the physical properties on the blue maize flour that had not researched before. The aim of this study was to evaluate the effect of $\text{Ca}(\text{OH})_2$ and $\text{C}_6\text{H}_{10}\text{O}_6\text{Ca}$ concentrations on the functional and physical properties of extruded and nixtamalised blue maize flours.

Materials and methods

Materials

Blue maize (*Zea mays* L.) from Babicora, Chihuahua, Mexico, was used. Calcium hydroxide [$\text{Ca}(\text{OH})_2$] and calcium lactate [$\text{C}_6\text{H}_{10}\text{O}_6\text{Ca}$] were used as the calcium sources. Proximal analysis was performed for maize characterisation. Test weight (Method 14-40, AACC,

2000) and flotation index (Salinas *et al.*, 1992) were also determined.

Extrusion process

Blue maize kernel was milled in a hammer mill (Pulvex model 200, Mexico) with a 5-mm sieve to obtain 2- to 3-mm grits and fine powder. Lots (850 g) of milled blue maize were mixed separately with different concentrations of $\text{Ca}(\text{OH})_2$ (0.1%, 0.2% and 0.3%) and $\text{C}_6\text{H}_{10}\text{O}_6\text{Ca}$ (0.3%, 0.6% and 0.9%). Then, each lot was moisturised with water to reach a moisture content of 30%. A lot without added calcium was prepared similarly to serve as a control (EF control). Each lot was packed in a polyethylene bag, stored for 14 h at 4 °C and then extruded in a single-screw extruder (CINVESTAV-IPN; Querétaro, Mexico). The extruder had a barrel (length, 428 mm; diameter, 25 mm) with three independent electrically heated zones. Screw compression ratio was 1:1, die diameter was 4 mm, and the system was operated under a 30-Hz speed screw and a constant feed rate (30 g min^{-1}), which was kept constant. Processing temperatures in the different zones (feed, compression and die) were kept constant at 50 °C, 70 °C and 80 °C, respectively. During each experimental run, the extruder was operated at a steady state for each condition set. This condition was monitored and controlled with constant amperage. The extrudate samples were collected and dried at 45 °C for 48 h in forced-air convection oven until a moisture content of 0.09 to 0.11 $\text{g kg}^{-1} \text{ dm}$ (dry matter) was achieved. These extrudates were ground, sieved (0.8 mm) and packed in hermetically sealed plastic bags and stored in the dark at 4 °C until analysis.

Nixtamalisation process

One-kilogram lots of blue maize were nixtamalised using two different calcium solutions. One lot was cooked in a solution of 1% $\text{Ca}(\text{OH})_2$, and the other was cooked in a solution of 2.95% $\text{C}_6\text{H}_{10}\text{O}_6\text{Ca}$ in a 1:3 maize/solution ratio at 90 °C for 40 min, followed by 15 h of steeping. The cooking liquor (nejayote) was discarded, and the cooked grain (nixtamal) was washed twice with water to remove excess $\text{Ca}(\text{OH})_2$ or $\text{C}_6\text{H}_{10}\text{O}_6\text{Ca}$. The nixtamal was ground in a stone mill (FUMASA, Mexico) and dried using a flash dryer, with an input temperature of 295 °C and an output temperature of 90 °C. The dehydrated flours were ground, sieved (0.8 mm) and packed in hermetically sealed plastic bags and stored in the dark at 4 °C until analysis.

Analytical methods

Moisture, total protein, total fat, fibre and ash contents of the maize were analysed according to AOAC

(1998) methods 950.02, 960.52, 920.39, 962.09 and 923.03, respectively.

pH

The pH value was determined by the standard AACC (2000) method using a pH meter (Corning Pinnacle, Corning, Inc., New York) calibrated with standard buffers. The analysis was carried out in triplicate.

Functional properties of blue maize flours

Expansion index

The expansion index (EI) was calculated for 20 samples by dividing the average diameter of the extruded products by the internal diameter of the extruder die nozzle (Baladrán-Quintana *et al.*, 1998; Zazueta-Morales *et al.*, 2002).

Bulk density

Bulk density (BD) was determined by pouring the ground extrudate (40/60 mesh) into a cylindrical container, scraping off excess extrudate and dividing the net weight of the powder by the volume of the container (Moreyra & Peleg, 1981). Bulk density was expressed in kilograms per litre (kg L^{-1}). The analysis was done in triplicate, and mean values were reported.

Water absorption and water solubility indexes

The water absorption index (WAI) and water solubility index (WSI) were determined in triplicate following the procedures described by Anderson *et al.* (1969) and were expressed as percentage. The methods measure the quantity of water incorporated in the flour and also the soluble solids dissolved in water at 30 °C. Samples were weighed (2.5 g) into plastic tubes and mixed with 30 mL of distilled water. The samples were manually shaken, the slurries were centrifuged for 10 min at 3200 g (Thermo IEC model CL3-R, USA), and the supernatant was decanted into preweighed porcelain capsules. Capsules were dried for 24 h at 105 °C and weighed. The gel remaining in the tubes after decanting the supernatant was weighed. The ratio between gel-forming solids and soluble solids was measured as grams of water per gram of flour.

Physical properties of blue maize flours

Thermal properties of blue maize extruded flours

The gelatinisation temperature and enthalpy of starch gelatinisation were determined by differential scanning calorimetry following the method described by Ruiz-Gutiérrez *et al.* (2011). Two milligrams of flour was placed in a pan with 20 μL of distilled water. The pan was sealed hermetically; an empty pan was used as a reference. The calorimeter from TA Instruments (Q-200, Crawley, UK), with a program of 30 °C to 110 °C and

a temperature ramp of 5 °C min^{-1} , was used. The thermograms obtained were analysed with Universal Analysis software (TA Instruments, Crawley, UK). The starch gelatinisation onset temperature (T_o), peak temperature (T_p) and end temperature (T_e) were obtained from the thermograms. The gelatinisation enthalpy (ΔH_g) was obtained by integrating the area under the curve. Each determination was done in duplicate.

