UNIVERSITAT POLITÈCNICA DE VALÈNCIA

Obtención de fibra alimentaria a partir del subproducto de la industria citrícola, a través de la aplicación de diferentes tecnologías de extracción

TESIS DOCTORAL

Presentada por:

Claudia Pérez Pirotto

Dirigida por:

Dra. Patricia Arcia Cabrera

Dra. Sonia Cozzano Ferreira

Dra. Isabel Hernando Hernando

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Dª. Patricia Arcia Cabrera, docente de alta dedicación de la Universidad Católica del Uruguay e investigadora de Latitud Fundación LATU, Dª. Sonia Cozzano Ferreira, decana de la Facultad de Ingeniería y Tecnologías de la Universidad Católica del Uruguay y Dª. Isabel Hernando Hernando, Catedrática de Universidad del Departamento de Tecnología de Alimentos de la Universitat Politècnica de València,

HACEN CONSTAR QUE:

El trabajo de investigación "Obtención de fibra alimentaria a partir del subproducto de la industria citrícola, a través de la aplicación de diferentes tecnologías de extracción" que presenta Dª. Claudia Pérez Pirotto por la Universidad Politécnica de Valencia, y que ha sido realizado bajo nuestra dirección en Latitud Fundación LATU, en la Universidad Católica del Uruguay y en el Grupo de Investigación de Microestructura y Química de Alimentos de la Universitat Politècnica de València, reúne las condiciones para optar al grado de Doctor.

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Fdo. Dra. Patricia Arcia Cabrera	Fdo. Dra. Sonia Cozzano Ferreira	Fdo. Dra. Isabel Hernando Hernando

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Resumen

La investigación de la presente tesis doctoral abarcó el desarrollo de un ingrediente a partir del subproducto de la industria citrícola. Se buscó obtener un ingrediente en polvo, con alto contenido de fibra soluble, que pudiera utilizarse en la formulación de otros productos. La investigación se enmarcó en una estrategia de aprovechamiento de subproductos de la agroindustria.

Para ello el trabajo se dividió en cuatro etapas. Tras la obtención del ingrediente por las diferentes metodologías, en las primeras dos etapas se analizaron su composición química y propiedades tecno funcionales. En la tercera etapa se realizó un ensayo de estabilidad del ingrediente tras su almacenamiento a diferentes actividades de agua. Finalmente, en la cuarta etapa se evaluó la incorporación del ingrediente, como fuente de fibra, en la formulación de un flan. Por un lado, se estudió el efecto de la información sobre el origen de la fibra agregada en la respuesta del consumidor y por otro, el efecto de la incorporación de dicha fibra sobre las propiedades sensoriales del producto.

Se ensayaron cuatro tecnologías no contaminantes (sin solventes) para la extracción de fibra soluble. Estas fueron agua caliente, extrusión + agua caliente, jet cooker y jet cooker + agua caliente. Después de los tratamientos, el sobrenadante de la extracción se secó en “spray dryer” para obtener un ingrediente en polvo. En la primera etapa, el contenido de fibra alimentaria de alto y bajo peso molecular fue analizado, junto con el contenido de azúcares simples (glucosa, fructosa y sacarosa) y de compuestos bioactivos. Los resultados mostraron que el contenido de fibra total varía entre 10 y 20 gramos por 100 gramos de producto entre los tratamientos ensayados, siendo el proceso de extrusión + agua caliente el que mayor contenido de fibra presentó. Además, este tratamiento fue el que presentó menor contenido de azúcares y mayor contenido de compuestos bioactivos. Estos últimos podrían ser liberados por el proceso de extrusión al romper las fibras y liberar los compuestos asociados a las mismas, así como también ser derivados de los procesos térmicos a los que fue sometida la muestra (compuestos formados en reacciones de Maillard o caramelización).

En la segunda etapa se analizaron la capacidad espumante y estabilidad de la espuma, retención de aceite, solubilidad en agua, higroscopicidad, microestructura, temperatura de transición vítrea, color y reología de las soluciones. Se observaron diferentes grados de aglomeración en los polvos, que se relacionaron con la baja temperatura de transición vítrea de los ingredientes, fruto de sus componentes de bajo peso molecular. Aunque todas las soluciones formaron espumas, la espuma del ingrediente obtenido por extrusión + agua caliente fue la más estable, lo que pudo deberse a su mayor viscosidad.

En la tercera etapa se almacenaron los ingredientes obtenidos a diferentes actividades de agua durante 15 semanas, y una vez transcurrido el tiempo se modeló su isoterma de sorción. Además, se estudió la transición vítrea, junto con el contenido de compuestos bioactivos, color y textura. Los polvos no fueron estables en su estado inicial, debido al alto contenido de humedad que presentaban. Se observó pardeamiento a medida que aumenta la actividad de agua. La dureza presentó un máximo en actividades de agua de alrededor de 0.5 (dependiendo del tratamiento 0.43 – 0.52) y después disminuyó. La única muestra para la cual se encontró condiciones de almacenamiento aceptables con su humedad de partida fue la resultante de extrusión + agua caliente, la cual debería almacenarse a temperaturas de refrigeración. En los otros casos sería necesario ajustar los parámetros del proceso para disminuir la humedad final del ingrediente.

En la última etapa se evaluó la incorporación del ingrediente en la formulación de un flan. Concretamente, se estudió el efecto de información sobre el ingrediente en la respuesta del consumidor y el efecto de su incorporación en las propiedades sensoriales del postre. Para evaluar el efecto de la información en la respuesta del consumidor, se realizó una encuesta en España y Uruguay tomando como variables de estudio la intención de compra, y las percepciones de saludable y amigable con el medio ambiente. Se evaluaron tres categorías de producto diferentes (un producto listo para consumo, un polvo para preparar el postre en casa y un flan casero), variando la información ofrecida (origen de la fibra y un logo haciendo referencia a la sostenibilidad). El comportamiento de ambas poblaciones fue diferente en las tres variables de estudio, aunque coincidieron en que el producto con mayor intención de compra fue el casero con logo de sostenible. La intención de compra no se correlacionó con la percepción de saludable y amigable con el medio ambiente en todos los casos, aunque estas dos últimas variables sí se mantuvieron correlacionadas.

Para evaluar la incorporación del ingrediente en la formulación de un flan "fuente de fibra" (3% de contenido de fibra), se ensayaron diferentes porcentajes de sustitución de inulina (muestra control) del postre por el ingrediente de naranja obtenido a partir de la extrusión + agua caliente. La sustitución de un 30% del contenido total de fibra por el nuevo ingrediente logró un producto similar al control (con contenido de fibra correspondiente 100% a inulina), sin descriptores de sabor no agradables y con una mejora en

el brillo y la textura. Para lograr porcentajes de sustitución superiores sería necesario trabajar en optimizar el proceso de obtención del ingrediente con el objetivo de eliminar o reducir los sabores a quemado, amargo y ácido, ampliando así su potencial de uso.

Resum

La investigació de la present tesi doctoral va abastar el desenvolupament d'un ingredient a partir del subproducte de la indústria citrícola. Es va buscar obtindre un ingredient en pols, amb alt contingut de fibra soluble, que poguera utilitzar-se en la formulació d'altres productes. La investigació es va emmarcar en una estratègia d'aprofitament de subproductes de l'agroindústria.

Per a això el treball es va dividir en quatre etapes. Després de l'obtenció de l'ingredient per les diferents metodologies, en les primeres dues etapes es van analitzar la seua composició química i propietats tecno funcionals. En la tercera etapa es va realitzar un assaig d'estabilitat de l'ingredient després del seu emmagatzematge a diferents activitats d'aigua. Finalment, en la quarta etapa es va avaluar la incorporació de l'ingredient, com a font de fibra, en la formulació d'un flam. D'una banda, es va estudiar l'efecte de la informació sobre l'origen de la fibra agregada en la resposta del consumidor i per un altre, l'efecte de la incorporació d'aquesta fibra sobre les propietats sensorials del producte.

Es van assajar quatre tecnologies no contaminants (sense solvents) per a l'extracció de fibra soluble. Aquestes van ser aigua calenta, extrusió + aigua calenta, jet cooker i jet cooker + aigua calenta. Després dels tractaments, el sobrenedant de l'extracció es va assecar en “spray dryer” per a obtindre un

ingredient en pols. En la primera etapa, el contingut de fibra alimentària d'alt i baix pes molecular va ser analitzat, juntament amb el contingut de sucres simples (glucosa, fructosa i sacarosa) i de compostos bioactius. Els resultats van mostrar que el contingut de fibra total varia entre 10 i 20 grams per 100 grams de producte entre els tractaments assajats, sent el procés d'extrusió + aigua calenta el que major contingut de fibra va presentar. A més, aquest tractament va ser el que va presentar menor contingut de sucres i major contingut de compostos bioactius. Aquests últims podrien ser alliberats pel procés d'extrusió en trencar les fibres i alliberar els compostos associats a aquestes, així com també ser derivats dels processos tèrmics als quals va ser sotmesa la mostra (compostos formats en reaccions de Maillard o caramel·lització).

En la segona etapa es van analitzar la capacitat espumant i estabilitat de l'espuma, retenció d'oli, solubilitat en aigua, higroscopicitat, microestructura, temperatura de transició vítria, color i reologia de les solucions. Es van observar diferents graus d'aglomeració en les pólvores, que es van relacionar amb la baixa temperatura de transició vítria dels ingredients, fruit dels seus components de baix pes molecular. Encara que totes les solucions van formar

espumes, l'espuma de l'ingredient obtingut per extrusió + aigua calenta va ser la més estable, la qual cosa va poder deure's a la seua major viscositat.

En la tercera etapa es van emmagatzemar els ingredients obtinguts a diferents activitats d'aigua durant 15 setmanes, i una vegada transcorregut el temps es va modelar la seua isoterma de sorció. A més, es va estudiar la transició vítria, juntament amb el contingut de compostos bioactius, color i textura. Les pólvores no van ser estables en el seu estat inicial, a causa de l'alt contingut d'humitat que presentaven. Es va observar pardejament a mesura que augmenta l'activitat d'aigua. La duresa va presentar un màxim en activitats d'aigua d'al voltant de 0.5 (depenent del tractament 0.43 – 0.52) i després va disminuir. L'única mostra per a la qual es va trobar condicions d'emmagatzematge acceptables amb la seua humitat de partida va ser la resultant d'extrusió + aigua calenta, la qual hauria d'emmagatzemar-se a temperatures de refrigeració. En els altres casos seria necessari ajustar els paràmetres del procés per a disminuir la humitat final de l'ingredient.

En l'última etapa es va avaluar la incorporació de l'ingredient en la formulació d'un flam. Concretament, es va estudiar l'efecte d'informació sobre l'ingredient en la resposta del consumidor i l'efecte de la seua incorporació en les propietats sensorials de les postres. Per a avaluar l'efecte de la informació en la resposta del consumidor, es va realitzar una enquesta a Espanya i l'Uruguai prenent com a variables d'estudi la intenció de compra, i la percepció de saludable i amigable amb el medi ambient. Es van avaluar tres categories de producte diferents (un producte llest per a consum, una pols per a preparar les postres a casa i un flam casolà), variant la informació oferida (origen de la fibra i un logo fent referència a la sostenibilitat). El comportament de totes dues poblacions va ser diferent en les tres variables d'estudi, encara que van coincidir que el producte amb major intenció de compra és el casolà amb logo de sostenible. La intenció de compra no es va correlacionar amb la percepció de saludable i amigable amb el medi ambient en tots els casos, encara que aquestes dues últimes variables sí que es van mantindre correlacionades.

Per a avaluar la incorporació de l'ingredient en la formulació d'un flam “font de fibra” (3% de contingut de fibra), es van assajar diferents percentatges de substitució d'inulina (mostra control) de les postres per l'ingredient de taronja obtingut a partir de l'extrusió + aigua calenta. La substitució d'un 30% del contingut total de fibra pel nou ingredient va aconseguir un producte similar al control (amb contingut de fibra corresponent 100% a inulina), sense descriptors de sabor no agradables i amb una millora en la lluentor i la textura.

Per a aconseguir percentatges de substitució superiors seria necessari treballar a optimitzar el procés d'obtenció de l'ingredient amb l'objectiu d'eliminar o reduir els sabors a cremat, amarg i àcid, ampliant així el seu potencial d'ús.

Abstract

This thesis covered the development of an ingredient from the by-product of the citrus industry. The aim was to obtain a powdered ingredient, with high soluble fiber content, that could be used in the formulation of other products. The research was framed in a strategy for the use of by-products of the agroindustry.

The work was divided into four stages. After obtaining the ingredient by the different methodologies, in the first two stages its chemical composition and techno-functional properties were analyzed. In the third stage, a stability test of the ingredient was carried out after storage at different water activities. Finally, in the fourth stage, the incorporation of the ingredient as a source of fiber, in the formulation of a flan was evaluated. On the one hand, the effect of information on the origin of the added fiber on consumer response was studied, and on the other, the effect of incorporating said fiber on the sensory properties of the product.

Four non-contaminant technologies (without use of solvents) for the extraction of soluble fiber were tested. These were hot water, extrusion + hot water, jet cooker, and jet cooker + hot water. After the treatments, the extraction supernatant was dried in a spray dryer to obtain a powdered ingredient. In the first stage, the content of high and low molecular weight dietary fiber and the content of simple sugars (glucose, fructose and sucrose) and bioactive compounds were analyzed. The results showed that the total fiber content varies between 10 and 20 grams per 100 grams of product among the tested treatments, being the extrusion + hot water process the one with the highest fiber content. In addition, this treatment was the one with the lowest sugar content and the highest content of bioactive compounds. The latter could be released by the extrusion process, by breaking the fibers and releasing the compounds associated with them, as well as being derived from the thermal processes to which the sample was subjected (compounds formed in Maillard reactions or caramelization).

In the second stage, the foaming capacity and stability, oil holding capacity, water solubility, hygroscopicity, microstructure, glass transition temperature, color and rheology of the solutions were analyzed. Different degrees of caking were observed in the powders, which were related to the low glass transition temperature of the ingredients, due to their low molecular weight components. Although all the solutions formed foams, the foam of the ingredient obtained by extrusion + hot water was the most stable, which could be due to its higher viscosity.

In the third stage, the ingredients obtained were stored at different water activities for 15 weeks, and once the time had elapsed, their sorption isotherm was modeled. In addition, the glass transition was studied, along with the content of bioactive compounds, color, and texture. The powders were not stable in their initial state, due to their high moisture content. Browning was observed as the water activity increased. The hardness presented a maximum in water activities of around 0.5 (depending on the treatment 0.43 – 0.52) and then decreased. The only sample for which acceptable storage conditions were found with its starting humidity was the one resulting from extrusion + hot water, which should be stored at refrigeration temperature. In other cases, it would be necessary to adjust the process parameters to reduce the final moisture content of the ingredient.

In the last stage, the incorporation of the ingredient in the formulation of a flan was evaluated. Specifically, the effect of information about the ingredient on consumer response and the effect of its incorporation on the sensory properties of the dessert were studied. To evaluate the effect of information on consumer response, a survey was carried out in Spain and Uruguay, taking purchase intention and healthiness and environmental friendliness perceptions as study variables. Three different product categories were

evaluated (a ready-to-eat product, a powder to prepare dessert at home and a homemade flan), varying the information offered (origin of the fiber and a logo referring to sustainability). The behavior of both populations was different in the three study variables, although they agreed that the product with the greatest purchase intention is the homemade product with a sustainable logo. The purchase intention was not correlated with healthiness and environmental friendliness perception in all cases, although these last two variables did remain correlated.

To evaluate the incorporation of the ingredient in the formulation of a "fiber source" (total fibre content 3%) flan, different percentages of substitution of inulin content (control sample) the total fiber content of the dessert by the orange ingredient obtained from extrusion + hot water were tested. The substitution of 30% of the total fiber content with the new ingredient achieved a product similar to the control (with fiber content corresponding to 100% inulin), without unpleasant flavor descriptors and with an improvement in brightness and texture. To achieve higher substitution percentages, it would be necessary to work on the extraction process, in order to eliminate or reduce burnt, bitter and acid flavors, thus expanding its potential use.

Introducción

La fibra alimentaria

La definición de fibra alimentaria ha ido variando a lo largo de los años y continúa siendo controvertida hoy en día. Esto se debe a varias razones, en primer lugar, comprende un grupo de compuestos complejos, y no un único tipo, y, en segundo lugar, no se le puede atribuir un efecto fisiológico en común, ya que no todas las fibras tienen el mismo efecto (De Menezes et al., 2013; Jones, 2014).

En un principio, la fibra se definía como los hidratos de carbono no digeribles por el sistema digestivo, asociados a la pared celular de las plantas. Con el tiempo se fueron agregando otros compuestos, tales como gomas, mucílagos y almidones resistentes (Dhingra et al., 2012). Además de la definición basada en su composición, se ha definido a la fibra en cuanto a sus efectos fisiológicos, solubilidad, fermentabilidad o viscosidad de sus componentes (Esteban et al., 2017).

Se ha ido formando un consenso en torno a la definición del CODEX del 2009, en la que se define la fibra como los carbohidratos de grado de polimerización mayor a 10, que no puedan digerirse por las enzimas endógenas del intestino delgado del ser humano. Se incluyen polímeros asociados a lignina u otros compuestos. Además, estos carbohidratos pueden o existir naturalmente en los alimentos, o ser extraídos de materiales alimentarios, o bien ser polímeros sintéticos. Para que estos dos últimos sean considerados fibra, sin embargo, deben haber demostrado ejercer algún tipo de efecto beneficioso en la fisiología (De Menezes et al., 2013; Esteban et al., 2017; Gidley & Yakubov, 2019; Jones, 2014; Maphosa & Jideani, 2016; Westenbrink et al., 2013).

Dentro de esta controversia también se encuentra el punto de corte de la polimerización para que se considere como fibra alimentaria. Aunque hace unos años la fibra comprendía los oligosacáridos de grado de polimerización mayor a 10, en los últimos años se ha empezado a incluir aquellos de polimerización entre 3 y 9, debido a que estos también ejercen efectos beneficiosos en el cuerpo humano (De Menezes et al., 2013; Jones, 2014; O'Grady et al., 2019; Westenbrink et al., 2013).

La fibra alimentaria normalmente se divide de acuerdo a su solubilidad en los fluidos biológicos (solución enzimática de pH controlado), en fibra insoluble y fibra soluble (Tosh & Yada, 2010). La fibra insoluble está asociada a un aumento del volumen fecal, y facilita el movimiento intestinal, la secreción de los ácidos biliares y además disminuye el tiempo de tránsito intestinal. Este tipo de fibra es el menos fermentable en el intestino grueso. Es muy abundante en el salvado de cereales, principalmente compuesto por ligninas y celulosa. También forman parte de la fibra insoluble algunas hemicelulosas, compuestas por esqueletos de glucosa unidas por enlaces de tipo β, pero más cortas que la celulosa y a menudo ramificadas con otros azúcares, como xilosa, galactosa, manosa y arabinosa, entre otros (Dhingra et al., 2012; Y. O. Li & Komarek, 2017; Maphosa & Jideani, 2016).

La fibra soluble, por otro lado, pasa el proceso digestivo y es fermentada por la microflora del intestino grueso. Se encuentra en frutas, verduras, legumbres, entre otros, mayoritariamente. Este tipo de fibra se asocia a un aumento de la viscosidad de la ingesta, por lo que se enlentece el vaciado gástrico y la absorción de nutrientes. También se asocia a una reducción de la respuesta glicémica y de los niveles de colesterol, a la capacidad de quelado del sodio, y a una mejora del estreñimiento, entre otras. Está compuesta por oligosacáridos, pectinas, gomas, fructanos del tipo de la inulina, betaglucanos y algunas hemicelulosas (Bader Ul Ain, 2019; Gidley & Yakubov, 2019; Mudgil, 2017).

Además de los efectos fisiológicos anteriormente mencionados, la fibra alimentaria del tipo soluble es muy importante por sus efectos como prebiótico. Su fermentación en el intestino delgado es importante para el mantenimiento de la microbiota intestinal sumado a que los productos de la fermentación, tales como ácidos grasos de cadena corta, ejercen efectos beneficiosos para la salud (Garcia-Amezquita et al., 2018; Maphosa & Jideani, 2016; Redgwell et al., 2011)

La proporción fibra insoluble: fibra soluble en los diferentes alimentos es muy variable. La del tipo insoluble es más abundante en los alimentos, ya que la mayoría de los alimentos que contienen fibra tiene aproximadamente dos tercios del tipo insoluble (Mudgil, 2017).

Aparte de los efectos fisiológicos de este grupo de compuestos, la fibra alimentaria tiene beneficios tecno funcionales que pueden ser aprovechados en la formulación de alimentos. Destacan el aumento de volumen, absorción de agua y aceite, formación y estabilización de espumas y emulsiones, modificación de la textura y viscosidad, entre otras (Elleuch et al., 2011; Sharma et al., 2013; L. Wang, Xu, Yuan, Pan, et al., 2015)

La fibra soluble presenta beneficios tecnológicos, principalmente asociados a su solubilidad en agua. El uso de este ingrediente en la formulación de alimentos mejora la textura y sabor en comparación con la fibra insoluble, que impacta negativamente en el sabor, color y textura de los productos suplementados (Bader Ul Ain et al., 2019; L. Wang et al., 2015).

La brecha en el consumo de fibra

El estilo de vida actual trae aparejado un descenso en la ingesta diaria de fibra alimentaria. Esto trae consigo problemas de salud pública, ya que el bajo consumo de este tipo de compuestos está asociado a una alta prevalencia de enfermedades no transmisibles, tales como diabetes, diverticulosis, cáncer de colon, enfermedades coronarias, entre otras (O'Keefe, 2019).

La ingesta diaria de fibra alimentaria recomendada por la OMS es de al menos 25 gramos al día (Maphosa & Jideani, 2016). Algunos países tienen sus propias recomendaciones, basadas en la ingesta calórica, por lo que se diferencia por edad y sexo de la persona, como, por ejemplo, Estados Unidos y Canadá, donde la ingesta diaria recomendada es de 25 gramos para las mujeres y 38 gramos para los hombres. El Reino Unido, por otro lado, recomienda una ingesta diaria de 30 gramos (Evans, 2020; O'Keefe, 2019). La variabilidad de los valores diarios recomendados se debe a las diferentes definiciones de fibra adoptadas por cada país. A pesar de estas diferencias, en la mayoría de los países el consumo está por debajo de ese valor (Jones, 2014; Maphosa & Jideani, 2016; O'Keefe, 2019).

Una forma de contribuir a alcanzar el consumo de fibra recomendado es la fortificación de alimentos con fibra alimentaria (L. Wang et al., 2017). Teniendo en cuenta los beneficios tecno funcionales de estos compuestos, esta fortificación no solo es una buena forma de aumentar la ingesta de fibra, sino que además podría permitir la sustitución total o parcial de determinados aditivos tales como emulsionantes, espesantes, estabilizantes y sustitutos de grasa, y de esta forma crear productos con un etiquetado limpio o "clean label" (Asioli et al., 2017; Feng et al., 2017; Gu et al., 2020). Esta fortificación puede aumentar la retención de agua y de aceite, formar geles y emulsiones, modificar texturas, y reducir la sinéresis (Elleuch et al., 2011; Maphosa & Jideani, 2016).