Pasting properties of the extruded flours

The apparent viscosity of the flours was measured according to the method described by Zazueta-Morales *et al.* (2002) using a Rapid Visco Analyser (RVA SUPER 4, Newport Scientific, Sydney, Australia). Flour sample suspensions were prepared by weighing 4 g of extrudates milled and dried (50 ± 2 °C, 12 h) with 47.5 to 8.5% moisture content into an RVA canister and individually adjusting each sample to the total weight of 28 g, using distilled water. The rotating paddles were held at 50 °C for 2 min to stabilise the temperature and ensure uniform dispersion, then heated to 92 °C at 5.6 °C min^{-1} , which was held constant for 5 min, then cooled to 50 °C at the same rate and finally held at 50 °C for 1 min. The viscosity at 92 °C (V92), minimum viscosity (MinV or lowest viscosity at the end of heating constant period at 92 °C) and final viscosity (FinV or maximum viscosity attained during cooling to or holding at 50 °C) were recorded. The total setback viscosity (final minus minimum viscosity) was calculated from these parameter values. The viscosity with RVA was obtained in RVU units (1 RVU = 10 centipoises). Two determinations of each treatment were performed.

Scanning electron microscopy

Flour samples with particle sizes <0.15 mm and moisture content of 1% were stuck to stubs and coated with a gold layer in a high vacuum using a Denton vacuum evaporator (Desk II) set to a pressure of 7.031×10^{-2} kg cm^{-2} . The samples were examined using a scanning electron microscope (JSM-5800LV, JEOL, Akishima, Japan) equipped with a secondary electron detector, at an acceleration rate of 10 kV.

Statistical analysis

The results were analysed by nested ANOVA, where factor A was the calcium source [Ca(OH)_2 and $\text{C}_6\text{H}_{10}\text{O}_6\text{Ca}$] and factor B that was nested in A was the concentration of each calcium source [0.1%, 0.2% and 0.3% for Ca(OH)_2 and 0.3%, 0.6% and 0.9% for $\text{C}_6\text{H}_{10}\text{O}_6\text{Ca}$]. In addition, a contrast analysis of the mean differences between the results for EF and NF was performed. Significance was defined as $P < 0.05$ using SAS software, version 9.2 (2007) (SAS Institute, Inc., Cary, NC).

Results and discussion

Blue maize characterisation

The proximate composition of blue maize is showed in Table 1. The pH, test weight and the flotation index were 5.87, 73.33 ± 2.72 kg hL⁻¹ and 94.66 ± 0.57 kg hL⁻¹, respectively, which were higher than those of other genotypes such as the white and yellow hybrids (Gutiérrez-Urbe *et al.*, 2010). These properties are related to endosperm texture. Betran *et al.* (2000) indicated that most of blue maize genotypes possess a floury endosperm that requires less cooking time.

Expansion index

The EI of the extruded blue maize (Table 2) was not affected ($P > 0.05$) by the calcium source or concentration. However, the EI of the EF prepared without a calcium source was greater and significantly different from that of the extruded prepared with either calcium source; thus, a lower calcium concentration resulted in a higher EI. The EI values for extruded flours indicate to some extent the starch damage caused by the extrusion process, which could result in sticky masa and tortilla with certain hardness degree, but with

Table 1 Proximate composition of blue maize

Component	Content* (%)
Moisture	9.60 ± 0.02
Protein	7.09 ± 0.09
Fat	4.44 ± 0.12
Fibre	2.26 ± 0.03
Ash	1.38 ± 0.05
Carbohydrate	75.23

*Mean ± SD. Carbohydrate was calculated by difference.

Flours	[Conc] (%)	EI	BD (kg L ⁻¹)	WAI	WSI
EF Ca(OH) ₂	0.1	0.95 ± 0.008 ^b	0.53 ± 0.02 ^c	2.61 ± 0.001 ^a	0.060 ± 0.004 ^b
	0.2	0.95 ± 0.001 ^b	0.54 ± 0.006 ^{bc}	2.58 ± 0.008 ^{bc}	0.057 ± 0.003 ^{bc}
	0.3	0.95 ± 0.005 ^b	0.57 ± 0.012 ^a	2.54 ± 0.002 ^d	0.055 ± 0.003 ^{bc}
EF (C ₆ H ₁₀ O ₆)Ca	0.3	0.95 ± 0.001 ^b	0.56 ± 0.001 ^{ab}	2.59 ± 0.003 ^b	0.055 ± 0.001 ^{bc}
	0.6	0.95 ± 0.001 ^b	0.57 ± 0.002 ^a	2.57 ± 0.003 ^c	0.053 ± 0.001 ^c
	0.9	0.95 ± 0.002 ^b	0.58 ± 0.002 ^a	2.51 ± 0.006 ^e	0.051 ± 0.001 ^c
EF control	0.0	0.98 ± 0.008 ^a	0.52 ± 0.007 ^c	2.36 ± 0.001 ^f	0.070 ± 0.001 ^a
NF Ca(OH) ₂	1.0	–	0.56 ± 0.013 ^{ab}	2.31 ± 0.016 ^g	0.069 ± 0.001 ^a
NF (C ₆ H ₁₀ O ₆)Ca	2.95	–	0.58 ± 0.014 ^a	2.16 ± 0.002 ^h	0.055 ± 0.001 ^{bc}

EF, extruded flours; NF, nixtamalised flours; EI, expansion index; BD, bulk density; WAI, water absorption index; WSI, water solubility index.

*Mean ± SD. Means by column with different letters show significant difference, contrast test ($P < 0.05$).

application perhaps for the elaboration of maize snacks. Zazueta-Morales *et al.* (2002) and Martínez-Bustos *et al.* (1998) reported similar EI results for maize extrudates. The decrease in the EI can be attributed to the damage in the starch molecules caused by Ca(OH)₂. Kervinen *et al.* (1984) reported that both acid and alkaline treatments during the extrusion process significantly decreased the molecular weight of the starch, decreasing its expansion. The starch–calcium complexes restrict the extent of the flash-off of internal moisture after the extrudate emerges from the die (Zazueta-Morales *et al.*, 2002) because the calcium ions electrostatically bind to the OH⁻ groups of the starch, making the molecule more compact (Camire & Clydesdale, 1981).

Bulk density

Despite that the BD values are similar, some significant differences can be seen (Table 2). Contrast analysis revealed that the BD for EF was slightly increased as calcium hydroxide concentration increases. NF with both calcium sources showed similar BD values to the EF with added calcium, being different to the EF control, which showed the lowest BD value. Thus, higher concentration of Ca(OH)₂ induced an increase in the BD values. This effect can be related to the EI obtained, where a high EI results in a low BD of the EF prepared without calcium source. These results are similar to those reported by Martínez-Bustos *et al.* (1998) and Zazueta-Morales *et al.* (2001) for maize instant flours. Most, BD values for EF match the values obtained for NF, suggesting that EF could be used for tortillas (slightly harder) as well as for maize snacks production.

Water absorption index

The WAI of the blue maize EF (Table 2) was affected ($P < 0.05$) by both calcium sources and their

Table 2 Functional and physical properties* of blue maize flours

concentrations. The WAI decreased as the calcium concentration increased ($P < 0.05$). The highest WAI value for EF with calcium lactate was obtained for 0.3%, whereas the EF control exhibited the lowest WAI compared with EF calcium added. Similar results have been reported by Zazueta-Morales *et al.* (2002) for maize extrudates. The NFs prepared using either calcium source displayed lower WAI, and the NF with $C_6H_{10}O_6Ca$ exhibited the lowest WAI. The highest values of WAI in the EF can be attributed to starch damage, caused by the extrusion cooking, which could lead to cohesive and sticky masa, yielding tortillas slightly hard (Mensah-Agyapong & Horner (1992), but with probable applications for maize snacks.

Water solubility index

The WSI of blue maize extruded flours for both calcium sources (Table 2) was not significantly affected for the different concentrations. However, both flours were different ($P < 0.05$) from the EF control. The WSI decreased slightly when the calcium concentration of either calcium source was increased. The same observation was reported by Arámbula *et al.* (1998) for extruded blue maize. This effect could be attributed to the added $Ca(OH)_2$ -forming complexes with the starch, causing starch swelling and therefore decreasing the WSI.

Thermal properties

Table 3 shows that the starch gelatinisation temperatures (T_o , T_p and T_e) and gelatinisation enthalpy (ΔH_g) values of the EFs prepared with both calcium sources at different calcium concentrations were not different ($P > 0.05$). This result is most likely due to the strong effects of the extrusion process on the starch molecules, resulting in lower gelatinisation enthalpy values. Similar findings were reported by Mondragón *et al.* (2004) for nixtamalisation with different $Ca(OH)_2$ concentrations; they found no significant effects on the thermal properties of the flours.

The gelatinisation enthalpy values of the NFs were significantly different from those of the EFs, which had the lowest values. These low gelatinisation enthalpy values indicate that there is a large amount of gelatinised starch in the EFs; therefore, less energy is required to gelatinise the rest of the starch (Mondragón *et al.*, 2004). The differences in gelatinisation enthalpy between EFs and NFs are shown in Figure 1.

Pasting properties

Table 3 shows the pasting properties of the flours. Both calcium sources at different concentrations did not affect ($P > 0.05$) the minimum viscosity (MinV)