En ese sentido, se ha estudiado la adición de fibra de diferentes fuentes a la formulación de alimentos, como forma de aumentar la ingesta diaria de fibra alimentaria. Ciertos compuestos con capacidad antioxidante, tales como carotenoides y polifenoles, se encuentran embebidos en la fibra, por lo que la suplementación de ésta indirectamente aporta compuestos bioactivos (Palafox-Carlos et al., 2011; Saura-Calixto, 2011). En la Tabla 1 se presentan ejemplos de productos formulados con incorporación de fibra alimentaria. Es interesante mencionar que estas fibras provienen de materiales considerados subproductos de la agroindustria.

Tabla 1. Productos formulados con incorporación de fibra alimentaria

Producto fortificado	Subproducto utilizado	Referencia
Galletas	Bagazo de arándanos	Perez et al. (2018)
	Bagazo de uva	Lou et al. (2022)
	Bagazo de mora	Reißner et al. (2021)
	Bagazo de naranja	Fernández-Fernández et al. (2021)
Helado	Fibra de avena, de trigo, y de manzana	Soukoulis et al. (2009)
	Fibra de naranja	Loffredi et al. (2021)
Panificados	Bagazo de naranja	Romero-Lopez et al. (2011)
	Bagazo de mora	Diez-Sánchez et al. (2019) Diez-Sánchez et al. (2020)
	Bagazo de olivas	Simsek & Süfer (2021)
Postres lácteos, yogures	Bagazo de uva	Iriondo-DeHond et al. (2020)
	Bagazo de manzana	Varnaitė et al. (2022)
Productos cárnicos	Bagazo de uva	Solari-Godiño et al. (2017)
Bebidas	Bagazo de naranja	Dong et al. (2016)
	Bagazo de cervecería	Curutchet et al. (2022)

Tratamientos sobre la fibra soluble

Debido a los beneficios, tanto nutricionales como tecno funcionales, la fibra soluble presenta especial interés frente a la fibra insoluble. Sin embargo, como ya se ha comentado, la mayoría de los productos de base vegetal contienen mayor cantidad de fibra insoluble. Por lo tanto, los esfuerzos se han focalizado en estudiar diferentes tecnologías para llevar esa relación hacia un aumento de la fibra soluble sobre la insoluble; estos procedimientos mejoran el perfil tecno funcional del ingrediente (Bader Ul Ain et al., 2019; Tejada-Ortigoza et al., 2017). Dentro de los procesos utilizadas, destaca el aumento en el uso de tecnologías "verdes", reduciéndose el uso de solventes, implicando un menor impacto ambiental (Chemat et al., 2012). Estas tecnologías pueden utilizar fuerzas mecánicas (diferentes moliendas, micronización), extrusión, procesos térmicos (agua caliente, uso de vapor, esterilización, microondas), fermentaciones microbiológicas, altas presiones, o la combinación de alguna de ellas.

En la Tabla 2 se presenta un breve resumen de algunos trabajos que aplican este tipo de tecnologías al tratamiento de subproductos. Como característica común de estos trabajos reportados, es que en todos ellos se han utilizado diversos subproductos como sustratos para la extracción de fibra soluble, con el consiguiente objetivo de disminuir la generación de residuos en un marco de economía circular

Tabla 2. Tecnologías de extracción de fibra sobre subproductos

Tecnología	Subproducto	Referencia
Agua caliente	Cáscara y semillas de naranja	Gutiérrez Barrutia et al. (2019)
Altas presiones hidrostáticas	Piel de diferentes frutas: mango, naranjas, tunas	Tejada-Ortigoza et al. (2017)
	Patatas moradas	Xie et al. (2017)
Extrusión	Cáscara de naranjas	Garcia-Amezquita et al. (2019)
	Bagazo de mora	Diez-Sánchez et al. (2019)
	Bagazo de manzana	Schmid et al. (2020)
Explosión a vapor	Okara	B. Li et al. (2019)
	Residuo del boniato	T. Wang et al. (2017)
	Cáscara de naranja	L. Wang, Xu, Yuan, Pan, et al. (2015)
Extracción asistida por ultrasonido	Semillas de cítricos	Karaman et al. (2017)
	Cáscara de papaya	Zhang et al. (2017)
	Flaxseed	Moczkowska et al. (2019)
Jet cooker	Cítricos	Cameron et al. (2015, 2017)

La extrusión resulta interesante, ya que logra modificar la relación fibra soluble – insoluble mediante la combinación de alta presión, temperatura y fuerza de cizalla. El proceso rompe los enlaces glicosídicos de los polisacáridos y, al hacerlos más cortos, los solubiliza. Además del aumento del contenido de fibra soluble, esta tecnología aumenta la capacidad de retención de agua y disminuye la capacidad de retención de aceite del producto extruido (Huang & Ma, 2016).

La aplicación de procesos térmicos con uso de vapor se basa en la capacidad del vapor de expandirse dentro de la matriz alimentaria y romper los enlaces glicosídicos, acortando las cadenas de polisacáridos (Felker et al., 2018). Este es el caso de la explosión a vapor, en la que la matriz es inyectada con vapor y luego descomprimida de forma rápida: esta descompresión del vapor hace que las burbujas formadas exploten y rompan las paredes celulares, disminuyendo el peso molecular de los compuestos presentes (Wang, Xu, Yuan, Fan, et al., 2015). Por otro lado, en la inyección de vapor que se da en el proceso de "jet cooker" no tiene lugar esa expansión del vapor fruto de la descompresión. Sin embargo, la fuerza de cizalla generada con la propia inyección de vapor podría romper los enlaces glicosídicos de los polisacáridos. De hecho, Felker et al. (2018) estudiaron el efecto de esta tecnología en los gránulos de almidón y observaron que se rompen y liberan la amilosa y la amilopectina tras pasar por este proceso.

Otra alternativa de tratamiento térmico es el uso de agua caliente. Gutiérrez Barrutia et al. (2019) estudiaron esta técnica para la revalorización de la cáscara de naranja mediante la obtención de ingredientes funcionales, y obtuvieron un ingrediente rico en antioxidantes y con contenido de fibra soluble.

La extracción asistida por ultrasonido es una alternativa atractiva, ya que es una tecnología de rápida aplicación y de bajo consumo energético. Dado que no utiliza temperaturas elevadas, los compuestos bioactivos presentes en la muestra no se degradan. Las ondas de ultrasonido logran romper los tejidos vegetales debido a la cavitación acústica, liberando los compuestos extraíbles (Pollini et al., 2022). Esta tecnología a menudo se combina con álcalis o con enzimas, y se utiliza como una asistencia a la extracción de fibra (Moczkowska et al., 2019; Zhang et al., 2017).

La cáscara de naranja y su potencial como fuente de fibra

A nivel mundial se producen 49.0 millones de toneladas de naranjas (*Citrus sinensis*) (USDA 2021-2022). Esta fruta es una de las más cultivadas, siendo Brasil, China, Estados Unidos, México y algunos países europeos los principales productores (De Medina-Salas et al., 2020; Lado et al., 2018).

Aunque la producción y el consumo anual de jugo de naranja han ido disminuyendo en los últimos diez años en la Unión Europea y Estados Unidos (USDA 2021-2022), éste sigue siendo el principal destino de la fruta industrializada. En esta producción, solamente el 50% del peso fresco se transforma en jugo (De Medina-Salas et al., 2020; Karaman et al., 2017;

Negro et al., 2017). Por ello, se estima que anualmente se producen alrededor de 10 millones de toneladas de subproducto procedente de la obtención de zumo, compuesto por cáscara (60 – 65%), pulpa (30 – 35%) y semillas, y frutas que no cumplen los requerimientos de calidad (Fernández-Fernández et al., 2021; Negro et al., 2017).

Tradicionalmente el subproducto se destina a la alimentación animal, compostaje, o extracción de pectinas. Estas soluciones agregan poco valor al subproducto siendo poco atractivas económicamente para las empresas. El subproducto tiene un sabor amargo y bajo valor nutricional para la alimentación animal, y además debe ser secado, con su correspondiente costo energético. Por otro lado, el compostaje es costoso y no siempre es de interés para el mercado (Negro et al., 2017)

Recientemente se han propuesto otras formas de valorización de este residuo: biocombustibles, biorrefinería, extracción de aceites esenciales, colorantes o compuestos fenólicos, entre otros (De Medina-Salas et al., 2020; Özcan et al., 2021).

La cáscara de naranja fresca tiene una humedad de alrededor del 80%, el residuo seco se compone mayoritariamente por azúcares simples (glucosa, fructosa y sacarosa), fibra alimentaria (pectina, celulosa y hemicelulosa) y aceites esenciales (De Medina-Salas et al., 2020). Además, las cáscaras de naranja tienen alto contenido de metabolitos secundarios producidos por la fruta en respuesta al estrés medioambiental, como los polifenoles, flavonoides y carotenoides, con alto poder antioxidante (Özcan et al., 2021).

Dentro de los componentes anteriormente mencionados, destaca el contenido de fibra alimentaria, con propiedades de retención de aceite y de agua, quelado de cationes y capacidad de hinchamiento superior a la celulosa, entre otras. Estas propiedades de la fibra alimentaria, junto con el contenido de metabolitos secundarios, convierten a este residuo en un ingrediente con alto potencial para su uso en la formulación de nuevos alimentos y como matriz de extracción de otros ingredientes tales como pectinas y aceites esenciales (de Moraes Crizel et al., 2013; Pacheco et al., 2019).

La respuesta de los consumidores a los alimentos enriquecidos

Cerca de un tercio de los alimentos producidos globalmente se desperdicia. Una forma de reducir esta cantidad es lo que se denomina “upcycled foods”, en los que los desperdicios se utilizan como fuente de otro ingrediente (Coderoni & Perito, 2021; Grasso & Asioli, 2020; Spratt et al., 2020).

Como se ha mencionado anteriormente, el residuo de la industria citrícola podría destinarse a ello, específicamente, a la obtención de fibra soluble para la fortificación de alimentos. Sin embargo, aunque esto teóricamente resulte muy atractivo, en la práctica hay algunos obstáculos que superar. Además del costo asociado al desarrollo del nuevo ingrediente, existe incertidumbre por la falta de información de cómo reaccionará el consumidor frente a estos alimentos, así como también regulación respecto al etiquetado de este tipo de productos (Asioli & Grasso, 2021; Bhatt et al., 2021; Grasso & Asioli, 2020).

La reutilización de los residuos sólidos en la formulación de "upcycled foods" es una forma de contribuir a la sostenibilidad de la cadena alimentaria, por medio de la reducción del desperdicio de alimentos. En este contexto, el etiquetado de alimentos en relación a su sostenibilidad es una forma de concienciar al consumidor de lo que está ingiriendo, y de esta forma modificar sus patrones de consumo (Kaczorowska et al., 2019).

Fernández-Serrano et al. (2022) estudiaron mediante el uso de "eye-tracker" la percepción de los consumidores frente a vinos con mensajes asociados a la sostenibilidad, bajo el uso de un "claim" y de un logo, y, aunque los dos captan la atención de los consumidores, el logo fue el preferido al ser considerado más atractivo.

Por otro lado, existe una tendencia en aumento, sobre todo en los países desarrollados, del interés de los consumidores por consumir alimentos naturales, frescos y mínimamente procesados (Plasek et al., 2021; Román et al., 2017). Pero ¿qué es un alimento natural? Aparentemente, la percepción de natural está más afectada por el proceso de producción del alimento, que por el alimento en sí mismo.

En esa percepción de natural, el envase y la información que se brinda juegan un papel primordial. A la hora de la decisión de compra, los consumidores se basan tanto en factores intrínsecos como en factores extrínsecos. Los primeros son los asociados al producto en sí mismo, mientras que los extrínsecos se relacionan con él, pero no son parte de lo que se consume. Estos últimos son en los que principalmente el consumidor se basa a la hora de realizar su compra. Un resumen de ambos tipos se presenta en la Figura 1 (Plasek et al., 2021).

En su estudio, Plasek et al. (2021) encontraron que la presencia de un "claim" de ingrediente es uno de los factores que más afecta la percepción de saludable de una bebida funcional. Tanto Curutchet et al. (2021) como Grasso & Asioli (2020) encontraron que la aceptabilidad de sus productos aumentaba al informarle al consumidor del origen de la fibra.

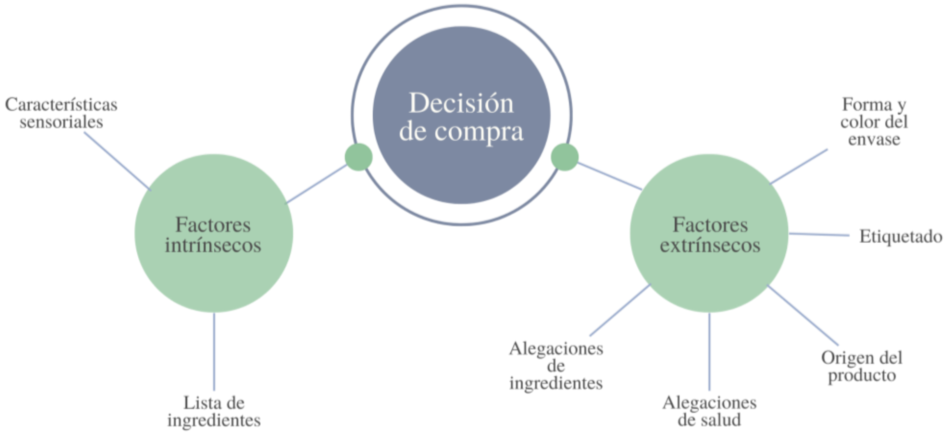

Figura 1. Factores intrínsecos y extrínsecos que afectan la decisión de compra (Plasek et al., 2021).

Aunque la mayoría de las personas son conscientes de que deben alimentarse de manera saludable, es difícil juzgar, en este sentido, de forma correcta a los alimentos. Por esto, los consumidores a menudo interpretan por saludables factores extrínsecos del producto, como su categoría, los descriptores de salud y las etiquetas. Por ejemplo, los consumidores tienden a encontrar más naturales los alimentos que están mejor presentados, y asocian a estos el concepto de más saludable (Hagen, 2021).

De acuerdo con los antecedentes anteriormente expuestos, la extracción de fibra soluble de la cáscara de naranja representa una estrategia innovadora de revalorización de este residuo industrial. El ingrediente obtenido podría utilizarse en la formulación de alimentos mejorando su perfil nutricional, y de esta forma contribuir a aumentar el consumo de fibra. Además, se podrá fomentar su consumo a través de estrategias efectivas que permitan informar al consumidor sobre el origen de estos alimentos.

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Objetivos

Como objetivo general de la tesis se planteó obtener y caracterizar ingredientes con alto contenido de fibra soluble a partir del subproducto de la industria de zumo de naranja, utilizando tecnologías verdes de extracción.

Para ello, se proponen cuatro objetivos específicos.

- Estudiar la composición en carbohidratos simples y complejos y compuestos bioactivos de los ingredientes enriquecidos en fibra obtenidos.
- Estudiar la microestructura y propiedades tecno funcionales de los ingredientes.
- Estudiar la estabilidad de cada ingrediente durante el almacenamiento a diferentes humedades.
- Estudiar la percepción de los consumidores sobre el uso de la fibra obtenida en la formulación de un postre tipo flan y su impacto en las propiedades sensoriales.

Estructura de la tesis

Esta tesis se divide en cuatro capítulos que buscan cubrir los objetivos planteados.

Los primeros dos capítulos se centran en la composición y propiedades de los ingredientes obtenidos.

En el tercer capítulo se estudia la estabilidad de los ingredientes en el almacenamiento a diferentes humedades, para determinar las condiciones óptimas que conservan sus características.

Finalmente, el último capítulo estudia la percepción de los consumidores uruguayos y españoles de un postre tipo flan con el agregado de fibra de naranja. También se realiza una caracterización sensorial del flan formulado con uno de los ingredientes obtenidos para la fortificación del contenido de fibra.

Capítulo 1: Métodos de extracción de fibra soluble del subproducto de la industria citrícola para la obtención de un ingrediente funcional en polvo

Different green extraction technologies for soluble dietary fibre extraction from orange by-product. *International Journal of Food Science and Technology, (2022), 10.1111/ijfs.15756*

Capítulo 2: Caracterización tecno funcional de los ingredientes obtenidos a partir del subproducto de la industria citrícola

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Capítulo 3: Estudio de la estabilidad en el almacenamiento de los ingredientes obtenidos a partir del subproducto de la industria citrícola

Sorption isotherms, glass transition and bioactive compounds of ingredients enriched with soluble fibre from orange pomace. Enviado a *Foods*

Capítulo 4: Percepción de los consumidores. Incorporación a un postre tipo flan.

Towards halving food waste: A comparative study using orange juice by-product in dairy desserts. Enviado a *Heliyon*

Incorporating an upcycled orange fibre on flan formulation. Impact on sensory properties. Short communication, en preparación para envío a *LWT – Food Science and Technology.*

Resultados y discusión

Capítulo 1: Métodos de extracción de fibra soluble del subproducto de la industria citrícola para la obtención de un ingrediente funcional en polvo

Different green extraction technologies for soluble dietary fibre extraction from orange by-product

Perez-Pirotto, Claudia [a, b], Cozzano, Sonia [a], Hernando, Isabel, [b], Arcia, Patricia [a,c]

[a] – Departamento de Ingeniería, Universidad Católica del Uruguay. Montevideo 11600, Uruguay.

[b] - Food Microstructure and Chemistry Research Group, Department of Food Technology, Universitat Politècnica de València, Valencia 46022, Spain

[c] - Latitud Latu Foundation. Montevideo 11500, Uruguay.

Corresponding author: Patricia Arcia, Latitud Latu Foundation, Av. Italia 6201, 11500, Montevideo, Uruguay. E-mail: parcia@latitud.org.uy

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Abstract

The aim of this work was to extract soluble dietary fibre from orange by-product, by testing four different green (non-contaminant, solvent free) extraction technologies: hot water, extrusion + hot water, jet cooker and jet cooker + hot water. Starting from orange pomace, the treatments were assayed and sample was separated in soluble and insoluble fractions. The processing and analysis of the soluble fraction was continued, through subsequent spray drying until obtaining a soluble fibre enriched powder. Powders were analysed: dietary fibre, sugar profile and bioactive characterization (total polyphenol content, antioxidant capacity). Through the application of these green technologies, it was possible to obtain a functional ingredient with soluble dietary fibre from orange by-product. Extrusion + hot water was the treatment that yielded the highest amount of soluble dietary fibre, the lowest content of glucose, sucrose and fructose, and the highest polyphenol content and antioxidant capacity.

Keywords

Green technologies, soluble fibre, orange by-product, antioxidants, extrusion, jet cooker.

1. Introduction

According to FAO, in 2019 approximately 76.3 million tons of oranges were harvested worldwide (FAO, 2021), and almost 17 million tons were destined for industrialization. From these fruits, around 50% w/w (wet weight) is waste, composed of seeds, peels and internal tissue (Wilkins et al., 2007), which means nearly 8.5 million tons were produced. This waste is normally discarded as a residue (landfill, composting), used as animal feed, or as a substrate for pectin extraction (Rafiq et al., 2018; Negro et al., 2017; Sharma et al., 2016). However, these approaches neglect the potential health benefits of this orange by product, mainly due to dietary fibre and bioactive compounds.

Regarding dietary fibre, its definition has been controversial throughout the years, since it cannot be defined as one specific component. However, all definitions include carbohydrate polymers and oligomers that reach the large intestine without being digested. Depending on its water solubility, it can be classified as soluble and insoluble. More recently, several groups have started to include low molecular weight fibre, which is composed of short chain oligomers (degree of polymerisation 3-9).

In oranges, the proportion of soluble fibre is higher than in other sources of dietary fibre (such as cereals). There is evidence that fibre consumption lowers the prevalence of several diseases (Jones, 2014, Chutkan et al., 2012). Its consumption prevents several non-transmittable diseases, such as lower blood lipid and glucose levels and some types of cancer. Soluble fibre has been found to play a major role in these health benefits (Wang et al., 2015). Unfortunately, there is a worldwide shortage of fibre consumption, therefore a strategy to reverse this situation is to incorporate fibre in food preparation.

Extracting fibre and especially soluble fibre from the orange peel or orange by-product, would allow to obtain a functional ingredient to be used in the supplementation of other foods, in order to close the so-called “fibre gap” (Jones, 2014).

In this context, it is interesting to focus on the characteristics of the extraction processes to be used according to today's consumer trends. Consumers are now becoming more health conscious and demand “clean label” products. Hence, the ingredients to be used should be obtained from natural sources (Elleuch et al., 2011). Moreover, there is an increasing interest in environmental care, demanding for processes that are less contaminant and use less solvents. The commonly named “green” technologies fulfil these demands, as they are solvent-free and non-contaminant. One of the principles of these technologies is the use of water as an alternative solvent (Chemat et al., 2012). Moreover, several solvent free processes have been tried to extract soluble fibre, including ultrasound, extrusion, steam explosion, among others (Arora et al., 2020; Renard, 2018).

Therefore, the aim of this study was to try different green technologies, such as hot water, extrusion and jet cooking, to obtain a new ingredient, with a high content of soluble dietary fibre, from orange pomace.

2. Materials and methods

2.1. Orange pomace

Orange pomace was obtained as a by-product of industrial juice production, supplied by Novacore (Uruguay).

2.2. Treatments for fibre extraction

Four different treatments for fibre extraction (hot water, extrusion + hot water, jet cooking and jet cooking + hot water) were used (Figure 1). They were performed in duplicate, in pilot scale equipment.

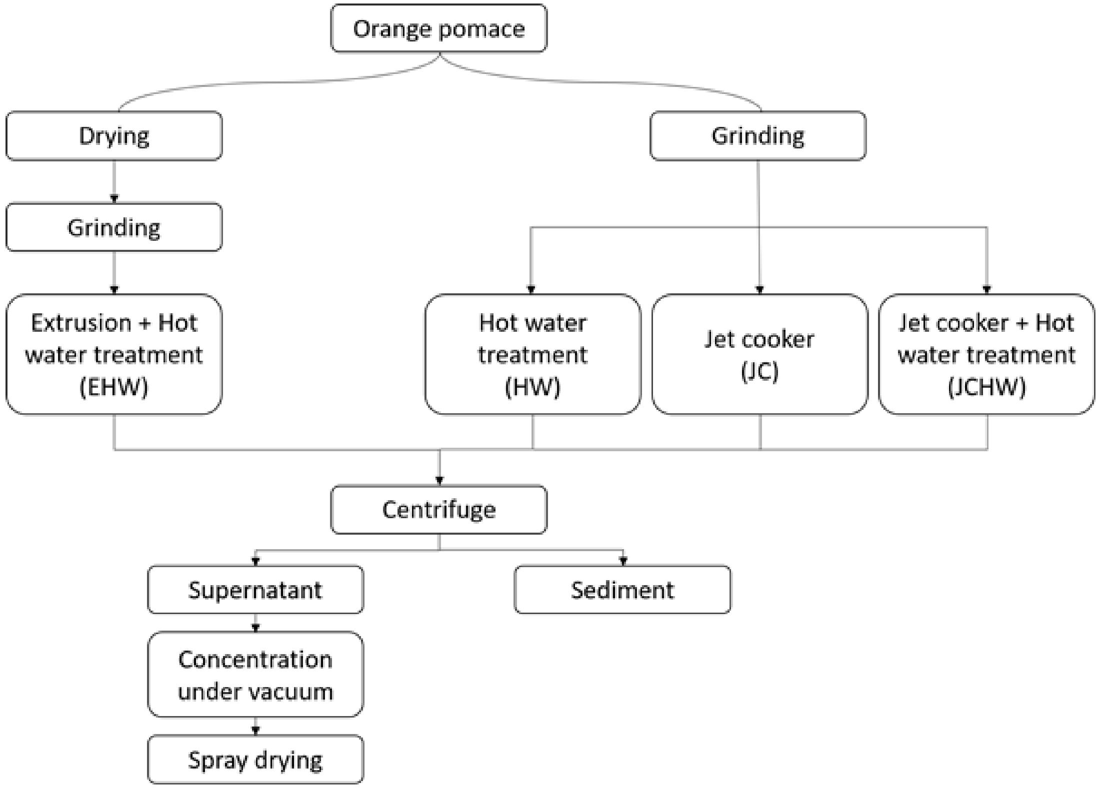

Figure 1. Processing of the orange pomace for fibre extraction

2.2.1. Sample preparation

Except for extrusion + hot water extraction, all procedures were performed on wet orange pomace. It was ground in an industrial cutter to particle size of less than 1.0 mm. The resulting product was stored frozen at -18°C on closed polyethylene bags until use.

For extrusion + hot water extraction, orange pomace was dried in a convection oven at 50°C for 72 hours. It was ground in a laboratory mill

(Retsch ZM 200) to obtain a particle size of less than 1.0 mm. Powder was stored on polyethylene bags at room temperature until use.

2.2.2. Hot water (HW)

Hot water extraction was performed based on Gutiérrez Barrutia et al. (2019), with modifications. Orange pomace was mixed with water (1:2, w/v) in a mechanically agitated (31 rpm) boiler. Mixture was indirectly heated with steam and kept at 75°C for 45 minutes. The mixture was cooled and filtered with a cheesecloth. Supernatant and sediment were kept separated.

2.2.3. Extrusion + hot water (EHW)

Extrusion was performed according to Huang & Ma (2016), with modifications. Orange pomace powder's humidity was adjusted to 15% and left overnight at room temperature to reach equilibrium. The slurry was then passed through a single screw extruder (Brabender E330) with barrel temperature 129°C and screw speed of 230 rpm. After grinding the extruded sample to a size of less than 1.0 mm in a laboratory mill (Retsch ZM 200), it was mixed with water (1:16.6, w/v) in a mechanically agitated (31 rpm) boiler. Slurry was indirectly heated with steam, and kept at 75°C for 45 minutes.

2.2.4. Jet cooker (JC)

Jet cooker equipment is composed of a pump, a pipe, a steam injection valve and an exit valve. In this continuous process the sample is pumped through the pipe in which steam is injected. Procedure was performed according to Felker et al. (2018), with modifications.

Wet orange pomace was mixed with cool water (ratio 1:2.5 respectively w/v). The obtained slurry was passed through the jet cooker, with a residence time in the heating element between 3 and 5 seconds. It was operated using a temperature of 85°C, steam pressure 2.8 bar. Exit pressure was 1.0 bar. After cooling the slurry, it was filtered with a cheesecloth and supernatant and sediment were separated.

2.2.5. Jet cooker + hot water (JCHW)

After going through the jet cooker and before being filtered, the sample passed through the hot water extraction procedure.

2.3. Spray - drying of the soluble fraction

Supernatants from HW, JC, and JCHW, and slurry from EHW were centrifuged (Sigma 6-16 KS) at 9500 rpm for 40 minutes at 4°C. Supernatant and sediment were frozen separately.

Supernatants from all treatments were concentrated under vacuum at 75°C. After reaching 15 ± 2 ˚Bx, they were dried on a Buchi B290 spray dryer. Whey protein isolate (WPI - Provon 292, Glanbia Nutritionals Inc.) was used as an encapsulating agent (8% on total solids basis). Conditions for drying were: 130°C inlet air temperature, atomization air flow rate 414 L/h, liquid feed pump rate 9 mL/min, main drying air flow rate 35 m^3/h. Outlet temperature was 70 ± 4˚C (Edrisi Sormoli & Langrish, 2016).

Powders were stored in laminated pouches at room temperature.

2.4. Proximate analysis of raw material

Orange pomace composition was analysed. Protein was determined using AOAC 984.13 method (AOAC, 2012). Ash was determined following procedure ISO-2002-2002. Fat was estimated following the ISO-1999-1999 procedure. Carbohydrate content was calculated by difference. Insoluble and soluble dietary fibre were determined by AOAC 991.43 method (AOAC, 2012), using the TDF kit from Megazyme.

2.5. Powder characterization

Powder obtained from the four treatments assayed were characterized according to next determinations.

2.5.1. Dietary fibre quantification

Since only supernatants were spray dried, dietary fibre is expected to be from soluble type. Spray dried powders' fibre content (high molecular and low molecular weight dietary fibre, HMWDF and LMWDF, respectively) were analysed by the enzymatic-gravimetric-liquid chromatography method AOAC 2009.01 (AOAC, 2012). This method separates the conventionally measured dietary fibre (insoluble, and soluble dietary fibre, which precipitates with ethanol: water 4:1), which is high molecular weight dietary fibre (HMWDF), from the nonprecipitable soluble dietary fibre of degree of polymerization ≥ 3, which is considered low molecular weight dietary fibre (LMWDF). Megazyme K-ITDF kit was used. D-Glucose from Sigma Aldrich was used as the calibration standard for HPLC. D-sorbitol was used as internal standard, provided by Megazyme kit. A Waters Sugar-Pak® 6.5 x 300 mm column was used. Manual deionization was performed using Ambersep® 200 H and Amberlite® FPA OH^- resins, from Megazyme.

2.5.2. Carbohydrate analysis

Sugar analysis was performed by high performance liquid chromatography, following AOAC 977.20. Fructose, glucose, sucrose, xylose and arabinose were quantified. Standards were of HPLC grade, from Sigma-Aldrich.

2.5.3. Bioactive characterization

2.5.3.1. Extraction for polyphenol content and antioxidant capacity

Polyphenols and antioxidants were extracted with ethanol. Briefly, 50 mg of the sample were mixed with 1.0 mL of 96% ethanol and centrifuged at 4°C for 10 minutes at 10000 rpm. 0.9 mL of the supernatant were removed and stored, and the same amount of 96% ethanol was added. Samples were centrifuged again and supernatants were mixed. Volume was adjusted to 10 mL with ethanol. Extractions were performed in triplicate and all extracts were kept at -20°C until use in glass bottles protected from light.

2.5.3.2. Total polyphenol content

Total polyphenol content was determined by Folin Ciocalteu method. Briefly, 1.0 mL of extract (or standard dilution) was mixed with 6 mL of distilled water and 0.5 mL of Folin-Ciocalteu reagent (ITW Reagents, Spain). Tubes were vortexed and left in the dark for 3 minutes, when 1 mL of 20% Na_2CO_3 solution and 1.5 mL of water were added. Tubes were once again vortexed and samples were left in the dark at room temperature for ninety minutes. Absorbance was measured at 765 nm in plastic cuvettes. Gallic acid (ITW

Reagents, Spain) was used for calibration purposes. Total polyphenol content was expressed as mg of gallic acid equivalents per g of dry matter.

2.5.3.3. Antioxidant capacity

FRAP

Ferric reducing ability of plasma (FRAP) assay was performed following Pulido et al. (2000) procedure, with minor modifications. Briefly, 30 μL of water and 30 μL of sample (ethanol for blank) were mixed with 900 μL of freshly prepared FRAP reagent and left at 37°C for 30 minutes in the dark. Absorbance was measured at 595 nm after that time. Trolox (Santa Cruz Biotechnology, USA) was used for calibration purposes.

DPPH

Antioxidant activity of the ingredients was also evaluated by DPPH assay, following Shah et al. (2016) method, with modifications. 1 mL of extract was mixed with 0.004% (w/v) freshly prepared DPPH reagent (Sigma Aldrich, USA) in ethanol and left in the dark for 30 minutes at room temperature. Control was prepared with DPPH and solvent (ethanol). Absorbance was read at 517 nm. Trolox (Santa Cruz Biotechnology, USA) was used for calibration purposes.

2.6. Statistical analysis

Analyses were performed in duplicate. Results are expressed as mean ± SD. One way analysis of variance (ANOVA) was performed. Differences between samples were determined by the LSD Fisher test with p-value 0.05. Aiming to study correlation between fibre content, antioxidant capacity, polyphenol count and sugar content, a Pearson correlation test was performed, with p-value 0.01.

All statistics analysis were performed using XLSTAT 2021.2.1 software (Addinsoft 2021, New York, NY, USA).

3. Results and discussion

3.1. Orange pomace composition

Protein content was 3.94 ± 0.09 g/100 g dwb, ash content was 2.61 ± 0.08 g/100 g dwb, and fat content was found 0.73 ± 0.37 g/100 g dwb. Carbohydrates' content was 49.93 g/100 g dwb.

All contents were lower than those reported by Gutiérrez Barrutia et al. (2019) These differences could be due to the ripening stage of the oranges at the moment of harvest, as well as to different varieties of orange in the pool sample.

As Garcia-Amezquita et al. (2018), did not find significant differences on dietary fibre content in orange peels between traditional methodology (soluble and insoluble fibre contents - AOAC 991.43) and integrated methodology (AOAC 2011.25), orange pomace was analysed according to traditional methodology. Total dietary fibre content was 42.76 ± 0.77 g/100 g dwb, from which 26.41 ± 2.91 g/100 g dwb correspond to insoluble dietary fibre and 16.35 ± 2.14 g/100 g dwb to soluble dietary fibre. These results agree with those reported by Gutiérrez Barrutia et al. (2019)

3.2. Effect of treatments in fibre content

Results of high molecular weight (HMWDF), low molecular weight (LMWDF) and total dietary fibre (TDF) are shown on Table 1.

The treatment that yielded the higher amount of total soluble fibre was extrusion + hot water (EHW). This result is in agreement with those reported by bibliography (Huang & Ma, 2016; Redgwell et al., 2011). Extrusion significantly increases the amount of soluble fibre in the sample, by fostering the conversion of insoluble to soluble fibre. This conversion may be due to the mechanical transformation during extrusion that increases the soluble fibre content (Arora et al., 2020). Previous studies (data not shown) showed that the extrusion pre-treatment of the orange pomace powder did not change total dietary fibre but increased soluble fibre and decreased insoluble fibre.

Table 1. Values (mean ± SD) of powders' high (HMWDF), low (LMWDF) molecular weight and total dietary fibre (TDF). HW: hot water; EHW: extrusion + hot water; JC: jet cooker; and JCHW: jet cooker + hot water

	HMWDF (g/100 g dwb)	LMWDF (g/100 g dwb)	TDF (g/100 g dwb)
HW	8.00 ± 0.32^{a}	1.48 ± 0.56^{a}	9.48 ± 0.45^{a}
EHW	14.15 ± 0.80^{c}	6.05 ± 3.07^{b}	20.20 ± 3.69^{b}
JC	7.96 ± 1.37^{a}	2.29 ± 1.30^{a}	10.25 ± 0.89^{a}
JCHW	10.01 ± 0.24^{b}	3.03 ± 1.57^{ab}	13.03 ± 1.8^{a}

Mean values in each column followed by different letters were significantly different ($p<0.05$).

According to Maphosa & Jideani (2016), soaking modifies the composition and availability of fibres. Also, heat changes the ratio soluble: insoluble fibre. In the case of hot water extraction treatment (HW), results were similar to those reported by Gutiérrez Barrutia et al. (2019), who obtained a fibre content of 10.20 ± 0.05 g/100 g sample dwb in the supernatant of the extraction performed by lab scale.

The use of a jet cooker (JC) did not significantly ($p>0.05$) improve fibre extraction compared to hot water (HW). To this day, no bibliography was found on jet cooker usage for fibre extraction in fruits. Work has been done studying the effect of jet cooking, especially in legumes and cereals (de la Rosa-Millán et al., 2020; Felker et al., 2018). Jet cooking may result in changes in the ratio soluble: insoluble fibre fractions, because of the shear forces that are generated due to the hydrothermal process. These shear forces disrupt the internal structure of the cell walls, increasing soluble fibre (de la Rosa-Millán et al., 2020). Felker et al. (2018) found significant increase in the soluble fibre content when treating pinto beans with the jet cooker. de la Rosa-Millán et al. (2020), also found an increase in soluble dietary fibre when jet cooking chickpea flour. Both authors found that total dietary fibre content did not vary significantly. Several factors may explain the difference between these cases and the orange pomace one. Firstly, they measured the amount of soluble fibre in the whole ingredient, while in our study an extraction was performed and only the supernatant was analysed. Thus, the sediment may still have soluble fibre that was not extracted. Adding the hot water extraction after the jet cooking step (JCHW) significantly ($p<0.05$) improved high molecular weight fibre extraction when comparing to both treatments on their own. This may indicate that keeping the sample at a high temperature would improve the extraction of the linkages that may have been debilitated in the jet cooking process.

No significant differences ($p>0.05$) were found on the amount of low molecular weight dietary fibre extracted by hot water, jet cooking and the combination of both. However, extrusion improved extraction significantly ($p<0.05$) when compared to jet cooking and hot water, but not compared to the combination of both. Although the inclusion of these compounds in the amount of fibre is controversial, they are able to be fermented by the microorganisms in the large intestine, promoting growth and activity of bacteria in the digestive tract (Mudgil, 2017).

3.3. Sugar content

As dietary fibre is composed of saccharides, the content of some sugars (fructose, glucose, sucrose, xylose and arabinose) was assayed in order to check the degree of breakage of fibre. Neither arabinose nor xylose were detected (Figure 2). The absence of the latter two compounds suggests that rhamnogalacturonans were not degraded in the processes.

Extrusion + hot water (EHW) treatment yielded the least amount of sugars. Lower fructose and sucrose levels were observed by Schmid et al. (2020) in apple pomace when comparing extrusion with the apple pomace itself; they found that these sugars were degraded under thermomechanical stresses.

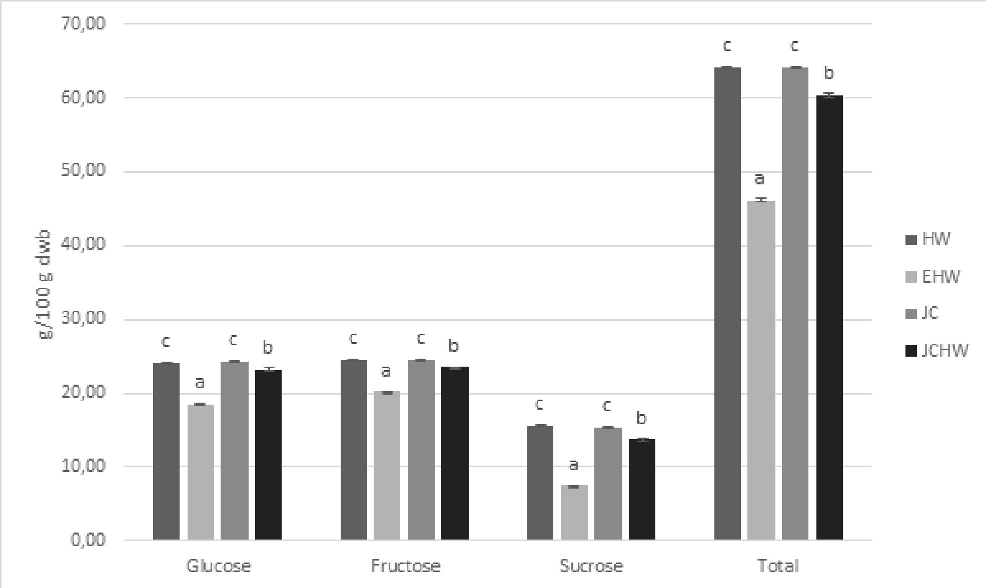

Figure 2. Glucose, fructose, sucrose y total sugar content of powders obtained after each extraction treatment. HW: hot water; EHW: extrusion + hot water; JC: jet cooker; and JCHW: jet cooker + hot water. Results are expressed in g/100 g dwb. Bars with different letters in the same group were significantly different ($p<0.05$).

Jet cooking (JC) and hot water (HW) showed the highest content in all the sugars analysed, with no significant differences ($p>0.05$). Jet cooking + hot water extraction had significantly lower contents ($p<0.05$) than JC and HW in all the sugars analysed. Since temperature fosters Maillard reactions and caramelization, and as JCHW is longer under higher temperatures, sugars could have undergone these reactions and be diminished (Zhang et al., 2019).

Although no bibliography was found regarding jet cooking effect on sucrose, glucose and fructose, Felker et al. (2018) studied the effect of this treatment on raffinose family oligosaccharides, finding an increase on them. This suggests that the treatment of the samples with steam may foment the release of mono and disaccharides from oligosaccharides, making them more extractable.

3.4. Bioactive characterization

Polyphenols and antioxidant compounds are described to be bound to dietary fibre (Rafiq et al., 2018; Tejada-Ortigoza et al., 2015, Marín et al., 2007), and orange pomace is known to have good antioxidant capacity (Gutiérrez Barrutia et al., 2019).

Total polyphenols content and antioxidant capacity are shown on Table 2.

Extrusion + hot water (EHW) yielded the powder with the highest polyphenol content and antioxidant capacity, both in FRAP and DPPH analyses. DPPH results were lower than those reported by Hernández-Carranza et al. (2016).

Table 2. Values (mean ± standard deviation) of total polyphenol content and antioxidant capacity of powders obtained after each extraction treatment. HW: hot water; EHW: extrusion + hot water; JC: jet cooker; and JCHW: jet cooker + hot water. GAE = gallic acid equivalent.

	Total polyphenol content (mg GAE/g sample dwb)	**DPPH (µmol Trolox/ g sample dwb)**	**FRAP (µmol Trolox/ g sample dwb)**
HW	4.34 ± 0.16^{b}	11.12 ± 0.37^{a}	48.26 ± 1.24^{b}
EHW	8.17 ± 0.38^{d}	14.36 ± 0.19^{b}	89.94 ± 3.25^{c}
JC	3.70 ± 0.26^{a}	10.67 ± 0.01^{a}	36.64 ± 1.55^{a}
JCHW	5.14 ± 0.28^{c}	11.86 ± 0.78^{a}	52.12 ± 2.52^{b}

Mean values in each column followed by different letters were significantly different ($p<0.05$).

Unlike what was observed by Gutierrez Barrutia et al. (2019), the negative correlation of temperature effect on supernatant antioxidant capacity is not seen in our work, as antioxidant capacity is highest in the treatment with the highest temperature (EHW). This could be due to extrusion breaking fibres and releasing the antioxidant components that cannot not be extracted when they are bounded to the fibre. Also, extrusion's higher temperatures may result in the formation of Maillard compounds that may have antioxidant capacity, such as reductones and melanoidins (Hafsa et al., 2021; Nooshkam et al., 2019).

Hot water and jet cooking + hot water had no significant differences ($p>0.05$) between them in antioxidant capacity, both DPPH and FRAP. Jet cooking (JC) showed the lowest antioxidant ($p<0.05$) capacity. JC is a continuous process, with a short exposure time. As the main difference between jet cooking and jet cooking + hot water extraction is the time the sample was exposed to temperature, it could indicate that a longer time is needed to release the antioxidants and polyphenols from the matrix, or that these types of compounds are being produced by longer exposures to temperature, as indicated by Liu & Tsai (2012).

For all treatments, lower total polyphenol contents were found when compared to those reported by Gutierrez Barrutia et al. (2019), using water extraction at lab scale. Values for total polyphenol content of orange peels found in bibliography are extremely variable, with values ranging from 0.0139 mg GAE/g to 11.09 mg GAE/g in orange peels (Montero-Calderon et al., 2019).

Considering antioxidants and polyphenols are compounds fruits have to protect against environmental stress (Lado et al., 2018), these differences may be due to climate differences and ripening stages of the fruits.

3.5. Relation between sugar content, fibre content, antioxidant capacity and total polyphenol content

In order to establish correlations between sugar, total fibre contents, antioxidant capacity and total polyphenol content, Pearson correlation's test was performed. Results are shown on Table 3.

A high correlation was found on high molecular weight dietary fibre content and polyphenols content ($r = 0.990$, $p<0.01$). Also, total sugar content was negatively correlated with total fibre content ($r = -0.993$, $p<0.01$). This would indicate that when treatments are low in fibre extraction, they exhibit a higher content of glucose, fructose and sucrose. This could imply that the extraction treatments may break dietary fibre linkages more than desired, liberating sugars instead of oligosaccharides chains.

Moreover, a high correlation was found between DPPH and TPC, and FRAP and TPC ($r = 0.997$, $r = 0.995$, respectively, p-value<0.01). These results may indicate that the antioxidant capacity of the samples is strongly related to the presence of polyphenols.

Table 3. Pearson correlation table.

	HMWDF	LMWDF	TDF	Glucose	Fructose	Sucrose	Total sugars	TPC	DPPH	FRAP
HMWDF	**1**	0,983	**0,997**	-0,986	**-0,995**	**-0,994**	**-0,993**	**0,990**	0,978	0,971
LMWDF	0,983	**1**	**0,994**	-0,974	-0,984	-0,988	-0,983	0,955	0,938	0,927
TDF	**0,997**	**0,994**	**1**	-0,985	**-0,995**	**-0,996**	**-0,993**	0,980	0,966	0,957
Glucose	-0,986	-0,974	-0,985	**1**	**0,997**	**0,997**	**0,999**	-0,990	-0,989	-0,984
Fructose	**-0,995**	-0,984	**-0,995**	**0,997**	**1**	**1,000**	**1,000**	**-0,991**	-0,985	-0,979
Sucrose	**-0,994**	-0,988	**-0,996**	**0,997**	**1,000**	**1**	**1,000**	-0,988	-0,980	-0,974
Total sugars	**-0,993**	-0,983	**-0,993**	**0,999**	**1,000**	**1,000**	**1**	-0,990	-0,985	-0,979
TPC	**0,990**	0,955	0,980	-0,990	**-0,991**	-0,988	-0,990	**1**	**0,997**	**0,995**
DPPH	0,978	0,938	0,966	-0,989	-0,985	-0,980	-0,985	**0,997**	**1**	**1,000**
FRAP	0,971	0,927	0,957	-0,984	-0,979	-0,974	-0,979	**0,995**	**1,000**	**1**

Values in bold are significantly different from 0 ($p<0.01$)

4. Conclusions

Four different treatments were assayed on orange juice production by-product to obtain a soluble fibre enriched ingredient. Extrusion was the most effective treatment to extract soluble dietary fibre from orange pomace, having also the lowest sugar content. Regarding antioxidant capacity and total polyphenol content, extrusion yielded the highest values, while jet cooking treatment resulted in the lowest. Adding a hot water step after jet cooking increased the antioxidant capacity. This could be related to the formation of Maillard products, as reductones, in the most aggressive treatments (extrusion and jet cooking + hot water). Further investigation is required to elucidate the effect of jet cooking on dietary fibres.

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Conflicts of Interest

The authors have no competing interests to declare that are relevant to the content of this article.

Data availability statement

The authors confirm that the data supporting the findings of this study are available within the article.

Ethical guidelines

Ethics approval was not required for this research.

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Capítulo 2: Caracterización tecno funcional de los ingredientes obtenidos a partir del subproducto de la industria citrícola

Techno functional characterization of green – extracted soluble fibre from orange by-product

Perez-Pirotto, Claudia [a,b], Moraga, Gemma [b], Quiles, Amparo [b], Hernando, Isabel [b], Cozzano, Sonia [a], Arcia, Patricia [a,c]

[a] – Departamento de Ingeniería, Universidad Católica del Uruguay. Montevideo 11600, Uruguay.