Table 3 Thermal properties and viscosity* of blue maize flours

Flours	[Conc] (%)	T_o (°C)	T_p (°C)	T_e (°C)	ΔH_g (J g ⁻¹)	MinV (Cp)	FinV (Cp)	Setback viscosity (Cp)	pH
EF $Ca(OH)_2$	0.1	65.0 ± 0.1 ^{ab}	70.3 ± 0.1 ^b	75.7 ± 0.2 ^{bc}	4.4 ± 0.2 ^c	19.8 ± 1.1 ^{ab}	972.0 ± 1.41 ^g	1115.8 ± 10.6 ^c	6.6 ± 0.02 ^c
	0.2	65.3 ± 0.1 ^{ab}	70.3 ± 0.3 ^b	75.4 ± 0.1 ^c	3.9 ± 0.2 ^c	19.5 ± 0.7 ^{ab}	991.0 ± 5.65 ^f	1253.0 ± 19.1 ^c	6.8 ± 0.02 ^b
	0.3	65.3 ± 0.3 ^a	70.4 ± 0.1 ^b	75.6 ± 0.7 ^c	4.5 ± 0.2 ^c	18.0 ± 0.7 ^b	1110.5 ± 6.36 ^e	1321.8 ± 14.1 ^c	7.0 ± 0.01 ^a
EF ($C_6H_{10}O_6$)Ca	0.3	64.9 ± 0.2 ^{ab}	70.5 ± 0.1 ^b	76.2 ± 0.1 ^b	4.3 ± 0.1 ^c	18.0 ± 1.4 ^b	941.5 ± 3.35 ^g	667.5 ± 12.0 ^d	6.0 ± 0.03 ^f
	0.6	65.4 ± 0.1 ^a	70.6 ± 0.0 ^{ab}	76.1 ± 0.2 ^{bc}	4.4 ± 0.5 ^c	21.0 ± 1.4 ^a	900.0 ± 1.41 ^h	628.5 ± 7.8 ^d	5.8 ± 0.01 ^g
	0.9	65.4 ± 0.3 ^a	70.6 ± 0.4 ^{ab}	76.0 ± 0.2 ^{bc}	4.0 ± 0.01 ^c	18.8 ± 0.4 ^{ab}	883.0 ± 6.36 ^h	601.3 ± 17.0 ^d	5.7 ± 0.02 ^h
EF Control	0.0	64.6 ± 0.3 ^b	71.0 ± 0.2 ^a	76.3 ± 0.2 ^{ab}	4.2 ± 0.3 ^c	21.0 ± 2.8 ^a	1253.8 ± 30.75 ^d	1173.3 ± 18.0 ^c	6.2 ± 0.04 ^d
NF $Ca(OH)_2$	1.0	64.5 ± 0.4 ^b	70.6 ± 0.4 ^{ab}	77.0 ± 0.2 ^a	5.9 ± 0.5 ^b	11.5 ± 0.7 ^c	3193.5 ± 36.06 ^c	2500.5 ± 62.9 ^b	6.8 ± 0.01 ^b
NF ($C_6H_{10}O_6$)Ca	2.95	64.8 ± 0.4 ^b	71.0 ± 0.2 ^a	77.0 ± 0.6 ^a	5.9 ± 0.2 ^b	13.5 ± 0.7 ^c	4022.0 ± 19.80 ^b	2543.5 ± 81.3 ^{ab}	6.1 ± 0.01 ^e
Blue maize	0.0	62.9 ± 0.1 ^c	69.3 ± 0.6 ^c	75.9 ± 0.01 ^{bc}	7.0 ± 0.3 ^a	12.5 ± 0.7 ^c	4461.5 ± 10.60 ^a	2731.5 ± 10.6 ^a	5.9 ± 0.04 ^a

EF, extruded flours; NF, nixtamalised flours; T_o , onset temperature; T_p , peak temperature; T_e , end temperature; ΔH_g , gelatinisation enthalpy; MinV, minimum viscosity; FinV, final viscosity.

*Means values ± SD. Means by column with different letters show significant difference, contrast test ($P < 0.05$).

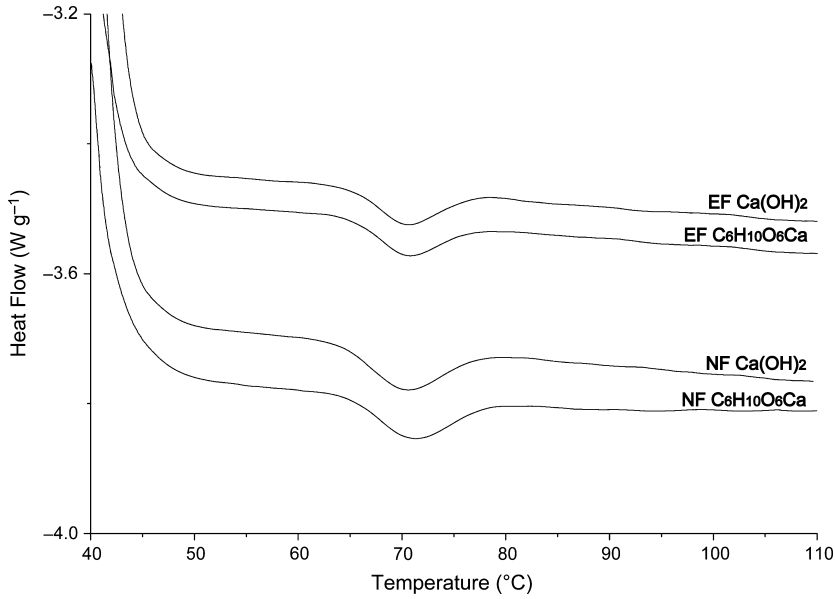


Figure 1 Differential scanning calorimetry (DSC) thermograms of EFs and NFs.

for EF. The EF exhibited MinV values similar to that of the control and different ($P < 0.05$) from that of both NFs. Moreover, the MinV values of the two NFs were not significantly different and showed the lowest MinV values, which were similar to that obtained for raw blue maize.

The final viscosity (FinV) is inversely related to the degree of starch gelatinisation. Therefore, processed flour that contains gelatinised starch does not develop an increased viscosity, whereas unprocessed flour that contains mainly native starches develops viscosity to its maximum capacity. In this study, the FinV (Table 3) was affected ($P < 0.05$) by both calcium sources and their concentrations. The FinV values of

EF increased as Ca(OH)_2 concentration increased, whereas increasing the $\text{C}_6\text{H}_{10}\text{O}_6\text{Ca}$ concentration caused the FinV values to decrease (Fig. 2). Similar results were reported by Mensah-Agyapong & Horner (1992) and Zazueta-Morales *et al.* (2002) for maize extrusion with Ca(OH)_2 . This phenomenon was attributed to the formation of complexes of the starch components with the calcium from the lime (Robles *et al.*, 1988; Islam & Azemi, 1994; San Martín-Martínez *et al.*, 2003). For the EF prepared with $\text{C}_6\text{H}_{10}\text{O}_6\text{Ca}$, the FinV values decreased with increasing calcium source concentration (Fig. 2), most likely due to the low ionisation of $\text{C}_6\text{H}_{10}\text{O}_6\text{Ca}$ compared with that of Ca(OH)_2 , which did not promote the release of Ca^{2+}