[b] - Food Microstructure and Chemistry Research Group, Department of Food Technology, Universitat Politècnica de València, Valencia 46022, Spain

[c] - Latitud Latu Foundation. Montevideo 11500, Uruguay.

Correspondence to: Claudia Perez-Pirotto, Food Microstructure and Chemistry Research Group, Department of Food Technology, Universitat Politècnica de València, Valencia 46022, Spain, clpepi@upv.edu.es

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Abstract

This work studies the techno-functional properties of the spray dried water-soluble extracts from orange pomace, obtained from different green extraction technologies. Orange pomace, a by-product from the juice production industry, was used for soluble fibre extraction. Starting from pomace, four extraction procedures were assayed: hot water, extrusion + hot water, jet cooker and jet cooker + hot water. After treatments, the supernatant was spray dried with whey protein isolate to obtain soluble fibre enriched powders. Microstructure by field emission scanning electron microscopy, color, particle size, moisture content, hydration properties, foaming capacity and stability, rheology, and glass transition temperatures were assayed. All powders exhibited an orange shade. In microstructure, particle size, and glass transition temperature, the powder obtained from extrusion + hot water presented a different behavior from the other powders: less caking and agglomeration, and smaller particles. Also, this powder showed the highest foaming stability and viscosity, as well as glass transition temperature, properties that make it interesting for the food industry. Through valorization of orange pomace fiber-enriched powders could be obtained; these powders

can be used as ingredients in the industry to address the fibre gap and provide technological functionalities.

Keywords

Orange pomace; techno functional properties; soluble dietary fibre; microstructure.

1. Introduction

Dietary fibre is a class of compounds that includes a mixture of plant carbohydrate polymers resistant to digestion by human small intestinal enzymes (O'Keefe, 2019). The intake of these compounds has numerous documented health benefits, such as reduced risk of coronary heart disease, diabetes and obesity, among others. Also, fibre, especially the soluble type, is well reputed for its ability to lower blood lipid levels (Elleuch et al., 2011).

However, there is a worldwide shortage on its consumption, despite its health effects being well known. Although there is no consensus on recommended daily intake, both EFSA and FAO recommend a minimum intake of 25 g/day. Notwithstanding consumers' efforts to increase it, they are not reaching it (Santos et al., 2022). For example, the USA currently has an intake of 16g/day, while the UK has an intake of about 18 g/day (O'Keefe, 2019).

Dietary fibre is also appealing from the technological point of view, as it exhibits some properties that make it an interesting ingredient to add to products. Examples of these are water and oil holding capacity, increasing viscosity and emulsification, among others (Dhingra et al., 2012; Elleuch et al., 2011). Fibre has been added to various types of products, such as bread and sweet bakery products, as a substitute of wheat flour (Quiles et al., 2018). Studies have also been done with ice cream, where fibre played a cryoprotective role (Soukoulis et al., 2009). Fiber-enriched jams and meats have also been prepared, to avoid synaeresis and modify textural properties (Figueroa & Genovese, 2019; Elleuch et al., 2011;).

Thus, dietary fibre is an ingredient that may be added to foods, not only to improve its nutritional value, but also because of its technological properties. Furthermore, supplementing massively consumed foods with dietary fibre is a good approach to address the fibre gap. This would result both in an increase of the daily intake, as well as a decrease of the intake of other ingredients, such as fats and synthetic emulsifiers, due to fibre aforementioned technological properties. Also, as consumers prefer natural supplements, adding fibre is a good alternative to synthetic ingredients (Elleuch et al., 2011).

In this context, in recent years a trend to find new sources of fiber enriched ingredients has arisen (Majerska et al., 2019; Rodríguez et al., 2006). Fruit by-products, especially pomaces, are a good source of these ingredients. These could be used as a new product, or if processed, as a way of enriching food products with compounds that enhance their physical and chemical properties (Majerska et al., 2019). Citrus fruits are one of the top global agricultural commodities, with a global production in 2021/2022 estimated to be about 48.8 million tons of oranges (USDA Foreign Agricultural Service 2022). About 19 million tons of these are expected to be destined for processing, and around 50% w/w (wet weight) are seeds, peels and internal tissue (Wilkins et al., 2007), which means in 2021/2022 nearly 9.5 million tons of by-product will be produced. This waste has been reported as high in dietary fibre content and rich in several bioactive compounds, such as polyphenols and flavonoids (Gutiérrez Barrutia et al., 2019).

Several processes have been used for fibre extraction as a way of giving added value to by-products. These include not only chemical processes, but also enzymatic or physical processes. Current trends tend to look for extraction technologies without the use of solvents, the so-called “green technologies” (Chemat et al., 2012). Examples of these are enzyme assisted extractions in *Laminaria japonica* (Gao et al., 2017) and in flaxseed gum (Moczkowska et al., 2019), and steam explosion in sweet potato (Wang et al., 2017), in apple pomace (Liang et al., 2018), and in okara (Li et al., 2019). Ultrasound has also been used for fibre extraction flaxseed gum (Moczkowska et al., 2019) and combined with acid extraction for fibre extraction in papaya peel (Zhang et al., 2017). High hydrostatic pressures have been used for fibre modification in purple-fleshed potatoes (Xie et al., 2017). Extrusion has been extensively studied to improve the ratio IDF:SDF, for example in lupin seed coat (Zhong et al., 2019), in garlic skins (Guo et al., 2018) and in orange pomace (Huang & Ma, 2016).

The aim of this work was to evaluate the technological properties of soluble fiber enriched powder obtained from orange pomace applying different green extraction technologies, to be used as an ingredient in food fibre fortification.

2. Materials and methods

2.1 Soluble fibre enriched powders

Powders were obtained according to Perez-Pirotto et al., 2022, as presented on Figure 1.

Briefly, starting from orange pomace four different treatments were assayed in pilot scale equipment (hot water – HW, extrusion + hot water - EHW, jet cooker - JC, jet cooker + hot water - JCHW) to extract soluble fibre. After performing the different treatments, mixtures were centrifuged, supernatant was concentrated under vacuum and spray dried on a Buchi B290 spray dryer.

Encapsulation was necessary to overcome stickiness in drying chamber. For this purpose, whey protein isolate (WPI – Provon 292, Glanbia Nutritionals Inc.) was added (8% total solids basis) to the concentrate before drying and was kept under magnetic stirring at 60˚C until completely dissolved. After spray drying, powders were stored under vacuum at room temperature in the dark until use.

Chemical composition of powders was analysed on a previous study (Perez-Pirotto et al., 2022). Briefly, TDF content was 9.48 ± 0.45 g/100 g dwb for HW, 20.20 ± 3.69 g/100 g dwb for EHW, 10.25 ± 0.89 g/100 g dwb for JC and 13.03 ± 1.8 for JCHW. Total sugars content was 64.15 ± 0.2 g/100 g dwb in HW, 46.15 ± 0.05 g/100 g dwb in EHW, 64.24 ± 0.13 g/100 g dwb in JC and 60.37 ± 0.31 g/100 g dwb in JCHW.

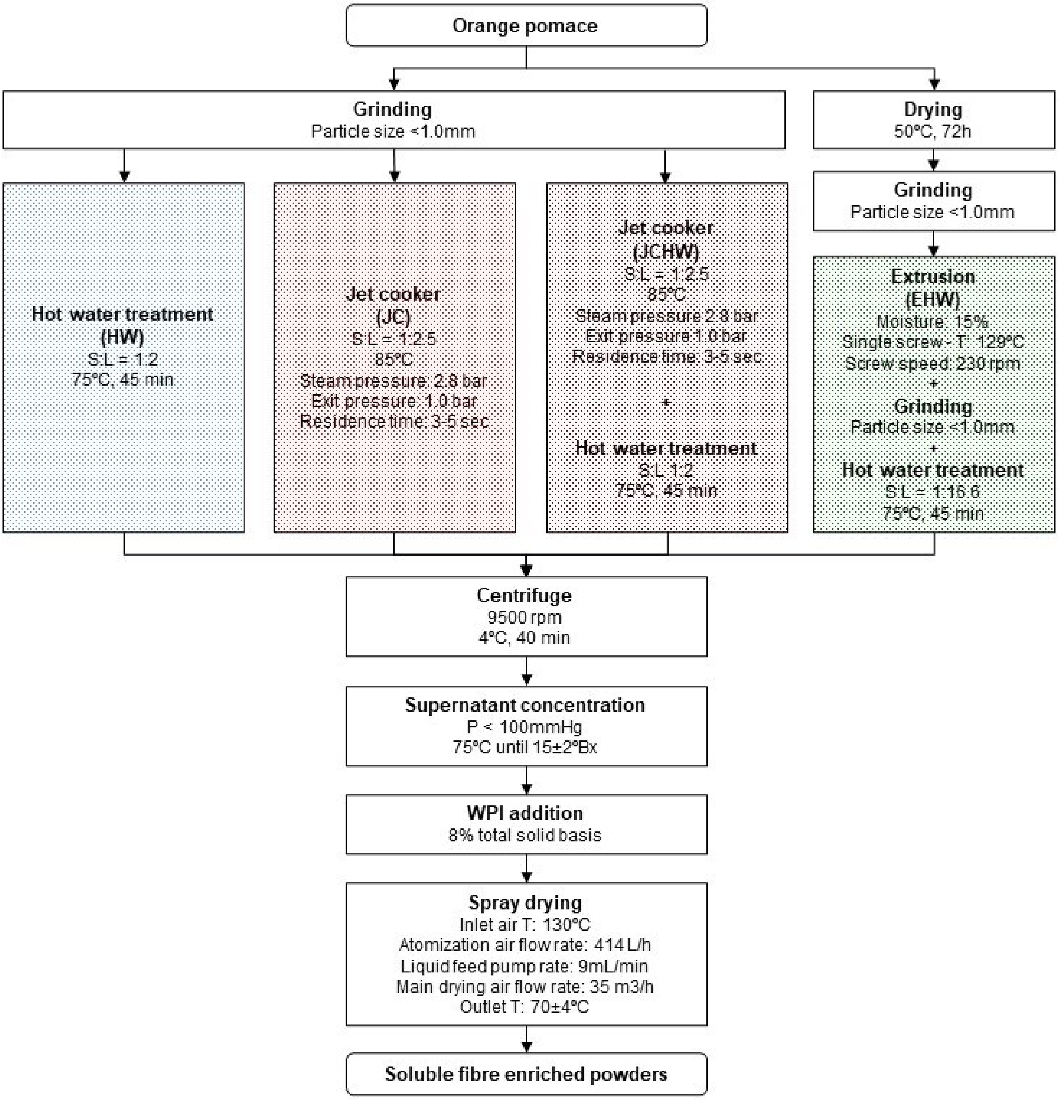

Figure 1. Treatments for fibre extraction

2.2. Microstructure

Samples were observed by Field Emission Scanning Electron Microscopy (FESEM). The sample was adhered to double-sided carbon tape and sputtered with a thin platinum layer. Samples were then photographed (Ultra 55 FESEM, Zeiss, Oberkochen, Germany) with an accelerating voltage of 1.5 kV.

2.3. Color

Powder color was measured with a LabScan XE (HunterLab, USA). Results were expressed as Hunter color values, L*, a* and b*. L* denotes lightness and darkness, a* redness and greenness, and b* yellowness and blueness. Chroma (ΔC) and Hue angle (º) were also calculated, following equations (1) and (2). Browning index, which is indicative of the brown color, an important parameter where browning reactions may take place, was calculated as suggested by Phuon et al., 2021, with equation (3).

$$Chroma = (a^{*2} + b^{*2})^{\frac{1}{2}} \quad (1)$$

$$Hue\ angle\ (H) = tan^{-1}(\frac{b^*}{a^*}) \quad (2)$$

$$Browning\ Index = \frac{[100\frac{(a + 1.75L)}{(5.645L + a - 3.012b)} - 0.31]}{0.17} \quad (3)$$

2.4. Particle size

A particle size analyzer (NanoPlus zeta potential and particle size analyzer, Particulate Systems, Atlanta, GA) was used to determine particle size distribution. Samples were suspended in distilled water and particle size was determined by light scattering. Distribution is expressed in terms of differential volume at a certain diameter. Also, D_{10}, D_{50} and D_{90} were calculated, where D_x represents the diameter for which x% of the particles have a smaller size. Span was calculated according to Fournaise et al., 2021.

2.5. Water content and hydration properties

2.5.1. Water content

The water content of the samples was obtained by vacuum drying the samples in a vacuum oven at 60˚C and a pressure < 100 mmHg, until constant weight.

2.5.2. Water solubility

Water solubility was assayed according to Wang et al. (2015). Briefly, 0.5 grams were accurately weighed (W) in a Falcon tube and 25 mL of distilled water were added. Tubes were vortexed at high speed for 30 seconds and left at 90°C for 30 minutes, with agitation, and afterwards centrifuged at 9000 rpm for 10 minutes. Supernatant was lyophilized and weighed (W_1). Water solubility was calculated according to the following formula:

$$WS\ (\%) = \frac{W_1}{W} \times 100 \quad (4)$$

2.5.3. Hygroscopicity

Hygroscopicity was evaluated following the method proposed by Oliveira et al. (2018). One (1.0) gram of sample was accurately weighed in glass Petri dishes, in duplicate (W_i), and placed in a hermetic container containing a glass with saturated NaCl solution, to get 75.5% humidity. This container was left at room temperature (20°C) standing for one week. Petri dishes were afterwards weighed (W_f) and hygroscopicity was expressed as grams of adsorbed moisture per 100 grams of sample, using equation (5):

$$Hygroscopicity = \frac{W_f - W_i}{\mathrm{W}} \times 100 \quad (5)$$

2.5.3. Oil holding capacity (OHC)

Oil holding capacity was performed according to Wang et al. (2015), with modifications. 0.5 grams were accurately weighed (W) in a previously weighed centrifuge tube. 5 mL of sunflower oil were added and mixed. Tubes were left standing at room temperature for 1 hour. Samples were centrifuged at 4200 rpm for 15 minutes. Sediment was weighed (W_1) and OHC was calculated according to the following formula:

$$OHC\ (\frac{g}{g}) = \frac{W_1}{W} \tag{6}$$

2.6. Foaming capacity

Foaming capacity was assayed following Dick et al. (2019) method, with some modifications. One (1.0) gram of sample was weighed in a flask and 50 mL of distilled water were added. Samples were kept overnight at 4˚C to ensure complete hydration. Volume was measured before whipping (V_b) Afterwards, the sample was homogenized with ultraturrax for 2 minutes at 10000 rpm and quickly transferred to the same graduated tube in which volume was previously measured. Volume of the foam was recorded (V_a). Foaming capacity (FC) was calculated according to the following formula:

$$FC(\%) = \frac{V_a - V_b}{V_b} \times 100 \tag{7}$$

For foam stability, foam volume was measured 5, 10, 30 and 60 minutes after whipping, and foaming capacity was calculated.

2.7. Rheology

Rheological properties were tested on a Rotational Rheometer (Kinexus Pro+, Malbera Panalyitical, Worcestershire, UK) equipped with a Peltier cartridge to control sample's temperature. Concentric cylinders were used.

Solutions at 8% (w/w) concentration were prepared the day before and left at 4˚C to ensure proper hydration of the samples. The day of the test, samples were removed from the refrigerator and left at room temperature to reach equilibrium.

Viscosity of the samples was analysed at 25˚C using shear rate from 0.1 to 200 s^{-1} during 180 seconds. Samples were allowed to reach equilibrium for 3 minutes before starting analysis.

2.8. Differential scanning calorimetry

Glass transition temperature was determined using Differential Scanning Calorimetry (DSC25 - TA Instruments, New Castle, Delaware USA). Around 10 mg of powder were accurately weighed in aluminum pans (Tzero pans – TA Instruments, New Castle, Delaware, USA) and sealed. Samples were equilibrated at 20˚C, cooled to -20˚C, and then heated to 60˚C at 10˚C/min in an inert atmosphere. An empty pan was used as a reference, and liquid nitrogen was used for sample cooling. Glass transition temperature was evaluated at the midpoint of the transition, using TRIOS Software (TA Instruments, New Castle, Delaware, USA).

2.9. Statistical analysis

Analyses were performed in duplicate. Results are expressed as mean ± SD. ANOVA analyses were performed, and differences between samples were determined by LSD Fisher test with p-value 0.05.

XLSTAT 2021.2.1 software (Addinsoft 2020, New York, NY, USA). was used for data processing.

3. Results and Discussion

3.1. Microstructure

After spray drying particles tend to be spherical, as this is the most stable droplet formation (Gagneten et al., 2019). Micrographs of the spray dried product are shown on Figure 2. In this case, particles are rather spherical, although some shrinkage is observed. Temperatures below 140°C render particles with wrinkled surfaces (Ferrari et al., 2012); so, taking into account drying operating conditions shrinkage may be due to drying inlet temperature.

In all cases, coalescence is observed: particles have agglomerated and formed greater irregular particles, as can be seen in Figure 2. However, this is more evident in the case of JC and JCHW (Figure 2E – 2H), and to a lower degree in HW and EHW powder (Figures 2A – 2D). In the latter case, the fusion of two or more particles is not as notorious as in the first cases: particles keep their individual shape and have not coalesced.

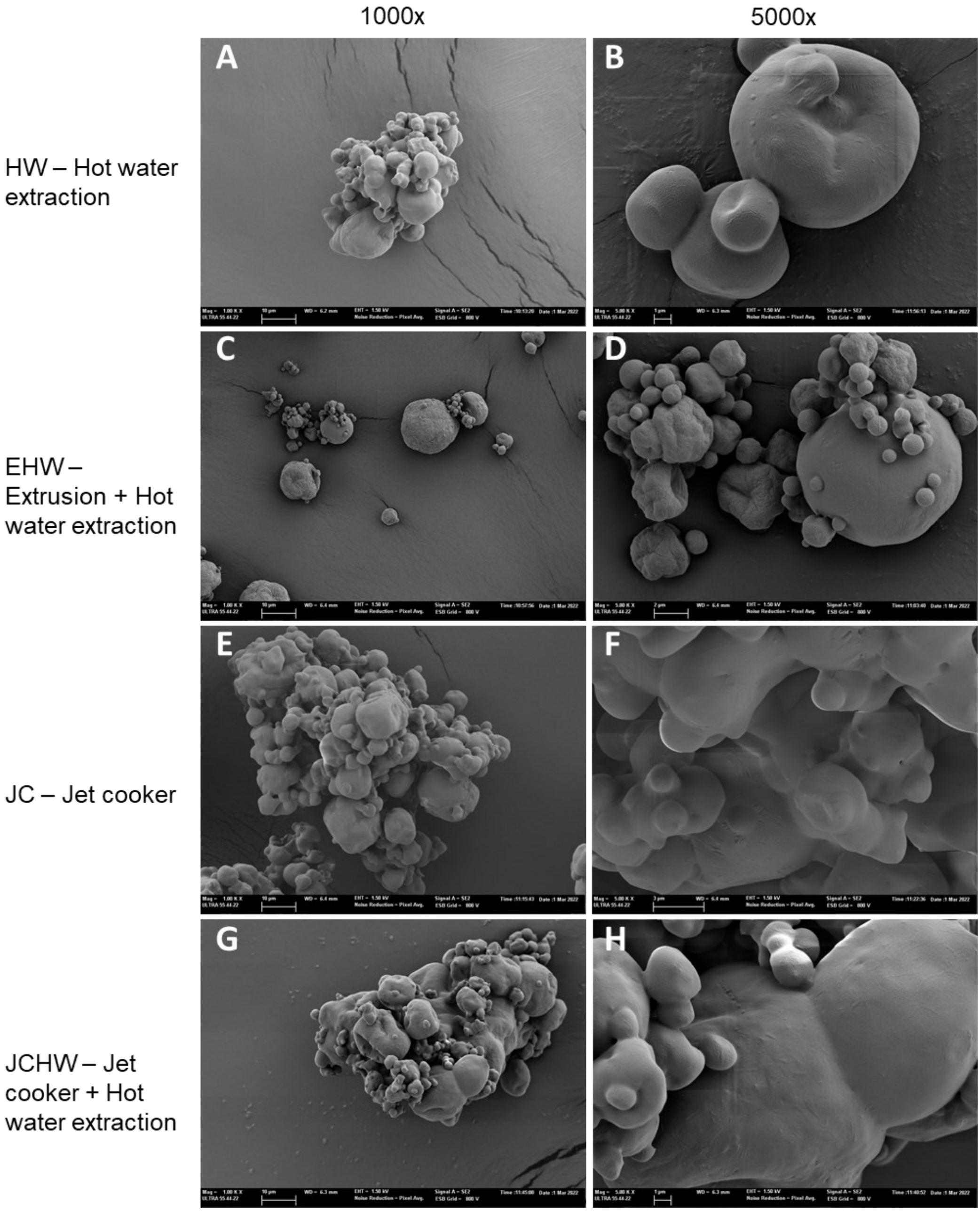

Figure 2. Microstructure of the spray dried powders observed by FESEM.

The fusion of particles is due to the liquid bridges established that connect particles with each other, which are formed using available moisture or absorbing the moisture from the environment (Edrisi Sormoli & Langrish, 2016). As this is part of the caking that is produced in sugars, EHW is expected to be the powder with the least fusion, as it is the one with the lowest sugar content (46 g/100 g dwb in EHW compared to 60 to 65 g/100 g dwb in the other samples, according to Perez-Pirotto et al., 2022).

3.2. Color

Color values obtained for the four powders are exhibited on Table 1. The powder obtained under the extrusion treatment (EHW) was the one with the darkest color as indicated by the lowest L* value. Also, the browning index for this powder is the highest and significantly different ($p<0.05$) from the others. Between the treatments assayed, the extrusion exposes the sample to the highest temperature, a condition that may favor non enzymatic browning reactions, such as caramelization or Maillard reactions. However, all browning indexes were lower than those observed by Phuon et al. (2021), for orange peels dried by different methods.

Table 1. Color properties of the different powders. HW: Hot water; EHW: Extrusion + hot water; JC: jet cooker; JCHW: jet cooker + hot water.

	L*	**a***	**b***	**Hue angle**	**Chroma**	**Browning index**
HW	81.07 ± 0.09^{b}	2.84 ± 0.02^{b}	24.13 ± 0.10^{b}	83.28 ± 0.07^{b}	24.30 ± 0.10^{b}	28.76 ± 0.21^{b}
EHW	78.46 ± 0.22^{a}	4.85 ± 0.17^{c}	24.41 ± 0.15^{b}	78.76 ± 0.37^{a}	24.89 ± 0.15^{b}	41.10 ± 0.32^{c}
JC	81.71 ± 1.11^{b}	2.58 ± 0.63^{b}	24.49 ± 1.15^{b}	84.03 ± 1.18^{b}	24.63 ± 1.20^{b}	37.24 ± 3.13^{b}
JCHW	84.84 ± 0.34^{c}	1.11 ± 0.17^{a}	21.00 ± 0.31^{a}	86.97 ± 0.43^{c}	21.03 ± 0.32^{a}	28.76 ± 0.73^{a}

Different letters in the same column indicate significant difference ($p<0.05$). Results are expressed as mean ± SD of two repetitions.

As a* and b* parameters are positive for all treatments, the four powders exhibit red and yellow shades, tending to have an orange tone. No significant differences ($p>0.05$) were observed on a* values between HW and JC. On b* values, the only significantly different from the others was JCHW. Therefore, although on yellow shades they are all similar, JCHW powder has a lower red component, which is also shown on the highest L* and Hue angle values.

3.3. Particle size

Particle size distribution is shown in Figure 3, and D_{10}, D_{50}, D_{90}, and span values are exhibited in Table 2.

Table 2. Particle sizes of the different powders. HW: Hot water; EHW: Extrusion + hot water; JC: jet cooker; JCHW: jet cooker + hot water.

	D_{10} (µm)	**D_{50} (µm)**	**D_{90} (µm)**	**Span**
HW	1.86 ± 0.47^{a}	2.63 ± 0.68^{b}	5.21 ± 1.29^{b}	1.28 ± 0.02^{d}
EHW	0.76 ± 0.15^{a}	1.10 ± 0.22^{a}	1.97 ± 0.33^{a}	1.11 ± 0.05^{c}
JC	6.32 ± 0.02^{b}	8.34 ± 0.06^{c}	12.15 ± 0.01^{c}	0.70 ± 0.00^{b}
JCHW	16.95 ± 0.98^{c}	21.17 ± 0.76^{d}	28.07 ± 0.39^{d}	0.53 ± 0.04^{a}

Different letters in the same column indicate significant difference ($p<0.05$). Results are expressed as mean ± SD of two repetitions.

As indicated by span values, HW and EHW gave more broader distributions, with a higher particle size range. JCHW gave the most homogeneous powder size, however it was the biggest one. Despite the difference on observed span values, all powders are mostly homogenous, as their span is around 1 in all cases.

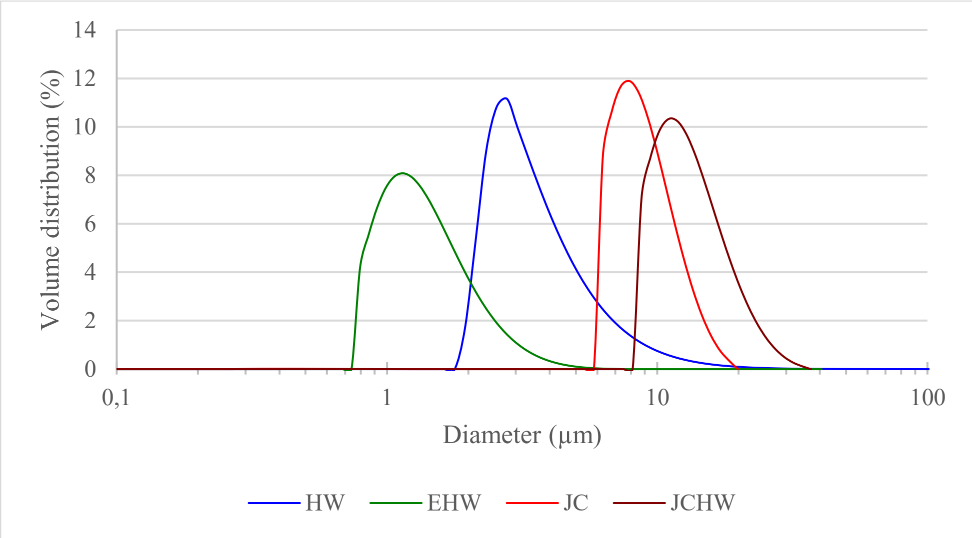

Figure 3. Particle size of the different powders. HW: Hot water; EHW: Extrusion + hot water; JC: jet cooker; JCHW: jet cooker + hot water.

As seen in section 3.1., JC and JCHW powders microstructure exhibit more caking than EHW and HW, which is clearly evidenced in sizes of the particles. Once they have established the liquid bridge, particles dissolute and form a bigger particle (Edrisi Sormoli & Langrish, 2016). In the case of EHW and WH, where aggregation is seen but not at such a degree, size is lower and also span values are higher, as each particle keeps its original size.

3.4. Moisture content, hydration properties and oil holding capacity

Moisture content is an important parameter to determine the powder stability during storage (Bhusari et al., 2014). It should be below 14% to prevent or minimize microbial growth and chemical deterioration (Mokhtar et al., 2018). All the powders had moisture contents below 6%, as shown on Table 3, being significantly lower ($p<0.05$) for EHW and JC, with no significant difference between them ($p>0.05$).

Hydration properties and oil holding capacity of the different powders are also presented on Table 3. In the three analyses (water solubility, hygroscopicity and OHC), the powder with the highest performance is EHW. As this powder has the smallest particle size (section 3.2)., its relationship surface/volume is higher, and the interface that is exposed to the surrounding environment (water, oil or moisture), is higher.

All powders show good solubility in water, being significantly highest ($p<0.05$) in EHW and lowest in the case of JCHW (Table 3). Water solubility is positively correlated with soluble fibre content, therefore it is expectable that the powder with the highest fibre content is the most soluble (Gu et al., 2020), in this case EHW with 20.2 g/100g total dietary fibre and JCHW with 13.0 g/100 g total dietary fibre (Perez-Pirotto et al., 2022).

Table 3. Moisture content, Water solubility, hygroscopicity and oil holding capacity (OHC) of different powders. HW: Hot water; EHW: Extrusion + hot water; JC: jet cooker; JCHW: jet cooker + hot water.

	Moisture content (%)	**Water solubility (%)**	**Hygroscopicity (g moisture/100 g sample)**	**OHC (g oil/ g sample)**
HW	4.99 ± 0.22^{b}	85.43 ± 0.21^{b}	$15.21 \pm 0.29^{a, b}$	3.12 ± 0.04^{a}
EHW	4.12 ± 0.03^{a}	90.44 ± 0.18^{c}	15.65 ± 0.35^{b}	3.41 ± 0.02^{b}
JC	4.42 ± 0.03^{a}	86.32 ± 1.42^{b}	$15.28 \pm 0.03^{a, b}$	$3.28 \pm 0.13^{a, b}$
JCHW	5.43 ± 0.35^{b}	83.04 ± 0.06^{a}	14.63 ± 0.18^{a}	3.36 ± 0.05^{b}

Different letters in the same column show significant difference ($p<0.05$). Results are expressed as mean ± SD of two repetitions.

Hygroscopicity is the ability of the powder to absorb moisture from the surroundings and cause stickiness (Wong et al., 2018). Higher values were observed in powders with the lowest moisture content. This is due to the gradient between moisture content of the surrounding environment with the powder.

OHC is important from a technological point of view, as it prevents fat loss during cooking and helps remove excess fat from foods (Gan et al., 2020). Also, it plays an important role in nutrition, where it can bind bile acids and increase their excretion, therefore helping reduce plasma cholesterol (Tosh & Yada, 2010). Oil holding capacity of samples can be seen in Table 3. Differences between samples may be related to fibre content, as higher fibre content renders a higher oil holding capacity (Wang et al., 2017).

3.5. Foaming capacity and stability

Foaming capacity and stability values can be seen in Figure 4.

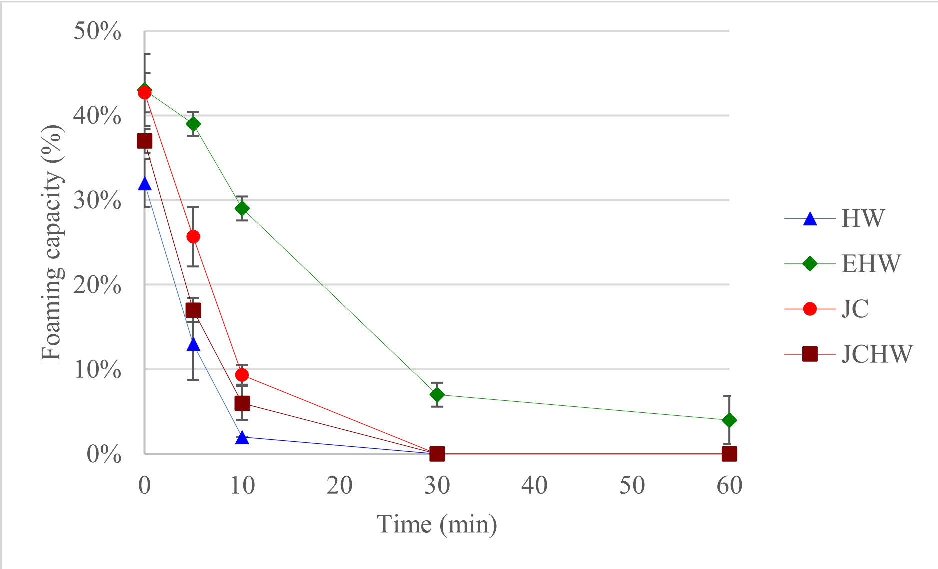

Figure 4. Foam stability of solutions. HW: Hot water; EHW: Extrusion + hot water; JC: jet cooker; JCHW: jet cooker + hot water.

Although at the beginning JC foaming capacity is similar to EHW, the latter is the most stable foam. All the other foams have disappeared after 30 minutes. As all powders have the same protein content (8 g/100 g, according to the WPI percentage used as encapsulating agent), differences in foaming capacity and stability could be due to polysaccharides content. By increasing viscosity, polysaccharides structure and stabilize lamellar water, retarding liquid drainage and air-bubbles coalescence. Also, they form a flexible film around the air bubbles, improving the foam formation (Dick et al., 2019). Therefore, EHW is expected to be the one with the most stable foam, as it is the one with the highest fibre content, and therefore, polysaccharides content, according to Perez-Pirotto et al., 2022.

3.6 Rheology

The four treatments exhibited a Bingham-like behaviour, Newtonian with yield stress. All models fit with r^2 values higher than 0.99. They all adjust to the equation $\sigma = \sigma_y + \eta_p \dot{\gamma}$, where σ_y represents yield stress and η_p is plastic viscosity. Viscosity values were significantly different ($p<0.05$) for the four of them. Results are shown on Table 4.

Highest viscosity and yield stress values were observed for EHW, which may be due to the higher amount of soluble fibre present in the sample (Perez-Pirotto et al., 2022). Dietary fiber, especially the soluble type, is well known for providing viscosity and changing rheological properties of the mixtures to which it is added. This thickening property of these compounds is related to the ability polysaccharides have to entangle physically with solution constituents. A positive and non-linear relationship exists between molecular weight of the fibres in the solution and the viscosity (Dikeman & Fahey, 2006). Therefore, extrusion seems to be the treatment that gives the fibres with the highest molecular weight and jet cooking the lowest ones. This could be due to the jet cooking breaking these to a higher extent than desired, ultimately liberating free sugars and decreasing the amount of soluble fibre (Perez-Pirotto et al., 2022).

Table 4. Yield stress and viscosity of the samples. HW: Hot water; EHW: Extrusion + hot water; JC: jet cooker; JCHW: jet cooker + hot water.

	σ_y (mPa)	η_p(cP)
HW	39.27 ± 0.27^a	6.02 ± 0.19^b
EHW	56.33 ± 1.82^b	25.46 ± 0.53^d
JC	37.76 ± 0.66^a	4.16 ± 0.03^a
JCHW	37.02 ± 0.25^a	7.13 ± 0.15^c

Different letters in the same column show significant difference ($p<0.05$). Results are expressed as mean ± SD of two repetitions

3.7 Differential scanning calorimetry

Thermograms obtained by DSC analysis are shown on Figure 5. In all cases there is a clear shift in the heat flow curve, related to a change in the specific heat of the sample associated to the glass transition.

Glass transition temperature is the temperature at which an amorphous system changes from a glassy state to a rubbery one. It is related to stability, as changes from glassy to rubbery state decrease viscosity and therefore increase molecular mobility (Daza et al., 2016). Glass transition values ranged from 3.49 ± 0.35 to 15.32 ± 0.40˚C, for HW and EHW, respectively. No significant difference was found between JC and JCHW glass transition temperature, which had a mean of 6.77 ± 0.85˚C ($p>0.05$). These values indicate that at room temperature all powders are in the rubbery state. Storing a product above its glass transition temperature may cause caking and agglomeration (Kaderides & Goula, 2017), which is clearly seen in the case of HW, JC and JCHW in Figure 2.

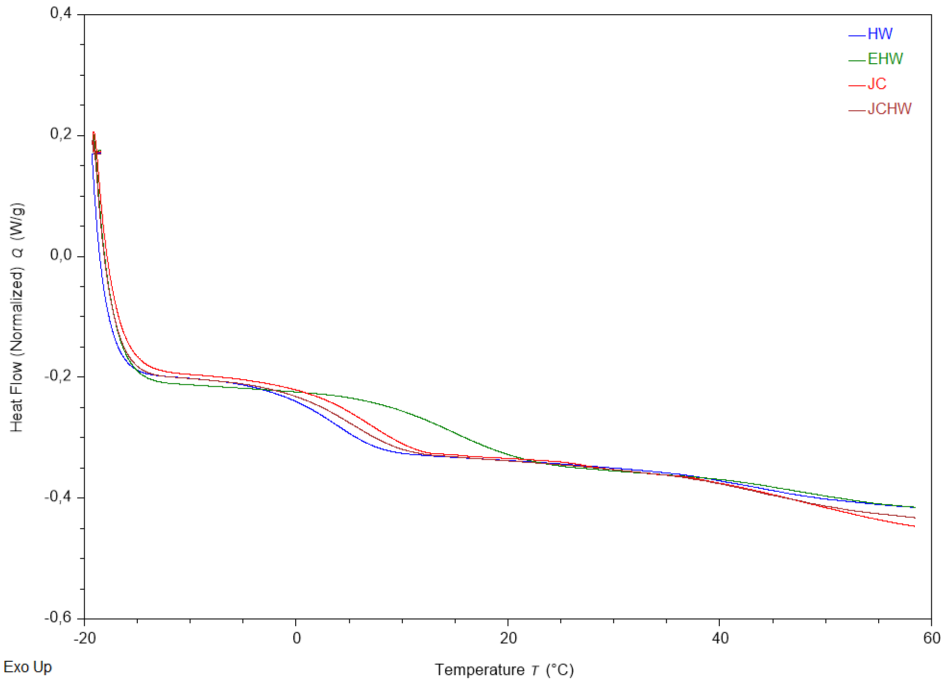

Figure 5. DSC analytical curve of different powders. HW: Hot water; EHW: Extrusion + hot water; JC: jet cooker; JCHW: jet cooker + hot water.

Glass transition temperatures of protein - sugars mixture have been extensively studied, and it has been found that measured *Tg* mainly reflects that of the sugars in the system, as described by Fang & Bhandari, 2012. Also, the differences in this parameter may be due to water content of the powders and molecular weight of its constituents (Moghbeli et al., 2020; Ahmed et al., 2010) . EHW has the highest glass transition temperature, as it is the powder with the least amount of sugars, lower moisture content and higher fibre content (Perez-Pirotto et al., 2022)

4. Conclusion

All products exhibit an orange shade and spheric microstructure, although caking is notorious in the case of HW, JC and JCHW. They all increase viscosity of water solution, while they do not change the behaviour of the solution (all Bingham fluids). Regarding foaming, they all exhibit foaming capacity, but only EHW provides stability over time. This powder is also the most interesting from hydration properties and glass transition temperatures, as it is the one with the highest OHC, water solubility and glass transition temperature. Through valorization of a by-product and applying different extraction technologies, it was possible to obtain soluble fibre enriched powders that can be used as ingredients in the food industry due to their techno functional characteristics.

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Conflicts of Interest

"The authors declare no conflict of interest."

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Capítulo 3: Estudio de la estabilidad en el almacenamiento a diferentes humedades de los ingredientes obtenidos a partir del subproducto de la industria citrícola

Sorption isotherms, glass transition and bioactive compounds of ingredients enriched with soluble fibre from orange pomace

Perez-Pirotto, Claudia [a, b], Moraga, Gemma [b], Hernando, Isabel [b], Cozzano, Sonia [a], Arcia, Patricia [a,c]

[a] – Departamento de Ingeniería, Universidad Católica del Uruguay. Montevideo 11600, Uruguay.

[b] - Food Microstructure and Chemistry Research Group, Department of Food Technology, Universitat Politècnica de València, Valencia 46022, Spain

[c] - Latitud Latu Foundation. Montevideo 11500, Uruguay.

Correspondence to: Gemma Moraga, Food Microstructure and Chemistry Research Group, Department of Food Technology, Universitat Politècnica de València, Valencia 46022, Spain, gemmoba1@tal.upv.es

Enviado a *Foods*

Abstract

Citrus fruits are one of the main crops worldwide. Its industrialization, primarily juice production, produces large amounts of by-products, composed of seeds and peels, that can be used to obtain new ingredients. In this study, sorption behaviour, glass transition, mechanical properties, colour and bioactives of four different soluble fibre enriched powders obtained from orange pomace using green technologies were studied. Powders were equilibrated at water activities between 0.113 and 0.680 for fifteen weeks at 20˚C, and studies were performed to indicate the best storing conditions to ensure the glassy state of the amorphous matrix and higher bioactive stability. By combining Gordon and Taylor model with Henderson isotherm, critical water activity and content for storage in the glassy state were determined. The ingredient obtained after extrusion + hot water is the most stable, which is also the one with the highest dietary fibre content. Powder obtained by jet cooking is the least stable, as it is not in the glassy state at any water activity at room temperature. To increase storage stability, these should be stored at refrigeration temperatures.

Keywords

Spray drying, water activity, mechanical properties, colour change, citrus by-products, powder stability.

1. Introduction

In last decades the interest in consuming foods with high content of dietary fibre has increased, as it has been linked to a lower prevalence of certain chronic diseases (Chau & Huang, 2003; O'Keefe, 2019). Following this trend, new sources of fibre are being investigated (Chau & Huang, 2003; Dhingra et al., 2012). Although cereals have a higher amount of dietary fibre, fruits' fibre has a better profile, with a higher share of soluble fibre. Citrus fruits are of special interest as their fibre has associated bioactive compounds, such as flavonoids and polyphenols (de Moraes Crizel et al., 2013).

Because of this particularity of citrus fruits, work has been done with orange peel waste, a by-product of the orange juice production (de Moraes Crizel et al., 2013; L. Huang et al., 2020; Quiles et al., 2018). As only 50% of the fresh fruit is used in juice production, and orange is one of the most cultivated fruits around the world, the amount of pomace produced is very big (Bussolo de Souza et al., 2018; Hervalejo et al., 2021). Also, in line with current trends, research has been done to improve the fibre profile of the orange pomace, with environmentally friendly technologies. This includes extrusion, steam use, hot water, high pressures, among others (Cameron et al., 2015, 2017; Garcia-Amezquita et al., 2019; Gutiérrez Barrutia et al., 2019; Perez-Pirotto et al., 2022; Tejada-Ortigoza et al., 2017; Wang et al., 2015). After treatments, soluble fibre can be extracted and dried to obtain a powder ingredient than can be used in the formulation of other foods (Perez-Pirotto et al., 2022).

A characteristic of fruits and ingredients derived from them is the high content of simple carbohydrates in their composition. As these have low molecular weight, their glass transition temperature is low. Therefore, at room temperature the matrix could be in the rubbery state, leading to stickiness, caking, and agglomeration in products (Mosquera et al., 2010; Mosquera et al., 2011; Mutlu et al., 2020). Also, these matrixes are very sensitive to small variations in temperature and moisture content, which are common in product storage (Moraga et al., 2012; Telis & Martínez-Navarrete, 2009). Hence, to prevent these changes that lead to losses in product quality, the matrix should be kept in the glassy state.

To predict physical, chemical, and microbiological stability changes, the knowledge of sorption isotherms may be very useful. These relate the water activity of the sample with its water content, at a given temperature. So, sorption isotherms are useful to determine the optimum conditions for storage, to preserve shelf life and for choosing appropriate packaging materials (Al-Ghamdi et al., 2020; Gabas et al., 2007). They are dependent on the structure and composition of the material, as well as temperature and pressure (Hawa et al., 2020; Mutlu et al., 2020).

By combining the isotherms with glass transition temperatures, the modified state diagram of the amorphous phase can be represented (a_w-moisture content-T_g diagram). With it a critical value of water content (CWC) and water activity (CWA) can be determined, depending on storage temperature. These values are important factors for the stability of the product (Moraga et al., 2012).

Several works studying this relationship have been done. Flores-Ramírez et al. (2022) have studied the effect of adding maltodextrin in a_w-moisture content-T_g relationship in freeze-dried juices. Work has been done too with dried honey and the effect of different carrier materials (Mutlu et al., 2020). Al-Ghamdi et al. (2020) have studied the aw-moisture content-T_g relationship in pumpkin, together with its color stability. González et al. (2020) studied this relationship in dried persimmon, and also the effect of glass transition in color and mechanical properties. This relationship has also been linked to chemical and physical properties, such as powder flowability or caking, as Cruz-Tirado et al. (2021) did on their work.

In this scenario, the aim of this work was to study the a_w-moisture content-T_g relationships for a fibre enriched ingredient, derived from orange pomace, obtained using novel extraction technologies. We pretended to determine the effect of glass transition on the mechanical properties and bioactive compounds during storage.

2. Materials and methods

2.1. Sample preparation

Orange fibre powders were obtained according to Perez-Pirotto et al. (2022). Four different treatments (hot water extraction – HW, extrusion + hot water extraction – EHW, jet cooker – JC and jet cooker + hot water extraction – JCHW) were assayed on orange pomace to obtain a soluble fibre enriched powder.

For extrusion, pomace was dried in a convection oven for 72 hours and milled to a particle size < 1.0 mm. Sample was extruded following Huang & Ma (2016) optimum conditions: moisture content of the sample 15%, 230 rpm and 129˚C in the three zones of a single-screw extruder. After extruding, sample was grinded again and a hot water extraction was performed, mixing it with water (1:16.6 m/v) and keeping it at 75˚C for 45 minutes under continuous agitation.

Hot water, jet cooking and jet cooking + hot water were performed on wet orange pomace after being grinded to a particle size < 1.0 mm. Hot water treatment was performed by mixing pomace with water (1:2 m/v) and kept at 75˚C for 45 minutes under continuous agitation. In jet cooking, sample was mixed with water (1:2.5 m/v) and the slurry was passed with a pump through the heating element, with a residence time between 3 and 5 seconds. Temperature was 85˚C and steam pressure 2.8 bar, while exit pressure was 1.0 bar. In jet cooking + hot water, the sample was heated at 75˚C for 45 minutes after coming out of the jet cooker, in a mechanically agitated boiler.

After recovering the supernatant from each treatment, it was concentrated under vacuum and spray dried on a spray drier (Buchi B290) with whey protein isolate as encapsulating agent (8% soluble solids). Drying conditions were 130˚C inlet air temperature, atomization air flow 414 L/h, liquid feed pump rate 35 m^3/h, and outlet temperature 70±4˚C. Powders were kept in laminated bags at room temperature until use. The composition of these powders has been previously studied by Perez-Pirotto et al. (2022). Briefly, results per 100 grams of dry sample were: total dietary fibre (TDF) 9.48 ±

0.45 g in HW, 20.20 ± 3.69 g in EHW, 10.25 ± 0.89 g in JC and 13.03 ± 1.8 in JCHW; total sugars content 64.15 ± 0.2 g in HW, 46.15 ± 0.05 g in EHW, 64.24 ± 0.13 g in JC and 60.37 ± 0.31 g in JCHW.