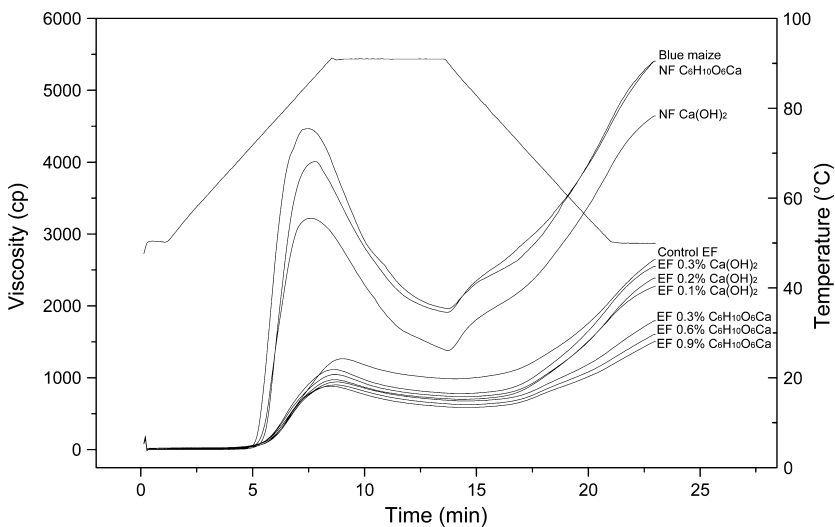


Figure 2 Amylographic profiles obtained with Rapid Visco Analyser of extruded, nixtamalised and control of blue maize flours with different calcium sources.

ions, limiting their interaction with the starch and consequently affecting viscosity. The control EF had a higher FinV (Fig. 2) than the other EFs, most likely due to the pH effect (Table 2) of the two calcium sources on the EFs. The FinV value of the NFs was higher than that of the EFs (Fig. 2). The NF prepared with $C_6H_{10}O_6Ca$ had a higher FinV value than the NF prepared with $Ca(OH)_2$ because of less damaged starch granules. FinV value trends in the maximum peaks between nixtamalised and EF indicate differences in the extent of the damage to the starch (Fig. 2). It suggests that EF could produce cohesive and slightly sticky masa and tortillas with some degree of toughness. Similar finding was reported by Mensah-Agyapong & Horner (1992).

Scanning electron microscopy

The scanning electron micrographs revealed that the EF with $Ca(OH)_2$ and $C_6H_{10}O_6Ca$ at different

concentrations contained starch granules showing complete loss of structure (Fig. 3). The damage that occurred during the extrusion process was notable in the large numbers of flattened and sheared granules. Similar results have been reported for corn semolina (Mercier *et al.*, 1979), corn grits (Gómez & Aguilera, 1984) and corn meal extrudates (Martínez-Bustos *et al.*, 1998). These results are due to starch dextrinisation, which is the principal mechanism of starch fragmentation during the extrusion process (Gómez & Aguilera, 1984). The same type of starch damage was observed in the control EF (Fig. 3g). In contrast, the NFs exhibited less damaged starch granules with spherical and polygonal shapes (Figs 3h,i). These morphological results were confirmed by the obtained ΔH_g values (Table 3). The damage to the starch by extrusion cooking process can be related with the development of cohesive and slightly adhesive masa, which could be used to obtain tortillas with some degree of toughness, suitable for snacks.