About 10 grams of each sample were placed in aluminum cups and stored in the dark in airtight containers with saturated saline solutions (LiCl, CH_3COOK, $MgCl_2$, K_2CO_4, $Mg(NO_3)_2$ and KI), with water activities (a_w) of 0.112, 0.230, 0.330, 0.430, 0.520, and 0.680, respectively (Greenspan, 1977). After fifteen weeks of storage, equilibrium moisture content was determined by weight difference (Flores-Ramírez et al., 2022). Initial water content was determined by vacuum drying in a vacuum oven at 60˚C until constant weight (Vaciotem, JP Selecta SA, Spain

2.2. Sorption isotherm

Water content data was fitted to Henderson, Caurie, and BET models. Nonlinear regression analysis was determined by statistical software Solver (Excel 2016). These models are predicted by equations (1) to (3), respectively.

$$w_e = 0.01\left[\frac{-\log(1-a_w)}{10^f}\right]^{1/n} \quad (1)$$

Where w_e is the equilibrium water content (g water/g solids), and *f* and *n* are characteristic parameters of the product.

$$w_e = exp\left[a_w \cdot ln(r) - \frac{1}{4.5 \ \cdot w_s}\right] \quad (2)$$

Where w_e is water content (g water/g solids), a_w is water activity, *r* is a characteristic constant of the material and w_s is the moisture security content that gives the highest stability to dehydrated product during storage.

$$w_e = \frac{w_o \ C \ a_w}{(1 - a_w)(1 + (C - 1)a_w)} \quad (3)$$

Where w_e is the equilibrium water content (g water/g solids), a_w is water activity, w_o is the monolayer moisture content (g water/g solids) and C is the sorption energy constant.

2.3. Glass transition temperature

Glass transition temperature of each powder at each water activity was determined by differential scanning calorimetry. About 10 mg of sample were placed in aluminum pans (Tzero pans – TA Instruments) and analysed using a DSC25 (TA Instruments). Heating rate was 10˚C/min, and the temperature ranged from -90˚C and 70˚C, depending on the sample's water content.

Nitrogen was used as the purge gas, with a rate of 40 mL/min. Mid-point temperature was considered the glass transition temperature.

Experimental data were fitted to Gordon & Taylor model, following equation (4):

$$\boldsymbol{T_g = \frac{(1 - x_w)T_g(as) + k\, x_w\, T_g(w)}{(1 - x_w) + k_w}} \quad (4)$$

x_w is the mass fraction of water (g water/g product), T_g is the glass transition temperature (˚C), $T_g(w)$ the glass transition temperature of amorphous water (-135˚C), $T_g(as)$ the glass transition temperature of anhydrous solids predicted by the model, and k is the model constant.

2.4. Colour analysis

Colour parameters were measured with a Colorimeter Minolta (Chroma Meter CR-400, Japan), using D65 illuminant/10˚ observer to obtain CIE $L^*a^*b^*$ colour coordinates. Total colour difference (ΔE^*) was calculated by Eq. (5).

$$\Delta E^* = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 (b^* - b_0^*)^2} \quad (5)$$

Where: L* is the luminosity, a* and b* the intensities of the green-red colour and the blue-yellow colour, respectively. The variables without subscript correspond to the equilibrated samples, while the others are from the lowest water sample (a_w=0.113) (Cruz-Tirado et al., 2021).

Chroma (C*) and hue angle (h*) were also calculated with equation (6) and (7).

$$C^* = \sqrt{a^{*2} + b^{*2}} \quad (6)$$

$$h^* = \tan^{-1} \frac{b^*}{a^*} \quad (7)$$

2.5. Texture analysis

A texture analyser (TA.XT2, Stable Micro Systems, Surrey, England), was used to measure hardness of the equilibrated samples. Using the compression mode, the force (N) required to penetrate the sample was measured. A 4 mm diameter, flat-tipped cylindrical probe was used. A penetration distance of 20 mm and descendent rate of 10 mm/s were used. Samples were placed in cylindrical cups and three samples per treatment per chamber were analyzed.

2.6. Bioactive characterization

Bioactive compounds were extracted with ethanol. Briefly, about 100 mg of sample were placed in centrifuge tubes, and 1.0 mL of ethanol 96% was added. Samples were vortexed and centrifuged at 10000 rpm for 10 minutes at 4˚C. After centrifugation, 0.9 mL were removed, and the same amount of fresh ethanol was added. After vortexing and centrifuging, the supernatant was combined with previously removed ethanol, and taken to a final volume of 10 mL with ethanol. Extracts were kept away from light at -18˚C until use.

Polyphenol content was determined by Folin Ciocalteu method. Antioxidant capacity was assayed using DPPH (Shah et al., 2016) and FRAP (Pulido et al., 2000) modified methods., as described by Perez-Pirotto et al. (2022).

2.7. Statistical analysis

All analyses were performed in duplicate. One way analysis of variance (ANOVA) was performed with XLSTAT 2022.1.2 (Addinsoft 2022, New York, NY, USA). A Tukey test was used to evaluate the significant differences ($p>0.05$) between samples.

3. Results

3.1. Sorption behaviour and glass transition

Three sorption isotherm models (BET, Henderson and Caurie) were fitted to the experimental data, and results are shown on Table 1.

Table 1. Sorption isotherms parameters

		HW	EHW	JC	JCHW
BET	w_o (g water/ g solids)	0.121	0.098	0.158	0.094
	C	1.240	1.785	0.756	2.356
	R^2	0.976	0.915	0.811	0.947
Henderson	f	-1.519	-1.571	-1.440	-1.666
	n	0.895	0.952	0.833	1.012
	R^2	0.997	0.996	0.997	0.995
Caurie	r	72.509	56.746	97.413	45.045
	w_s (g water/ g solids)	0.053	0.054	0.050	0.056
	R^2	0.962	0.970	0.953	0.975

HW: Hot water; EHW: Extrusion + hot water; JC: jet cooker; JCHW: jet cooker + hot water.

Only experimental data up to $a_w \leq 0.5$ were fitted to the BET model, as with higher values the model hypothesis failed. Brunauers' classification classifies sorption isotherms in four types according to their C value, a constant related to sorption energy (Brunauer et al., 1940). In HW, EHW and JC isotherms are type III, as C<2, commonly seen in products with relatively high fibre, sugar and protein content (Kaderides & Goula, 2017). This means these powders have low water adsorption until the water activity allows solubilization when adsorption increases. In JCHW, isotherm is type II, which is common in sugars and crystalline materials (Roos, 1995). This type indicates that the sample has an amorphous and porous structure and that sample's sorption consists of multilayer adsorption (Mutlu et al., 2020).

Monolayer moisture content (g water/ g solids) varied from 0.094 for JCHW to 0.158 in the case of JC. This value is interesting as it expresses the amount of water that is sufficient to cover the adsorbing surface with a layer of water molecules of the thickness of one molecule. Some authors have described it as an optimal water content for stability of some low moisture foods, as it is a critical product storage moisture content value to maintain physical, chemical, sensorial and microbial quality of dried foods (Mutlu et al., 2020; Roos, 1995).

Caurie and Henderson isotherms had the best fit to the data, as their R^2 are the highest. Caurie's w_s is the security water content, which has been considered as the one that would ensure the maximum stability to the dehydrated product during storage, as it related to the to the maximum water content that prevents an important increase in the rate of deteriorative reactions (Telis & Martínez-Navarrete, 2010). This parameter was lower than the monolayer water content (w_o) predicted by BET model in all cases.

The model that fitted best to experimental data was Henderson type. This model has been recommended for the description of the isotherms of polysaccharides materials (Stępień et al., 2020).

The variations in the environmental humidity imply changes in powders' water content and water activity, as modeled by the sorption isotherms. These variations can provoke the glass transition of the amorphous phase (Moraga et al., 2012). All samples showed glass transition in the analyzed temperature ranges. This relation between glass transition and water content can be modeled by Gordon and Taylor equation, as presented in equation (4). Table 2 shows the parameters of this model for the four powders.

Also, to obtain critical water content and activity values related to glass transition, the combined T_g-x_w-a_w data and the Henderson and Gordon & Taylor fitted models were used. Results are shown in Figure 1. In the cases of HW, EHW, and JCHW, powders could be stored at 20 ˚C, but in the case of JC at this temperature powder is already in its amorphous state.

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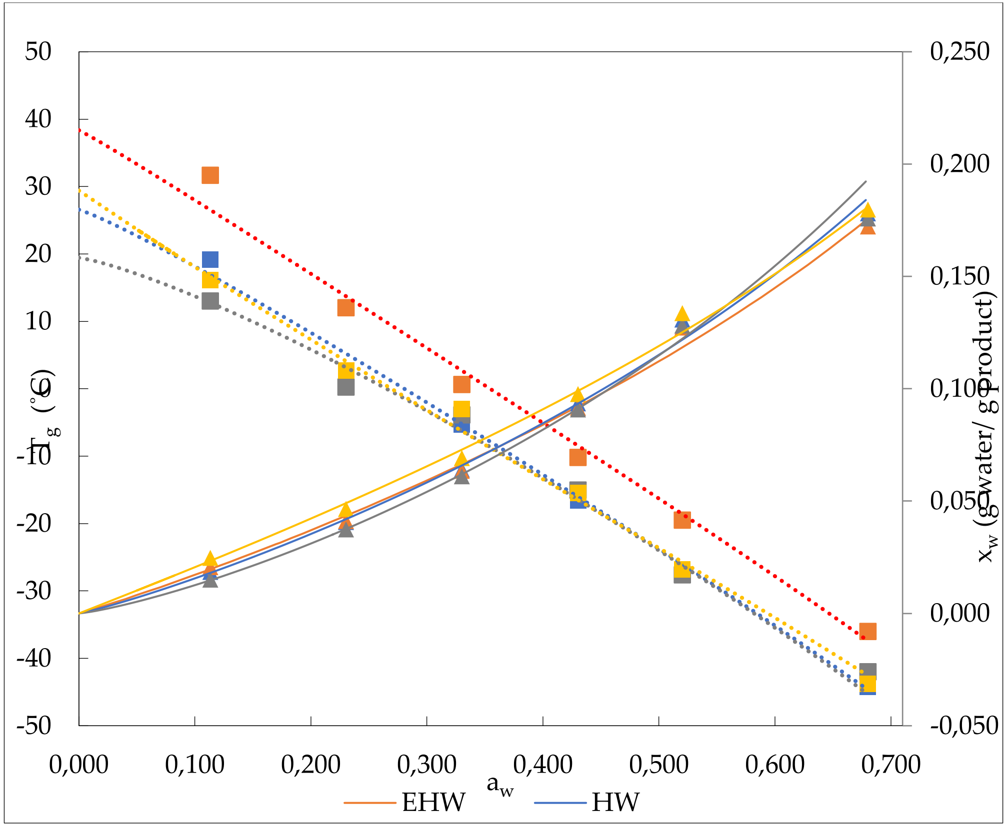

Figure 1. Temperature-water activity and water activity-water content relationship of different treatments. HW: Hot water; EHW: Extrusion + hot water; JC: jet cooker; JCHW: jet cooker + hot water. Experimental data (Tg ■ and xw ▲) and fitted models (Henderson and Gordon and Taylor models). Dashed line predicted with equation (1); continuous line predicted with equation (5).

Table 2. Gordon and Taylor model parameters and critical values of water activity and water content (g water/g product) related to glass transition at 5 and 20 ˚C

	Gordon and Taylor model			**Critical values at 20 ˚C**		**Critical values at 5˚C**	
	T_g (as)	*k*	R^2	CWC	CWA	CWC	CWA
HW	26.57	3.48	0.986	0.0120	0.080	0.0424	0.232
EHW	38.36	3.64	0.970	0.0315	0.173	0.0614	0.309
JC	19.43	3.01	0.989	-	-	0.0331	0.208
JCHW	29.40	3.52	0.988	0.0169	0.082	0.0471	0.222

HW: Hot water; EHW: Extrusion + hot water; JC: jet cooker; JCHW: jet cooker + hot water. Critical values were calculated at 20˚C and 5 ˚C in all cases. At 20 ˚C it was not possible to determine these for JC.

To keep the HW, EHW and JCHW powders in their glassy state at 20 ˚C, they must be stored in atmosphere with moisture lower than CWA, and their water content should be lower than the CWC. In the case of HW, the maximum relative humidity of the environment that would ensure this is 8.0%, and the product would need to have a maximum water content of 0.012 g water/g product. In the case of EHW, maximum relative humidity that would ensure glassy state is 17.3%, with the product having a maximum 0.0315 g water/g product. In the case of JCHW, the critical water activity was 0.082, so environmental humidity should be lower than 8.2%, and product's water content must be 0.0169 g water/g product maximum. In the three cases CWC was found lower than monolayer moisture content modelled by BET model

(w_o), which indicates that this value might fail to assure quality preservation during the storage of this type of products. This has also been seen in the case of freeze-dried grapefruits (Moraga et al., 2012) and freeze - dried persimmon (González et al., 2020). As reported by Roos (1993), some of the problems of dehydrated foods, such as collapse and stickiness, are more related to the glass transition than to the monolayer water content. Caurie's water security content (w_s) was also found higher than CWC at 20˚C in all cases, but the difference with predicted CWC is less pronounced than the one of the monolayer moisture content predicted by BET model.

At refrigeration temperatures (5˚C) it was possible to find CWC and CWA for all the powders. In this condition, powders would be allowed to have a higher water content and to be stored at higher water activities. Also, the storage of powders at refrigeration temperatures would slow down the rate of deteriorative reactions.

The observed differences in T_g predicted by Gordon and Taylor model can be due to the different composition of powders. As this parameter is mainly affected by water content and molecular weight, when keeping the former constant, the glass transition is expected to be decreased with lower molecular weights. According to Perez-Pirotto et al. (2022), JC is the powder with the highest amount of sugars and the lowest amount of dietary fibre, which would indicate its molecular weight is lower. The opposite happens in the case of EHW, with the highest T_g values.

3.3. Colour analysis

The parameters Lightness (L*), chroma (C*), Hue angle (h*) and colour differences (ΔE*) are presented in Table 3.

Lightness (L*) decreased with increasing water activity in all treatments, which indicates that samples were darker when water content was higher, as can be seen in Figure 2. Chroma (C*) values varied differently, as these were higher in water activities around 0.3 and 0.4. As chroma value represents the saturation or purity of the colour, obtained results show that colours were the purest around these water activities. At lower or higher values, colours tended to be less saturated.

Hue angle values in all cases increased with increasing water activity, showing colours tended to be more yellowish in lower water activities, and as water content increased the colour became more reddish. By combining these results, it is concluded that when water activity increases the powders tend to be more brownish (lower luminosity values and more reddish). This has also been seen in the case of pumpkin powder (Al-Ghamdi et al., 2020).

Table 3 – Colour attributes and color differences (ΔE* was calculated as the difference with the colour at a_w=0.113)

		0.113	**0.230**	**0.330**	**0.430**	**0.520**	**0.680**
L*	HW	82.39 ± 0.14^{Aa}	81.09 ± 0.21^{Aa}	77.02 ± 0.20^{Ba}	61.88 ± 0.84^{Cb}	39.83 ± 0.27^{Db}	34.73 ± 0.83^{Eb}
	EHW	74.74 ± 0.04^{Ad}	72.98 ± 0.24^{Bc}	69.33 ± 0.17^{Cc}	61.81 ± 0.56^{Db}	54.64 ± 0.76^{Ea}	39.54 ± 0.71^{Fa}
	JC	80.16 ± 0.05^{Ac}	79.38 ± 0.14^{Ab}	66.98 ± 0.67^{Bd}	54.51 ± 0.64^{Cc}	36.23 ± 0.45^{Dc}	32.54 ± 0.41^{Ec}
	JCHW	81.18 ± 0.12^{Ab}	79.43 ± 0.20^{Ab}	75.66 ± 0.30^{Bb}	68.36 ± 0.31^{Cd}	53.98 ± 1.69^{Da}	35.57 ± 1.19^{Eb}
C*	HW	20.90 ± 0.14^{Dc}	22.07 ± 0.13^{Cc}	25.06 ± 0.03^{Bb}	26.32 ± 0.18 ^{Aa}	20.54 ± 0.18^{Dc}	18.56 ± 0.45^{Ea}
	EHW	22.58 ± 0.05^{Da}	23.43 ± 0.17^{Ca}	25.45 ± 0.06^{Ab}	24.97 ± 0.41^{ABb}	24.61 ± 0.24^{Ba}	17.24 ± 0.15^{Eb}
	JC	22.13 ± 0.06^{Cb}	22.78 ± 0.02^{Cb}	26.35 ± 0.43^{Aa}	24.59 ± 0.55^{Bbc}	19.38 ± 0.32^{Dd}	16.60 ± 0.55^{Eb}
	JCHW	20.00 ± 0.10^{Cd}	21.09 ± 0.10^{Cd}	23.37± 0.06^{ABc}	24.15 ± 0.16^{Ac}	22.79 ± 0.54^{Bb}	15.77 ± 0.70^{Ec}
h*	HW	91.61 ± 0.08^{Ab}	90.25 ± 0.12^{Ab}	87.87 ± 0.13^{Ba}	82.09 ± 0.48^{Cb}	70.95 ± 0.72^{Dc}	68.79 ± 1.25^{Ea}
	EHW	81.82 ± 0.11^{Ad}	83.71 ± 0.12^{Bd}	81.61 ± 0.23^{Cc}	78.44 ± 0.07^{Dc}	74.47 ± 0.23^{Eb}	66.27 ± 0.24^{Fb}
	JC	90.64 ± 0.04^{Ac}	89.73 ± 0.04^{Ac}	83.96 ± 0.37^{Bb}	77.98 ± 0.85^{Cc}	68.13 ± 0.68^{Dd}	66.84 ± 0.20^{Eb}
	JCHW	91.81 ± 0.12^{Aa}	90.63 ± 0.04^{Ba}	88.10 ± 0.31^{Ca}	84.26 ± 0.33^{Da}	77.85 ± 0.13^{Ea}	67.18 ± 0.43^{Fb}
ΔE*	HW	-	1.84 ± 0.24^{Ea}	6.97 ± 0.15^{Db}	21.57 ± 0.87^{Cb}	43.21 ± 0.30^{Ba}	48.35 ± 0.89^{Ab}
	EHW	-	2.02 ± 0.18^{Ea}	6.28 ± 0.16^{Dc}	13.42 ± 0.47 ^{Cc}	20.65 ± 0.74 ^{Bc}	36.17 ± 0.69^{Ac}
	JC	-	1.08 ± 0.10^{Eb}	14.10 ± 0.46^{Da}	26.27 ± 0.63 ^{Ca}	44.75 ± 0.49^{Ba}	48.58 ± 0.44^{Aa}
	JCHW	-	2.11 ± 0.22^{Ea}	6.62 ± 0.30^{Db}	13.72 ± 0.43 ^{Cc}	27.84 ± 1.60 ^{Bb}	46.50 ± 1.15^{Ab}

HW: Hot water; EHW: Extrusion + hot water; JC: jet cooker; JCHW: jet cooker + hot water. Different small letters in the same parameter (L*, C*, h* and ΔE*) for the same aw indicate significant difference according to Tukey test ($p<0.05$) between treatments. Different capital letters in the same row indicate significant difference according to Tukey test ($p<0.05$) between water activities.

Colour changes were evaluated by the colour difference parameter (ΔE*). As all colour differences were higher than 1.5, these differences are perceptible to human eye (Obón et al., 2009). Non enzymatic browning is most likely to occur in low-moisture systems, due to the concentration of solutes being higher because of the low water content. In that situation, interactions between sugars and reducing amino acids are increased (Mosquera et al., 2011). However, as reported by Al-Ghamdi et al. (2020), this is higher when the matrix is in the amorphous state. Therefore, JC powder exhibited more color difference from lower water activities, probably due to the resultant browning. HW, JC and JCHW have a higher color difference from 0.520 water activity. Their water content was around 13g/100 g sample, well above the CWC at 20˚C, but similar to the 11% that promotes the enzymatic browning reactions according to Ling et al. (2005). In these cases, the colour changes seem to be related not only to the glass transition but also to the amount of water in the sample. Taking into account that above glass transition temperature the molecular mobility is higher and viscosity is lower, the reactions rate at higher water activities (with lower glass transition temperatures) is expected to be higher (Venir et al., 2007). Although colour difference is highest in all cases at a_w=0.680, the greatest increase is seen at that water activity only in EHW, probably due to its highest glass transition temperature.

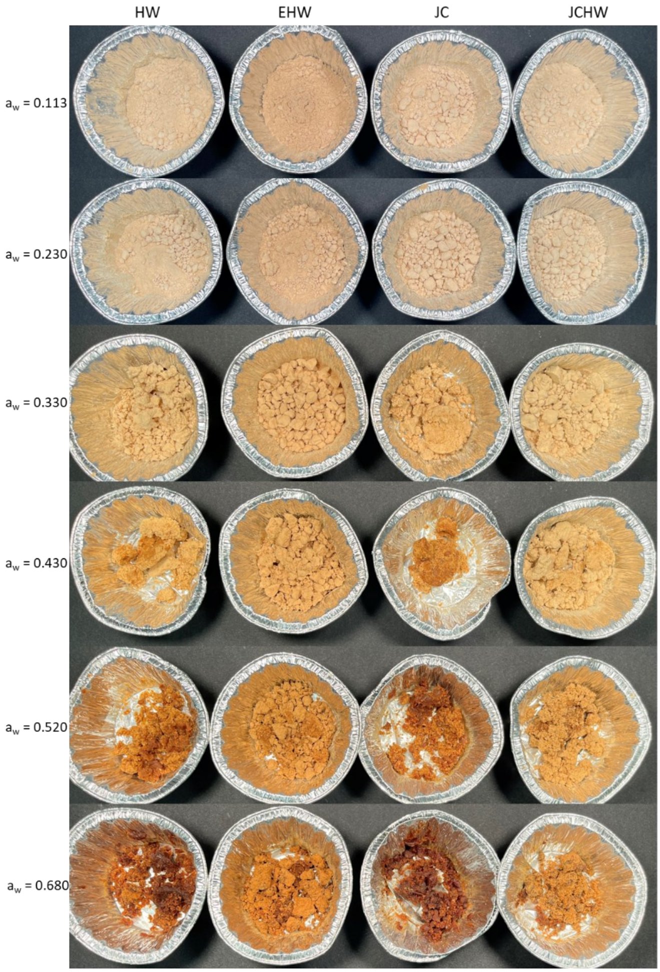

Figure 2. Powder photographs after three months of storage. HW: Hot water; EHW: Extrusion + hot water; JC: jet cooker; JCHW: jet cooker + hot water.

3.4. Texture analysis

Texture analysis results are shown on Figure 3. Lower water activities kept the powder looser, as can be seen in Figure 2. Higher water activities changed the product's appearance, a paste was formed which was softer as water content increased.

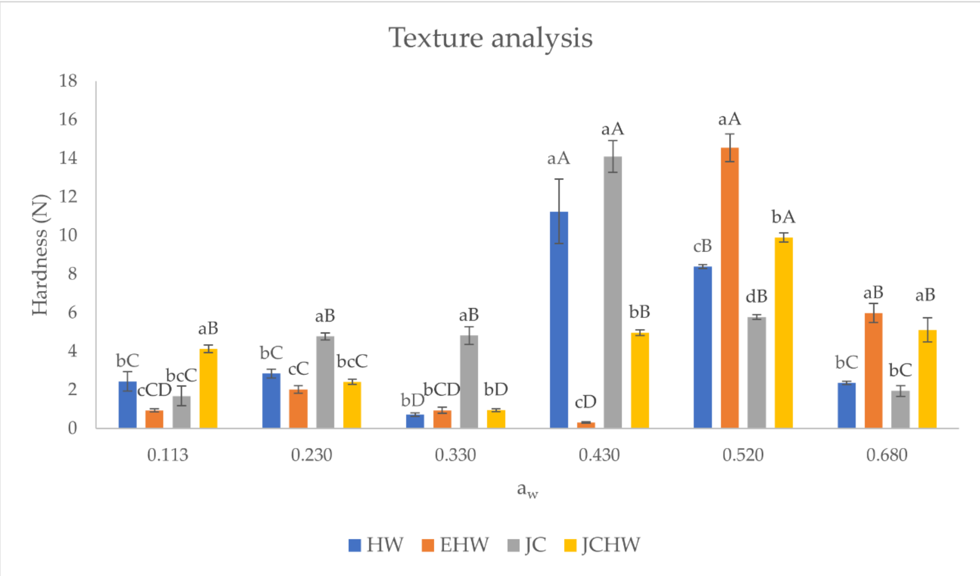

Figure 3. Hardness at different water activities. HW: Hot water; EHW: Extrusion + hot water; JC: jet cooker; JCHW: jet cooker + hot water. Different small letters for the same Aw indicate significant difference according to Tukey test ($p<0.05$) between treatments. Different capital letters in the same treatment indicate significant difference according to Tukey test ($p<0.05$) between water activities.

These changes in texture can be related to water content and crystalline state. When the environmental temperature approaches glass transition temperature water plasticizes the material and structure changes, collapsing and becoming denser. Also, as water content increases more liquid bridges between particles will be formed, increasing caking and sticking, as seen by Mosquera et al. (2011) in spray-dried borojó. This is because above glass transition temperatures molecules are able to rearrange from the glassy state to a very viscous, liquid-like state, allowing stickiness and viscous flow (Li et al., 2019).

The rate of caking is a function of T_g, time and relative humidity. As temperature difference T-T_g (ΔT) increases, the rate of caking is faster. When relative humidity is enough to keep the powder on its glassy state, no caking is observed. When relative humidity decreases glass transition temperature below storage temperature the caking begins. The rate of this phenomena will be higher if relative humidity is higher. At the beginning of caking the formation of inter-particle bridges will carry an increase in the force needed to compact the sample (Mosquera et al., 2011). This is what is seen in the case of JC and HW at a water activity of 0.430, while in the case of EHW and JCHW it happens when water activity reaches 0.520. These differences may be due to the differences in their composition (Perez-Pirotto et al., 2022), as JC and HW have a similar content of total sugars, and the lowest total dietary fibre content. On the other hand, EHW and JCHW have lower amounts of total sugars and higher dietary fibre contents, which implies their molecular weight is higher (Perez-Pirotto et al., 2022). Telis & Martínez-Navarrete (2009) found that the decrease in maximum force in texture analysis of grapefruit powder was dependent on the type of sugar added, and it happened at higher water activities when low dextrose equivalents maltodextrin or gum arabic were added, in comparison to high dextrose equivalents maltodextrin. This would indicate that in the case of higher molecular weight, the decrease in maximum force would happen at higher water activities.

Once caking is fully developed, the force needed to compact the sample will be lower, as sample will be completely liquefied (Mosquera et al., 2011). As the rate of caking is dependent on the temperature difference ΔT, it is expected that on higher water activities, where T_g is lower and therefore ΔT is higher, caking is fully developed, and samples are liquified (Telis & Martínez-Navarrete, 2010). These changes would be minimized if the samples were stored at refrigeration temperatures.

When relating texture changes to color changes, it seems mechanical properties have place before the highest color change, as in HW and JC the highest hardness is seen at aw=0.430, and in EHW and JCHW it is seen at aw=0.520. These samples exhibit the maximum colour difference increase at aw= 0.520 and aw=0.680, respectively. This was also seen by Telis & Martínez-Navarrete (2010).

3.5. Bioactive characterization

The total polyphenol content and antioxidant capacity of the four samples stored at each water activity are shown in Figures 4 and 5.

Total polyphenol content and antioxidant capacity (both in FRAP and DPPH) followed the same trend: EHW was the powder with the highest polyphenol content and antioxidant capacity at all water activities. According to Perez-Pirotto et al. (2022) this powder is the one with the highest dietary fibre content and may have a higher amount of polyphenols bonded to it.

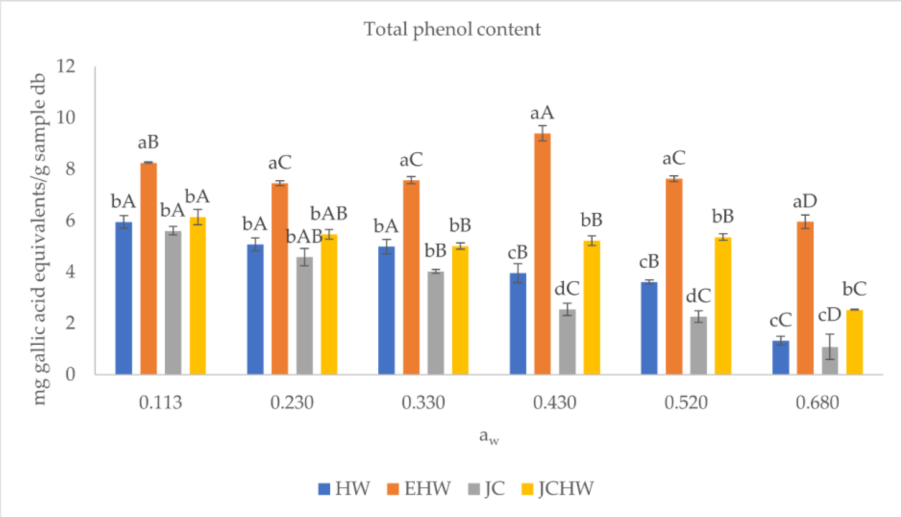

Figure 4. Total polyphenol content at different water activities. HW: Hot water; EHW: Extrusion + hot water; JC: jet cooker; JCHW: jet cooker + hot water. Different small letters for the same aw indicate significant difference according to Tukey test ($p<0.05$) between treatments. Different capital letters in the same treatment indicate significant difference according to Tukey test ($p<0.05$) between water activities.

In the case of EHW, the highest polyphenol content was observed at a water activity of 0.430. This maximum value may be related to a higher extractability of phenolic compounds, as observed by Maldonado-Astudillo et al. (2019), who found that at higher water activities phenolic compounds can be released, due to the matrix swelling and dissolution. The decrease seen at higher water activities (0.520 and 0.680) may also be due to an increase in degradation of these compounds.

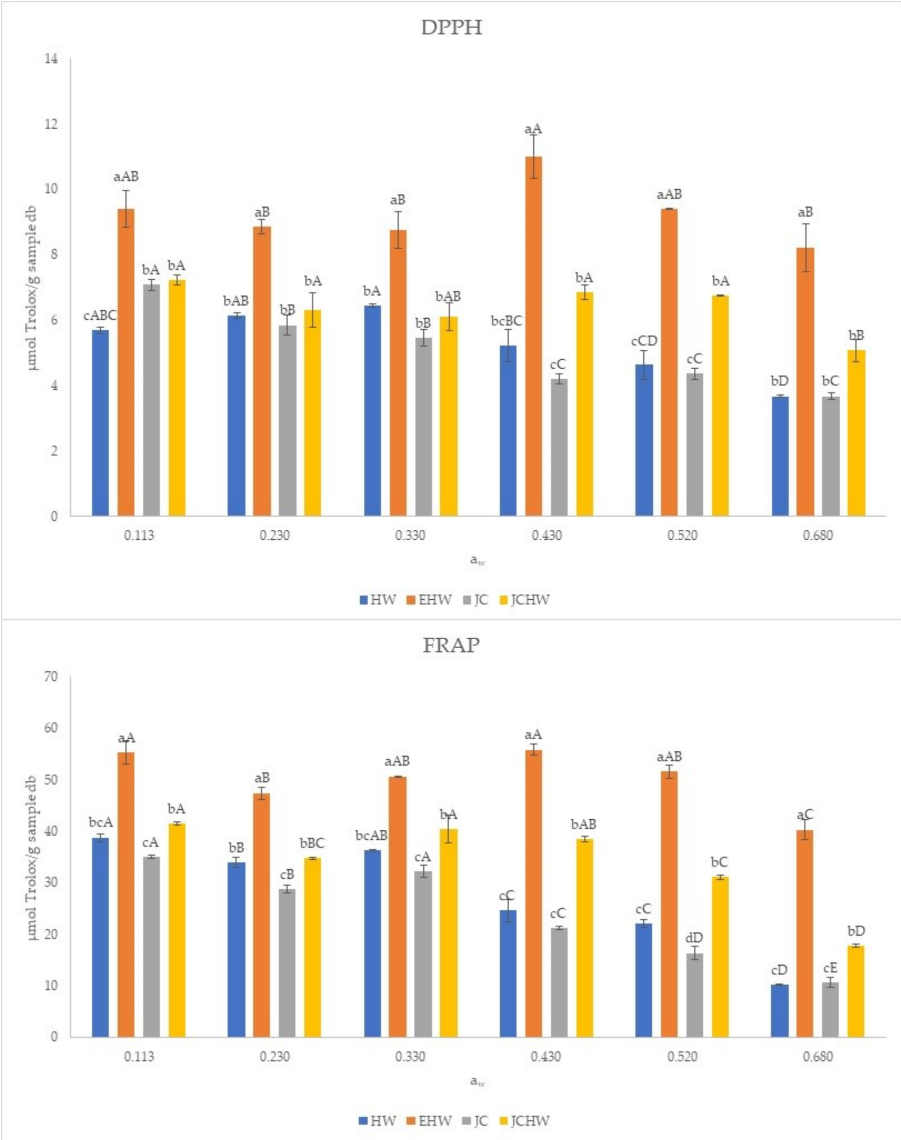

Figure 5. Antioxidant capacity of the samples (DPPH and FRAP) at different water activities. HW: Hot water; EHW: Extrusion + hot water; JC: jet cooker; JCHW: jet cooker + hot water. Different small letters for the same aw indicate significant difference according to Tukey test ($p<0.05$) between treatments. Different capital letters in the same treatment indicate significant difference according to Tukey test ($p<0.05$) between water activities.

Regarding antioxidant capacity, the same trend is observed. However, in the case of EHW and JCHW no significant difference was observed between the values for a_w 0.113 and 0.430 ($p<0.05$). EHW and JC are the ones with the most extreme thermic treatment, which could result in the formation of melanoidins and reductones from Maillard reactions, with antioxidanty capacity (Perez-Pirotto et al., 2022)

In HW and JC the stability of bioactive compounds (polyphenols and antioxidants) is lower, as a decrease is observed on antioxidant capacity and total phenol content when increasing water content. This was also reported in grapefruit powder after storage by Moraga et al. (2012), who found a negative linear correlation between antioxidant capacity and relative humidity.

In HW and JC cases, the decrease in antioxidant capacity and polyphenol content is more remarkable from a_w=0.430, where products have a moisture content of near 10 g/100 g sample. Until aw=0.330, products' moisture content (below 7g/100 g sample) was near the w_s predicted by Caurie's model. At higher water activities, moisture content is much higher than security water content, and the bioactive compounds are more affected. This decrease may be related to their lower stability, as they are the products with the lowest CWA and CWC.

4. Conclusions

The results of this investigation indicate that the properties and stability of the obtained powders depend on the extraction method used. The stability parameters confirm the problem of these kind of products, in which the high specific surface that can adsorb water makes the product very hygroscopic. This leads to collapse and bioactive compound loss.

To avoid these issues, it is necessary to ensure the matrix is in the glassy state throughout storage. However, in these cases the critical water activity and content that would ensure this state are extremely low, being the highest in the case of extrusion + hot water treatment. In this sample, with the highest fibre content, the changes in texture and color, and the bioactive compounds loss are less than in the other samples.

To increase the stability of these ingredients, especially the ones obtained by the other extraction procedures, samples should be stored at refrigeration temperatures.

The obtention of powdered ingredients from orange pomace is a way of adding value to the citric production chain. However, investigation is needed to test the possible application in food formulation.

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Conflicts of Interest

“The authors declare no conflict of interest.”

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Capítulo 4: Percepción de los consumidores. Incorporación a un postre tipo flan

Towards halving food waste: a comparative study using orange juice by-product in dairy desserts

Perez-Pirotto, Claudia [a, b], Hernando, Isabel [b], Cozzano, Sonia [a], Curutchet, Ana [a], Arcia, Patricia [a,c]

[a] – Departamento de Ingeniería, Universidad Católica del Uruguay. Montevideo 11600, Uruguay.

[b] - Food Microstructure and Chemistry Research Group, Department of Food Technology, Universitat Politècnica de València, Valencia 46022, Spain

[c] - Latitud Latu Foundation. Montevideo 11500, Uruguay.

Correspondence to: Patricia Arcia, Latitud Latu Foundation, Av. Italia 6201, 11500, Montevideo, Uruguay. E-mail: parcia@latitud.org.uy

Enviado a *Heliyon*

Abstract

By-products upcycling to produce ingredients has increased in the last years. However, the perception of foods with these ingredients must be studied to find the proper way to inform the consumer. The aim of this work was to study the purchase intention and healthiness and environmental friendliness perceptions of a flan dessert, enriched with orange juice by-product fibre. The effect of different product categories (ready-to-eat, powdered form, homemade type), fibre origin claim, and the addition of a sustainability logo in the packaging were studied through an online survey carried out in Spain (n=342) and Uruguay (n=307). Data were analysed by PLS and cluster analysis. Both Spanish and Uruguayan participants considered the product category the most important attribute in purchase intention and in healthiness and environmental friendliness perception, being the homemade product preferred by both. Logo presence was more important for purchase intention and perceived environmental friendliness, while information about fibre origin was more important for healthiness perception. The homemade product with a logo and the claim about fibre origin was the one that had the highest perception of being healthy and environmentally friendly in both populations.

Logo presence affected positively purchase intention for both groups, but the relative importance was higher in the case of Spanish consumers. The allegation "source of fibre" also increased healthiness and environmental friendliness perceptions, although not the purchase intention in Spain. Cluster analysis identified different groups of consumers on each country, who gave different relative importance to each attribute in purchase intention.

Keywords

Cluster analysis; survey; healthiness and environmental friendliness perception

1. Introduction

With the increasing concern over health and wellness, consumers are demanding natural products (Lunardo & Saintives, 2013). In the recent decades people have a strong preference for natural foods, willing for fresh, natural, and minimal processed foods. However, what is a natural food? Surprisingly, this is more influenced by the process in which the product is obtained, rather than by its content itself (Román et al., 2017). Moreover, foods are normally considered as healthy because of external factors, such as the product category, healthy descriptors, and labels (Hagen, 2021).

Melendrez Ruiz et al., 2021 studied the perception of differently processed pulses and found that the degree of processing of the product was the factor influencing the most its image. Raw products were perceived as natural and good for health, while the processed type (canned or ready to eat) were perceived as bad for health. Curutchet et al., 2022 found that fibre enriched bread and pasta were perceived as healthier than the ones without added fibre and were therefore more buyable.

Consumers are also becoming more interested in environmental sustainability (van Bussel et al., 2022). When it comes to clothes shopping, consumers seem to be aware of the impact of its production and prefer more environmentally friendly clothes, such as organic cotton (Hasan et al., 2022). On electronics, work has been done too to reduce waste, and sustainable options are being developed, as an eco-friendly alternative (Liao & Chuang, 2022). In the case of food, the effect of packaging type of foods and drinks in environmental friendliness perception has been studied, for example in plastic bottles (Galati et al., 2022) and in active packaging (Yan et al., 2022). However, knowledge regarding environmentally friendly food consumption is rather low. Because of the evident impact of meat production in environment, work has been done with the meat analogue's environmental friendliness perception (Hartmann et al., 2022). Lazzarini et al. (2016) studied the relationship between the perceived environmental friendliness and the life cycle assessment (LCA) of different protein products, such as meat, cheese, and plant-based products, and found that these are positively correlated. Hartmann et al. (2021) found that consumers with higher knowledge of environmental impact of foods and food production can choose more environmentally friendly menus (measured by eco points) than those with lower knowledge. To the best of our knowledge, environmental perception of other kind of foods has not been yet assessed. It seems that, as with naturalness, one of the factors affecting mainly this perception is the way in which food is produced, rather than the food itself.

Recently, United Nations have committed themselves to halving food waste as one of the sustainable development goals. One of the ways of helping reduce food waste is broadening what is normally considered as edible, such as peels and seeds of fruits (Aschemann-Witzel et al., 2022). These foods are normally described as "upcycled foods": those that produce ingredients that would be otherwise wasted, and that have benefits both for environment and society (Spratt et al., 2020). In this way, oranges' peels and seeds, that are normally discarded or used as animal feeding, may be upcycled to obtain dietary fibre. There is a worldwide shortage on its consumption, which could be addressed by supplementing massively consumed foods with this ingredient. However, its use must be conveniently communicated to consumers, to help increase the consumption of these type of foods. Plasek et al., 2021 found in their study that one of the main factors affecting healthy perception is the presence of an ingredient's claim. Also, Curutchet et al., 2021 found that the acceptability of an apple fibre enriched cake was higher when informing consumers about the fibre's origin. Grasso & Asioli, 2020, who studied the use of defatted sunflower cake flour as an upcycled ingredient in the formulation of biscuits, found that although the knowledge of upcycled ingredients is rather low among consumers, they would consider buying biscuits formulated with this kind of ingredients.

In this context, the aim of the present study was to study Uruguayan and Spanish consumers' perception of healthiness and environmental friendliness of different types-of dairy desserts (ready-to-eat, powdered form, homemade type) enriched with fibre obtained from an upcycled food, as well as their purchase intention. The main research questions were the following:

(1) Is the upcycled fibre perceived in the same way in foods with different processing levels?

(2) How does the fibre origin information affect purchase intention?

(3) Is the logo chosen to communicate about sustainability perceived as such by consumers?

(4) Is purchase intention related to perceived healthiness and environmental friendliness?

2. Materials and methods

The study consisted in evaluating the effect of information related to fibre enrichment origin and the information about sustainability, on different categories of a same product. We took into consideration the fact that effect of food processing might strongly influence consumer perceptions. According to Melendrez Ruiz et al. (2021), consumers mental representation of foods is dependent on the level of processing they have. Therefore, in the present study three different categories related to its processing level were considered: a home-made flan, an industrial powder product to prepare flan at home and an industrial ready-to-eat flan. As many authors reported that brand credibility is positively related with purchase intention of food (Ares et al., 2018; Sekhar et al., 2022), and that is even more important than sensory characteristics on consumer liking for food (Fernqvist & Ekelund, 2014), brand effect on consumer perception was also considered. Leader brands in both countries were used.

The study was conducted through an online survey where images of each category product with different information about fibre enrichment origin and information about sustainability were presented to consumer for its evaluation.

2.1 Product stimuli

For the product evaluation, images of each category product were presented next to information regarding the origin of fibre enrichment (variable 1, levels

0 and 1), and a sustainability logo (variable 2, levels 0 and 1), resulting in a total of 4 images for each category product. In all cases, the claim "source of fibre" was included next to the image. Table 1 shows the experimental design of the study.

Table 1. Experimental design: information presented in the image of the label for each category product.

Home-made flan	**Ready-to-eat flan**	**Powder preparation**
Source of fibre	Source of fibre	Source of fibre
Source of fibre With orange fibre	Source of fibre With orange fibre	Source of fibre With orange fibre
Source of fibre Sustainability logo	Source of fibre Sustainability logo	Source of fibre Sustainability logo
Source of fibre With orange fibre Sustainability logo	Source of fibre With orange fibre Sustainability logo	Source of fibre With orange fibre Sustainability logo

Figure 1. Example of product's image.

2.2 Online survey

Data collection was conducted using Qualtrics (Qualtrics, Provo, UT) between March and April 2022, both in Uruguay and in Spain. Participants were recruited via social media channels. Their informed consent to participate in the study was collected before beginning the survey, where they were informed that all information was being collected for investigation purposes and that it was confidential.

Consumers perception of environmental friendliness and healthiness, and purchase intention of all products shown in Table 1 were assessed. The images were presented one by one to participants, following a Williams' Latin square design. They were asked to look at the images and to rate their perception of environmental friendliness and perceived healthiness using a 7-point scale (1: not at all friendly, 7: completely friendly; and 1: not at all healthy, and 7: completely healthy, respectively), and purchase intention using a 7-point scale (1: I would definitely not buy it, 4: maybe yes, maybe no, 7: I would definitely buy it). Also, frequency of dairy desserts consumption was evaluated, and finally, consumers were asked about sociodemographic characteristics (age, gender, education level, income level).

G*Power version 3.1 was used to calculate the minimum sample size. According to these, a sample size of 246 participants was needed, to detect a medium sized effect (f = 0.25; power > 0.95). Surveys were completed by 342 participants in Spain and 307 in Uruguay. Social demographic information is presented in Table 2.

Table 2. Social demographic description of participants of each country.

		Frequency (%)	
		Spain	Uruguay
Sex	Feminine	53.8	66.1
	Masculine	42.4	33.6
	Prefers not to say	3.8	0.3
Age	17-29	56.4	26.1
	30-44	26.3	31.9
	45-60	14.9	25.4
	>60	2.3	16.6
Household income	Medium-low	78.7	40.1
	Medium-high	21.4	59.9
Educational level	Primary	0.6	2.9
	Secondary	15.2	5.9
	Third level (non-degree)	9.1	16.6
	Third level (degree or higher)	75.2	74.6
Dairy dessert consumption	More than once per week	48.3	28.3
	More than once per month	32.8	38.4
	Less than once per month	14.3	22.5
	Never	4.7	10.8

2.3 Data analysis

Pearson correlation tests (p-value 0.05) were performed on aggregated data of each country to analyse if correlation existed between perceived healthiness, environmental friendliness, and purchase intention.

To interpret data from the survey, part-worth utilities and relative importance of attributes were estimated by PLS. A one-way repeated measures ANOVA with post-hoc Tukey test was applied to compare attributes in terms of their utilities. Chi-square test was performed to compare relative importance between populations.

Hierarchical cluster analysis of purchase intention was employed to identify clusters that reflected the participants' different preferences. Kruskal Wallis Test was used for multiple comparison of data, with post hoc Dunn - Bonferroni test to examine significant differences (p-value 0.05) between clusters in their purchase intention.

A Principal Component Analysis (PCA) was performed to study the effect of the information in healthiness and environmental friendliness perception.

Statistical analysis was performed using XLSTAT (Addinsoft 2020; New York, NY, USA).

3. Results and Discussion

3.1 Relation between purchase intention and perceived healthiness and environmental friendliness

Correlation coefficients between purchase intention and participants' perceived healthiness and environmental friendliness are shown in Table 3.

Table 3. Pearson correlation between purchase intention and perceived environmental friendliness and perceived healthiness.

	Purchase intention		**Healthiness**		**Environmental friendliness**	
	Spain	Uruguay	Spain	Uruguay	Spain	Uruguay
Purchase intention	**1**	**1**	**0.738**	**0.880**	0.447	**0.773**
Healthiness	**0.738**	**0.880**	**1**	**1**	**0.618**	**0.809**
Environmental friendliness	0.447	**0.733**	**0.618**	**0.809**	**1**	**1**

Values in bold are significantly different from 0 ($p<0.05$).

Purchase intention was positively correlated with healthiness in both countries. However, environmental friendliness was positively correlated with purchase intention only in Uruguay. Healthiness and environmental friendliness perceptions were positively correlated in both countries. This was also seen in Lazzarini et al. (2016) study, who found a positive linear relationship between these two variables when evaluating different protein products. Curutchet et al. (2022) observed that purchase intention increased when the label included a sustainability logo and description of the added ingredient (in this case brewers spent grain) in the formulation of three different products: chocolate milk, pasta and bread.