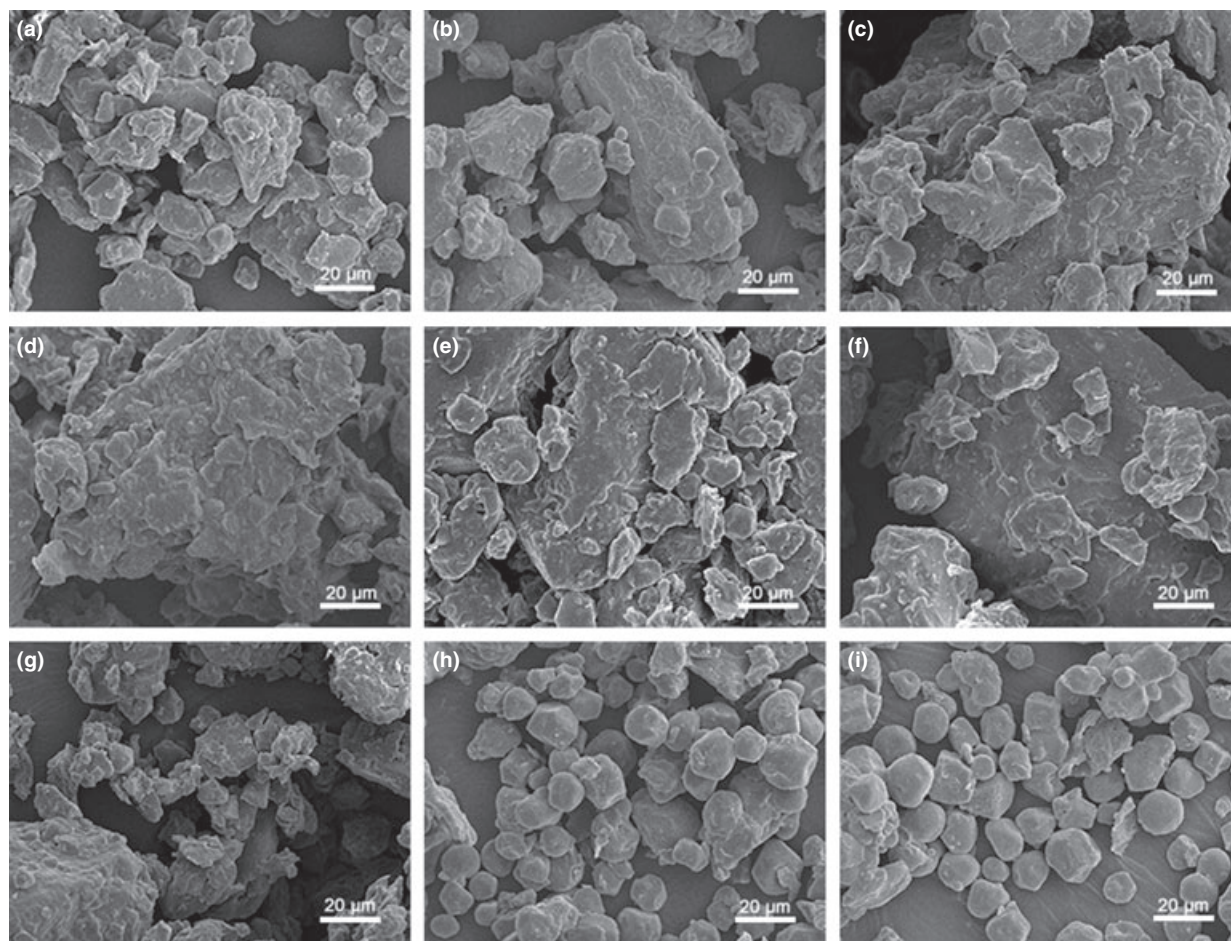


Figure 3 Micrographs of blue maize flours. (a) 0.1% $Ca(OH)_2$, (b) 0.2% $Ca(OH)_2$, (c) 0.3% $Ca(OH)_2$, (d) 0.3% $C_6H_{10}O_6Ca$, (e) 0.6% $C_6H_{10}O_6Ca$, (f) 0.9% $C_6H_{10}O_6Ca$, (g) EF control without calcium source, (h) NF with $Ca(OH)_2$, (i) NF with $C_6H_{10}O_6Ca$. a–i, 1000 \times .

Conclusions

The use of $C_6H_{10}O_6Ca$ as an alternative source to lime in extrusion and nixtamalisation of blue maize effectively improved the rheological properties of instant maize flours. The extrusion process severely damaged the starch compared with nixtamalisation, reducing the effects of calcium sources on the flour's physical and functional properties. The WAI and pasting properties of the extruded flours were influenced by the calcium sources and their concentrations. The pasting properties of NFs were higher than those of EFs, indicating the damaging effect of the extrusion process on the starch molecules, which was confirmed by the thermal and microscopic analyses. NF prepared with $C_6H_{10}O_6Ca$ had higher viscosity than NF prepared with $Ca(OH)_2$. Extrusion with 0.3% and nixtamalisation at 2.95% of $C_6H_{10}O_6Ca$ yielded high WAI value and the best rheological properties in blue maize flour, respectively, indicating for rheological properties less damaged starch and high viscosity values. This could be related to the degree of cooking, cohesiveness of the dough and chewiness of the final tortilla product, suggesting that these flours could be used for the manufacture of snacks or tortillas, with adequate quality characteristics. Additional studies are needed to evaluate the application of the blue corn flours in the development of maize products, costs and environmental benefits.

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ARTÍCULOS ENVIADOS
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Article Type: Research Article

Keywords: Encapsulation, cactus pear, bioactive compounds, spray-drying, soluble fiber

1 **Effect of Soluble Fiber on the Physicochemical Properties of Cactus Pear**
2 **(*Opuntia ficus indica*) Encapsulated by Spray Drying**

3
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17

18 **Short title:** Encapsulation of cactus pear

19

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21 **Summary** The effects of red cactus pear encapsulation by spray drying using soluble fiber
22 were evaluated. Cactus pear juice was mixed with 15%, 22.5%, or 30% soluble fiber and
23 dried at 160, 180, or 200°C. The juice showed high levels of polyphenols, quercetin,
24 kaempferol, isoharmetin, betacyanins, betaxanthins, and antioxidant activity. Increased
25 soluble fiber significantly increased pH, a_w , water solubility index, L^* , and b^* . Polyphenols
26 and betacyanins decrease significantly as soluble fiber increased and drying temperature
27 increased, respectively. Encapsulation at 160°C and 22.5% soluble fiber yielded good
28 physical properties and a higher content of bioactive compounds. Microscopy analyses
29 showed capsules with a spherical shape. These forms of capsules were affected by the
30 soluble fiber concentration added, found more collapsed capsules when less soluble fiber
31 was added. The soluble fiber utilized to encapsulate cactus pears resulted in a powder with
32 good properties that could be used as an ingredient in the food industry.

33

34 **Keywords:** Encapsulation, cactus pear, bioactive compounds, spray-drying, soluble fiber

35

36 **Introduction**

37 The consumer preference for tasty and healthy food products has increased in recent years
38 (1), a trend that has promoted the development of functional food. In Mexico, there are
39 numerous native fruits that could be considered as functional foods. Among these is the
40 cactus pear, which belongs to the Cactaceae family and has a diversity of forms and colors.
41 Recent studies have shown that the cactus pear contains components that have beneficial
42 health effects for consumers as the flavonoids quercetin, kaempferol, and isoharmetin in
43 yellow and red cactus pear cultivars (2), indicating that they can delay the effects of
44 oxidative damage by stabilizing free radicals (3). Further, other reports suggest that the

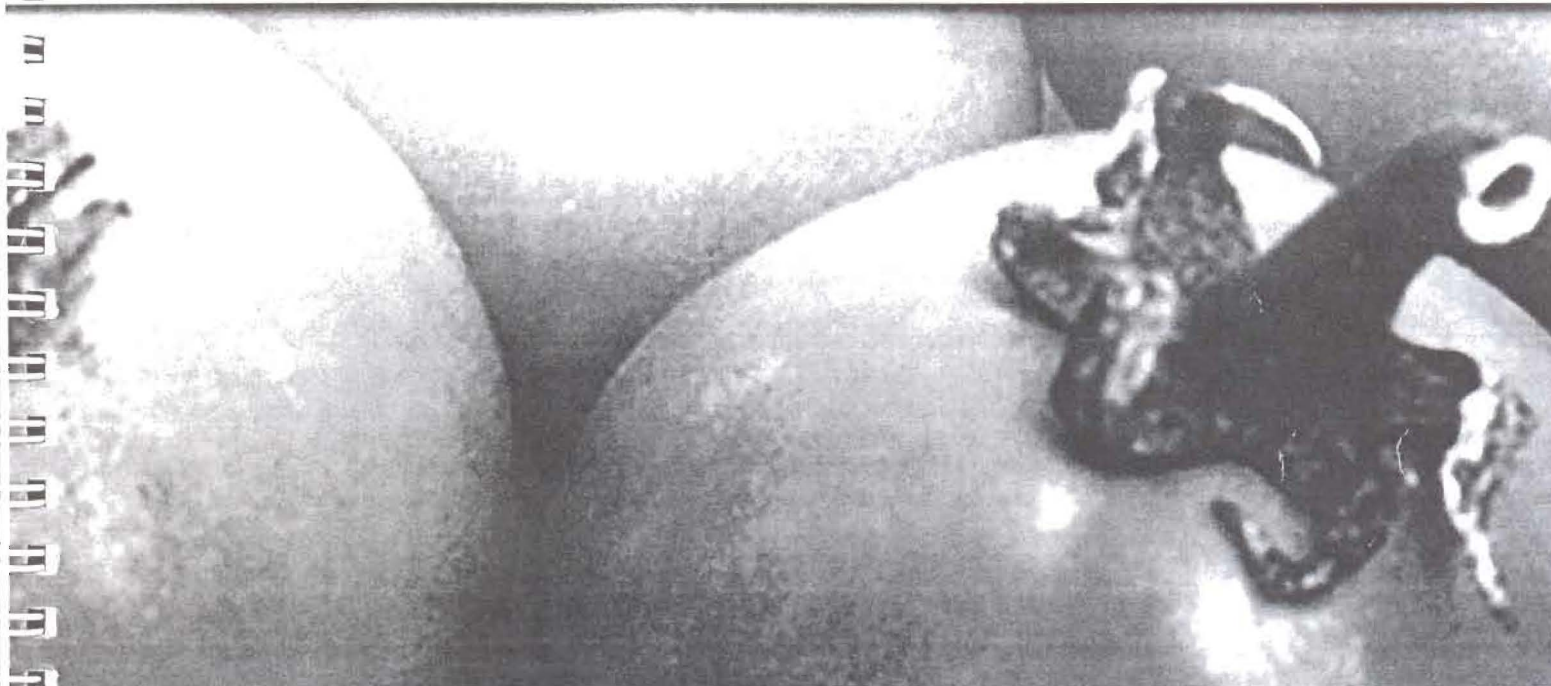
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	S. Zerizer*, Y. Necib, N. Bouthagane, Z. Kabouche, <i>University of Constantine 1, Algeria</i>
[P2.087]	The biodiversity of autochthon Sardinian food in longevity: proteomics, metabolomics and molecular biology research on the Sardinian's centenarians diet (acronym B. Al Akea) L. Deiana ^{*1} , S. Pasella ¹ , A. Baralla ¹ , S. Pinna ¹ , E. Canu ¹ , M. Deiana ² , G. D'hallewin ³ , B. Floris ¹ , A.F. Piana ¹ , C. Carru ¹ , ¹ <i>University of Sassari, Italy</i> , ² <i>Isola dei Centenari, Italy</i> , ³ <i>CNR-ISPA UOS, Italy</i>
[P2.088]	Prunus domestica fruit extract exhibit anti-oxidant activity in human umbilical endothelial cells G. D'hallewin ^{*1} , E. Canu ^{1,2} , S. Pinna ^{1,2} , A.M. Posadino ^{1,2} , G.F. Pintus ^{1,2} , M.G. Molino ¹ , E. Sotgiu ² , A. Baralla ^{1,2} , S. Pasella ^{1,2} , L. Deiana ^{1,2} , ¹ <i>CNR-ISPA UOS, Italy</i> , ² <i>University of Sassari, Italy</i>
[P2.089]	Investigation on Pb, Cd and Se values in the foods consumed by centenarians P. Occhineri ^{*1} , G. Forte ² , E. Muresu ¹ , B. Bocca ² , I. Mura ¹ , L. Murgia ¹ , V. Ventura ¹ , F. Pudda ¹ , F. Balzano ¹ , L. Deiana ¹ , ¹ <i>University of Sassari, Italy</i> , ² <i>Istituto Superiore Sanità, Italy</i>
[P2.090]	Identification of probiotic microorganisms in "pecorino sardo" cheese N. Mangia ^{*1} , F. Fancello ¹ , M. Deiana ² , A. Marongiu ¹ , P. Deiana ¹ , A. Mannu ¹ , A. Baralla ¹ , S. Pinna ¹ , A. Zinellu ¹ , L. Deiana ¹ , ¹ <i>University of Sassari, Italy</i> , ² <i>Isola dei Centenari, Italy</i>
[P2.091]	Seasonal variations of antimicrobial activity and chemical composition of Citrus limon L. Burm. spp. essential oils W. Randazzo ^{*1} , V. Guarrasi ² , A. Aleo ¹ , L. Settanni ¹ , M. Moschetti ² , C. Mammina ¹ , E. Palazzolo ¹ , M.A. Germanà ¹ , P.L. San Biagio ² , G. Moschetti ¹ , ¹ <i>Università di Palermo, Italy</i> , ² <i>Consiglio Nazionale delle Ricerche, Italy</i>
[P2.092]	Application of atmospheric gas plasma to fresh-cut kiwifruit slices: physico-chemical, metabolic and metabolomic study S. Tappi*, A. Berardinelli, L. Ragni, L. Laghi, U. Tylewicz, S. Romani, P. Rocculi, M. Dalla Rosa, <i>University of Bologna, Italy</i>
[P2.093]	Effect of different calcium sources on the bioactive compounds stability of extruded and nixtamalized blue maize flours M. Sánchez-Madrigal ¹ , A. Quintero-Ramos ^{*1} , F. Martínez-Bustos ² , C. Meléndez-Pizarro ¹ , M. Ruiz-Gutiérrez ¹ , A. Camacho-Dávila ¹ , P. Torres-Chávez ³ , B. Ramírez-Wong ³ , ¹ <i>Universidad Autónoma de Chihuahua, Mexico</i> , ² <i>Unidad Querétaro, Mexico</i> , ³ <i>Universidad de Sonora, Mexico</i>
[P2.94]	Effect of soya product and wheat fibre addition on some quality characteristics of pizza dough bas S. Romani*, V. Glicerina, F. Balestra, P. Rocculi, M. Dalla Rosa, <i>University of Bologna, Italy</i>
[P2.95]	Evaluation of anti-inflammatory effect of newbouldia laevis plant leaf and extract J.N. Okafor ^{*1} , T.M. Okonkwo ¹ , T.C. Ezeji ¹ , G.I. Okafor ¹ , R. Selvaraj ¹ , R. Shanmugasundaram ¹ , ¹ <i>Federal Institute of Industrial Research Oshodi, Nigeria</i> , ² <i>University of Nigeria, Nigeria</i> , ³ <i>Ohio State University & OARDC, USA</i>
[P2.096]	Application of infrared spectroscopy for dairy product authentication and quality assessment A. Gori, C. Cevoli, A. Fabbri, M.F. Caboni*, <i>Università di Bologna, Italy</i>
[P2.097]	Sargassum muticum and Osmundea pinnatifida food-grade extracts with antioxidant activity for novel functional foods D. Rodrigues ¹ , C. Silva ^{*2} , L. Pereira ³ , T. Rocha-Santos ^{1,4} , A.C. Freitas ^{1,4} , A.M. Gomes ² , A.C. Duarte ¹ , ¹ <i>University of Aveiro, Portugal</i> , ² <i>Universidade Católica Portuguesa, Portugal</i> , ³ <i>University of Coimbra, Portugal</i> , ⁴ <i>Instituto Piaget, Portugal</i>
[P2.098]	Biodegradable packaging effects on the shelf-life of fresh-cut apple and kiwifruit E. Cocci ¹ , C. Montanari ¹ , V. Siracusa ² , V. Glicerina ¹ , P. Rocculi ^{*1} , M. Dalla Rosa ¹ , ¹ <i>University of Bologna, Italy</i> , ² <i>University of Catania, Italy</i>
[P2.099]	A food additive for the metaphylaxis of infectious diseases in aquaculture M.A. Dinamarca*, C. Ibacache-Quiroga, G. Espinoza-Vergara, J. Ojeda, J.M. Troncoso, <i>Universidad de Valparaíso, Chile</i>
[P2.100]	Comparison of two methods, enzymatic and by polar solvents, for the extraction of avocado seed polyphenols S.J. Villanueva-Rodríguez*, L.A. Anguiano-Sevilla, D.H. López-Alvarez, G.C. Sandoval-Fabián, <i>Centro de Investigación y Asistencia en Tecnología y Diseño del Estado de Jalisco, .AC., Mexico</i>
[P2.101]	Tropical fruits as a source of bioactive compounds to preserve human health and promote healthier life V. Moo-Huchin ¹ , G. González-Aguilar ² , I. Estrada-Mota ¹ , L. Cuevas-Glory ³ , R. Estrada-León ¹ , E. Ortiz-Vázquez ³ , M. Vargas-Vargas ³ , E. Sauri-Duch ^{*3} , ¹ <i>Instituto Tecnológico Superior de Calkiní, Mexico</i> , ² <i>Centro de Investigación en Alimentación y Desarrollo, Mexico</i> , ³ <i>Instituto Tecnológico de Mérida, Mexico</i>
[P2.102]	Functional properties of carrot slices as affected by blanching in trehalose solutions and frozen storage C. Di Mattia, L. Neri, G. Sacchetti, D. Mastrocola, P. Pittia*, <i>University of Teramo, Italy</i>
[P2.103]	Extending quality and shelf-life of minimally processed apples by vacuum impregnation treatments