In Figure 2 the relationship between perceived environmental friendliness, perceived healthiness and purchase intention is represented. Bigger bubbles represent a higher purchase intention. General perception of healthiness and purchase intention of these types of desserts was higher for Uruguayan population than for Spanish. In Uruguay, the perception of environmental friendliness of the homemade dessert was the highest, as well as their perceived healthiness and purchase intention. In Spain the trend was the same, but the difference with the other type of products was less pronounced.

Regarding environmental friendliness, it is perceived as higher when information (either as the fibre origin or as logo) is provided for both Spanish and Uruguayan consumers. The same is observed in the case of healthiness perception, where in each category the highest is observed when both the logo and fibre origin are presented.

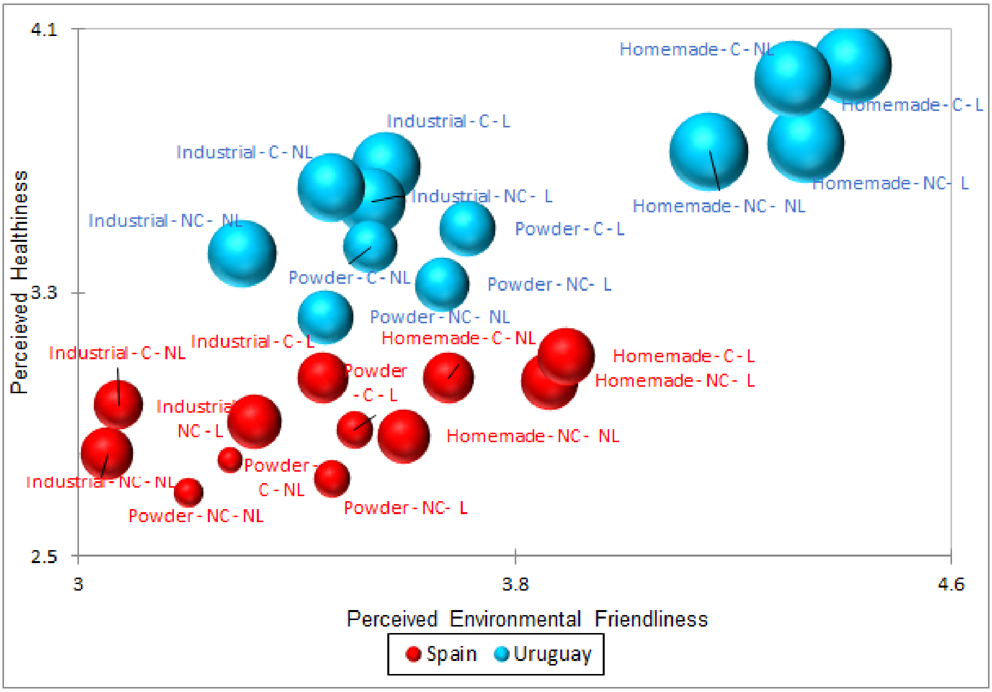

Figure 2. Perceived environmental friendliness and perceived healthiness related to purchase intention of Spanish and Uruguayan consumers. Larger bubbles indicate higher purchase intention. C: fibre origin claim; NC: no fibre origin claim; L: logo presence; NL: no logo presence.

It is interesting to note that in the case of Spanish consumers, although the presence of information about fibre origin caused an increase on perceived healthiness and environmental friendliness, this did not lead a positive impact on their purchase intention. In Uruguay, on the other hand, the higher perception of environmental friendliness and healthiness led to a higher purchase intention, as represented in Figure 2. Few studies have been done relating the perceived environmental friendliness of products and their purchase intention and are mostly about organic products consumption. In these cases, one of the reasons for consumers to eat that type of products is their perceived environmental friendliness, as reported by Gundala & Singh (2021) and Perito et al. (2020). Plamondon et al. (2022) found that communicating information about eco-friendly and nutritious menu choices to consumers within institutional settings could be relevant to enable them to make more eco-efficient food choices.

On the other hand, some studies indeed reported that a certain "fatigue" to the presence of labels appears over time and that their effectiveness is possibly short lived (Slapø, 2016). Similar results were reported by Koen et al. (2016), who observed that providing too much information on the label may confuse consumers.

3.2 Effect of product category and information about origin and sustainability on consumer response

Part worth utilities and mean relative importance of attributes in purchase intention, perceived healthiness and environmental friendliness are represented in Table 4. This table shows consumer preferences attached to each level of the attributes, indicating the influence of each factor level on respondents' preferences for a single combination (Lee et al., 2020).

Regarding purchase intention, product category was the attribute of highest importance both for Spanish and Uruguayan consumers. For both populations significant differences ($p<0.05$) were observed between the three products, the powdered product being the only one with a negative part worth utility value. The home-made dessert was preferred to industrial type in both consumer groups.

Table 4. Part-worth utilities of each level, mean relative importance of attributes (MRI) and significant effects in a one-way repeated measures ANOVA.

		Purchase intention				**Perceived healthiness**				**Percieved environmetal friendliness**			
		Uruguay		Spain		Uruguay		Spain		Uruguay		Spain	
Attribute	Attribute level	Mean part-worth utility	MRI	Mean part-worth utility	MRI	Mean part-worth utility	MRI	Mean part-worth utility	MRI	Mean part-worth utility	MRI	Mean part-worth utility	MRI
Product category	Home-made	$0,325^{a}$		$0,166^{a}$		$0,255^{a}$		$0,106^{a}$		$0,517^{a}$		$0,31^{a}$	
	Industrial	$0,021^{b}$	**93,30%**	$0,09^{b}$	**78,26%**	$-0,026^{b}$	**65,90%**	$0,024^{b}$	**53,78%**	$-0,321^{c}$	**79,80%**	$-0,224^{c}$	**65,10%**
	Powder	$-0,346^{c}$		$-0,256^{c}$		$-0,229^{c}$		$-0,13^{c}$		$-0,195^{b}$		$-0,086^{b}$	
Logo	With logo	$0,02^{a}$	**5,50%**	$0,048^{a}$	**14,74%**	$0,037^{a}$	**9,40%**	$0,046^{a}$	**18,97%**	$0,085^{a}$	**13,10%**	$0,135^{a}$	**28,30%**
	No logo	$-0,02^{b}$		$-0,048^{b}$		$-0,037^{b}$		$-0,046^{b}$		$-0,085^{b}$		$-0,135^{b}$	
Fibre origin	No claim	$-0,005^{a}$	**1,20%**	$0,023^{a}$	**7,00%**	$-0,096^{b}$	**24,70%**	$-0,066^{b}$	**27,25%**	$-0,046^{b}$	7,10%	$-0,031^{b}$	6,60%
	"Orange fibre"	$0,005^{a}$		$-0,023^{b}$		$0,096^{a}$		$0,066^{a}$		$0,046^{a}$		$0,031^{a}$	
	R^2	0,927		0,904		0,956		0,88		0,971		0,923	

Values within one column (and within one attribute) with different superscripts are significantly different according to Tukey's test ($p<0.05$). Bold percentages in mean relative importance for the same attribute were significantly different among countries according to z test ($p<0.05$).

Logo presence affected positively purchase intention for both groups, but the relative importance was higher in the case of Spanish consumers. Bhatt et al., 2021 found that the circularity of the logo increases the consumers' willingness to pay, and that green colour is associated with healthfulness and environmental benefits. These researchers found that the presence of a green circular logo improves the evaluation of upcycled foods, which result in an increase of purchase intention. In this sense, the perceived healthiness and environmental friendliness of products was higher when the logo was present. Furthermore, the European Union has been developing policies to achieve a more circular economy since the last decade, which has led to food companies developing solutions to address food loss (Sousa et al., 2021). It seems plausible, then, that consumers may be more willing to use upcycled products and therefore the logo presence has a higher impact in European consumers.

The attribute with the least relative importance was fibre origin for both populations. While Uruguayan consumers did not show significant differences ($p>0.05$) between product with fibre origin claim and no claim, Spanish people preferred the absence of claim.

For perceived healthiness, product category also was the attribute that contributed the most for both populations, followed by fibre origin and logo presence. Industrial and powdered products negatively affected the perceived healthiness for Uruguayan consumers, while Spanish only found powdered product to negatively affect this factor. This is in accordance with Román et al. (2017), who observed that product considered as healthiest is the one with the least processing perceived, in this case the home-made dessert. According to Laguna et al. (2020) consumers regard as unhealthier those products that are ready-to-eat, when compared to the homemade type of product. As they explain, the reason for this is that homemade meals are considered as healthier, as they do not contain additives and are not ultra-processed. Also, Rizk & Treat (2015) found that consumers tend to regard as healthier those products in which there is a qualifying nutrient: the presence of the claim seems to mask the other nutrients present (the healthiness of a rich in sugar product was regarded as higher when the product also had a high fibre content). Melendrez Ruiz et al. (2021) studied consumers response to different processing categories of pulses, finding that people prefer unprocessed products, as they consider the processed type as unhealthier. Products being perceived as healthier because of the presence of a favourable NutriScore also exhibit a higher purchase intention, as found by De Temmerman et al. (2021). According to Annunziata et al. (2016) European and north American consumers pay attention to nutritional labels when buying food, being the Americans the ones who spend the most time reading nutritional labels. Song et al. (2022) studied how information about processing of orange juice affected consumers choice, finding that Spanish consumers were found to be more affected by information than Danish consumers. These authors also found that the claims regarding products' benefits (such as longer shelf life or natural taste) are more important than information on processing.

In terms of environmental friendliness, the only category that affected positively its perception was the "home-made" type dessert, both for Spanish and Uruguayan consumers. This goes in line with what was described by Lazzarini et al. (2016), who found that processing negatively affected the perception of environmental friendliness. This was the variable that most influenced environmental friendliness perception, followed by logo presence and fibre origin. Regarding logo presence, Spanish consumers relative importance was 28.30%, while in Uruguayan case it accounted for 13.10% of the importance. Both populations found that products with logo presence and orange fibre claim were perceived more environmentally friendly than those who did not have them.

3.3 Consumer segments

Respondents were separated in three different clusters, for both Spanish and Uruguayan participants, based on differences on purchase intention on product category.

3.3.1 Spain

Three different group of consumers were identified, with different interests. Part worth utilities and mean relative importance of attributes in purchase intention of each cluster are presented in Table 5.

Although the three groups regarded the product category as the most important factor, Cluster 1 and 3 showed a higher importance for the logo presence, but they differ on the preferred product category. Cluster 1 prefers the industrial dessert, while cluster 3 prefers the homemade type. Cluster 2, on the other hand, regarded as more important the fibre origin claim. They also regarded as negative the logo presence, in contrast to the other groups.

Table 5. Part-worth utilities of attribute levels, mean relative importance of attributes (MRI) and significant effects in a one-way repeated measures ANOVA in purchase intention, for Spanish clusters

Attribute	Attribute level	Cluster 1		Cluster 2		Cluster 3	
		Mean part-worth utility	MRI	Mean part-worth utility	MRI	Mean part-worth utility	MRI
Product category	Home-made	0.067[b]	71.58%	0.191[a]	86.88%	0.171[a]	73.09%
	Industrial	0.174[a]		0.044[b]		0.114[b]	
	Powder	-0.241[c]		-0.235[c]		-0.285[c]	
Logo	With logo	0.089[a]	26.32%	-0.001[b]	0.40%	0.089[a]	22.94%
	No logo	-0.089[b]		0.001[a]		-0.089[b]	
Fibre origin	No claim	0.007[a]	2.11%	0.034[a]	12.72%	0.015[a]	3.98%
	"Orange fibre"	-0.007[b]		-0.034[b]		-0.015[b]	

Values within one column (and within one attribute) with different superscripts are significantly different according to Dunn's-Bonferroni test ($p<0.05$).

Table 6 shows demographics characteristics of each cluster. The composition of these clusters does not show big differences between them, other from the educational level of consumers in cluster 2. These people show a higher education level, which could explain the higher importance given to the fibre origin.

Table 6. Distribution of consumers (%) on Spanish clusters, according to demographics characteristics. Difference among proportions test, χ2 and probability (p) values

		Frequency (%)				
Characteristic	Level	Cluster 1	Cluster 2	Cluster 3	χ^2	p
Sex	Male	49	39	44	1.9	0.389
	Female	51	58	50	2.1	0.352
	Prefers not to say	0	3	6	3.4	0.183
Age	17-29	55	52	61	2.5	0.28
	30-44	28	28	24	6.0	0.628
	45-60	17	17	12	6.0	0.488
	> 60	0	3	3	1.3	0.514
Income level	Medium – Low	85	77	78	1.4	0.507
	Medium – High	15	23	22	1.4	0.507
Educational level	Primary	2	0	1	2.9	0,237
	Secondary school	21^{b}	5^{a}	24^{b}	6.0	<0.0001
	Third level (non degree)	15	11	5	5.4	0.066
	Third level (degree or higher)	62^{a}	84^{b}	70^{a}	12.9	0.002
Frequency of consumption	More than once per week	66^{b}	44^{a}	51^{ab}	9.8	0.007
	More than once per month	26	36	31	2.03	0.363
	Less than once per month	9	15	16	2.5	0.461
	Never	0^{a}	8^{b}	2^{a}	9.1	0.011

Percentage values in rows followed by different letters are significantly different ($p \leq 0.05$).

To analyze for each cluster the effect of each attribute studied on perceived healthiness and environmental friendliness, a principal component analysis was approached (Figures 3, 4 and 5).

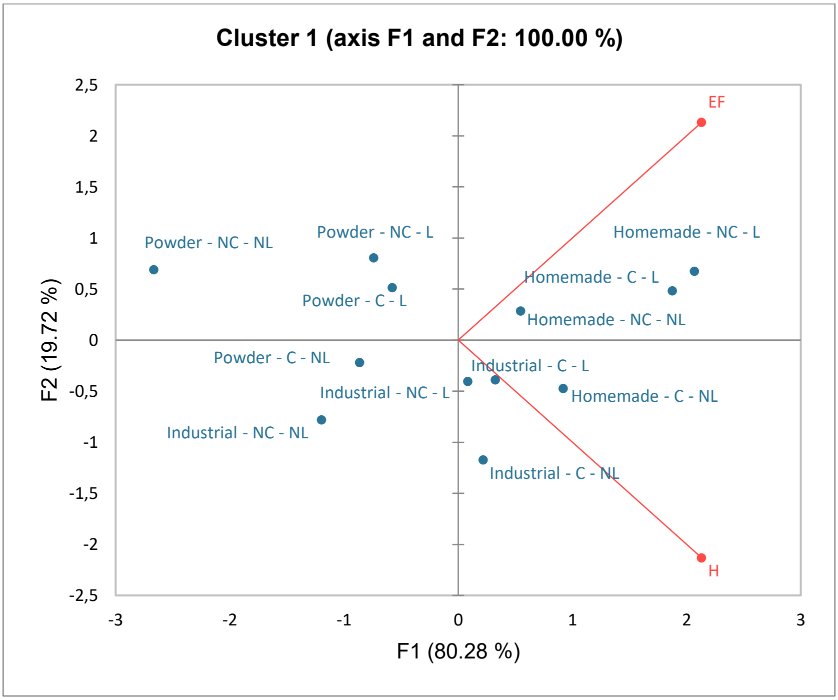

Figure 3. Principal component analysis for cluster 1. Relation between attributes and environmental friendliness and healthiness perception of Spanish consumers. C: fibre origin claim; NC: no fibre origin claim; L: logo presence; NL: no logo presence.

Cluster 1 regarded as environmentally friendly the homemade category and perceived the industrial and powdered product with no logo as not environmentally friendly. The industrial type was considered as healthy provided either information about fibre origin or logo were present, while powdered product was not considered healthy regardless of the provided information. This cluster is formed by regular consumers of dairy desserts (more than once per month), and equal share of men and women.

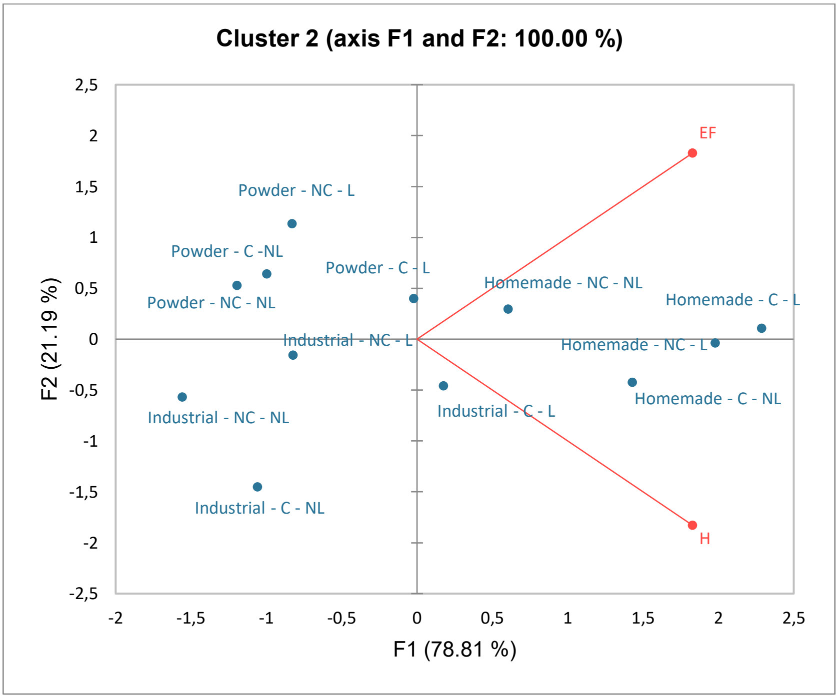

Figure 4. Principal component analysis for cluster 2. Relation between attributes and environmental friendliness and healthiness perception of Spanish consumers. C: fibre origin claim; NC: no fibre origin claim; L: logo presence; NL: no logo presence.

Cluster 2 considered the homemade dessert with no extra information was considered as environmentally friendly, while the industrial type was not. The homemade type was the healthiest, provided information was shown, either as logo or fibre origin claim. The powdered product was the less healthy. This group is the one with the highest share of people with third level education (95%), which could explain the healthiness evaluation. Also, this group is the one with the least options considered environmentally friendly, probably due to the higher education level as well.

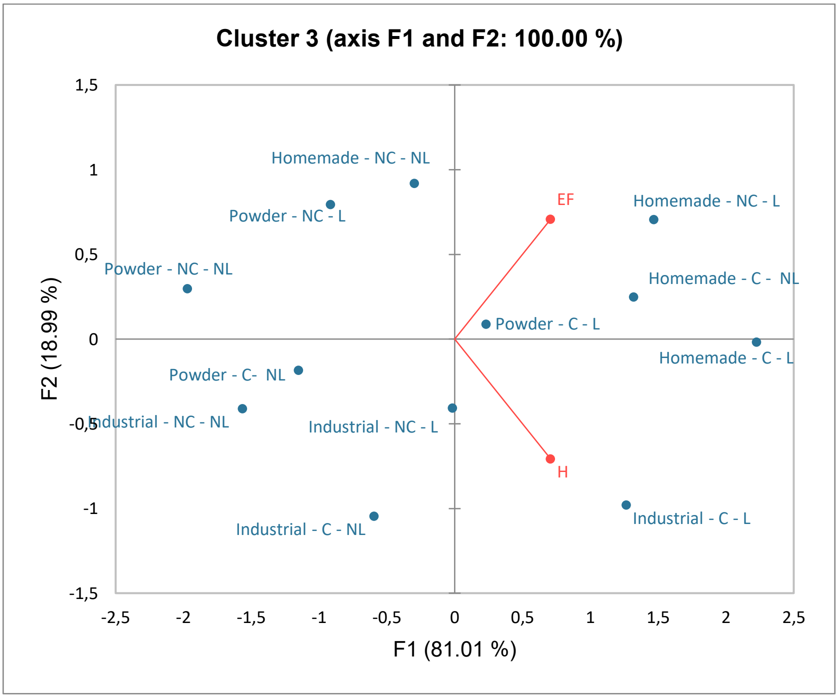

Figure 5. Principal component analysis for cluster 3. Relation between attributes and environmental friendliness and healthiness perception of Spanish consumers. C: fibre origin claim; NC: no fibre origin claim; L: logo presence; NL: no logo presence.

Cluster 3 considered industrial options as not environmentally friendly, and the logo or information presence did not improve this perception. Only homemade type without logo and information was perceived as healthy, which means the addition of information improved the perception of industrial and homemade product. It is interesting though how this group perceives the industrial type as healthier than the other categories. This cluster is formed by young people (18-29 years old).

3.3.2 Uruguay

In Uruguay, the three clusters regarded as the most important the product category, followed by fibre origin and then the logo presence. Part worth utilities and mean relative importance of attributes in purchase intention of each cluster are presented in Table 7.

Cluster 3 gave more importance to the fibre origin than the logo presence, when compared to cluster 2. This cluster is also the only one in which the industrial category is regarded worse than the powder, having negative part worth utility.

Table 7. Part-worth utilities of attribute levels, mean relative importance of attributes (MRI) and significant effects in a one-way repeated measures ANOVA in purchase intention, for Uruguayan clusters.

Attribute	Attribute level	Cluster 1		Cluster 2		Cluster 3	
		Mean part-worth utility	MRI	Mean part-worth utility	MRI	Mean part-worth utility	MRI
Product category	Home-made	0.326^{a}	85.60%	0.147^{a}	64.30%	0.394^{a}	71.80%
	Industrial	0.098^{b}		0.06^{b}		-0.222^{c}	
	Powder	-0.425^{c}		-0.207^{c}		-0.173^{b}	
Logo	With logo	0.023^{a}	4.70%	0.040^{a}	12.50%	0.004^{a}	0.60%
	No logo	-0.023^{b}		-0.040^{b}		-0.004^{b}	
Fibre origin	No claim	-0.048^{b}	9.70%	-0.075^{b}	23.20%	-0.151^{b}	27.60%
	"Orange fibre"	0.048^{a}		0.075^{a}		0.151^{a}	

Values within one column (and within one attribute) with different superscripts are significantly different according to Dunn's-Bonferroni test ($p<0.05$).

The composition of these clusters (Table 8) did not vary significantly in terms of age, income level and gender ($p>0.05$), but groups were significantly different in terms of educational level and frequency of consumption.

Table 8. Distribution of consumers (%) on Uruguayan clusters, according to demographics characteristics. Difference among proportions test, χ2 and probability (p) values

		Frequency (%)				
Characteristics	Level	Cluster 1	Cluster 2	Cluster 3	χ^2	p
Sex	Male	38	31	23	5.5	0.063
	Female	62	69	77	5.9	0.053
	Prefers not to say	0	0	0	0.5	0.785
Age	17-29	25	24	30	0.6	0.738
	30-44	34	24	30	1.3	0.514
	45-60	28	28	17	3.5	0.171
	> 60	13	24	24	6.0	0.054
Income level	Medium – Low	41	48	34	2.1	0.357
	Medium – High	59	52	66	2.1	0.357
Educational level	Primary	2	3	4	0.6	0.727
	Secondary school	4[a]	17[b]	7[a]	6.0	0.014
	Third level (non-degree)	19	10	13	2.4	0.308
	Third level (degree or higher)	75	69	76	0.6	0.751
Frequency of consumption	More than once per week	28[a]	55[b]	18[a]	13.8	0.001
	More than once per month	43	31	28	5.7	0.059
	Less than once per month	24[b]	7[a]	24[b]	4.5	0.107
	Never	5[a]	7[a]	30[b]	34.2	<0.0001

Percentage values in rows followed by different letters are significantly different ($p \leq 0.05$).

To analyse for each cluster the effect of each attribute studied on perceived healthiness and environmental friendliness, a principal component analysis was approached (Figure 6, 7 and 8).

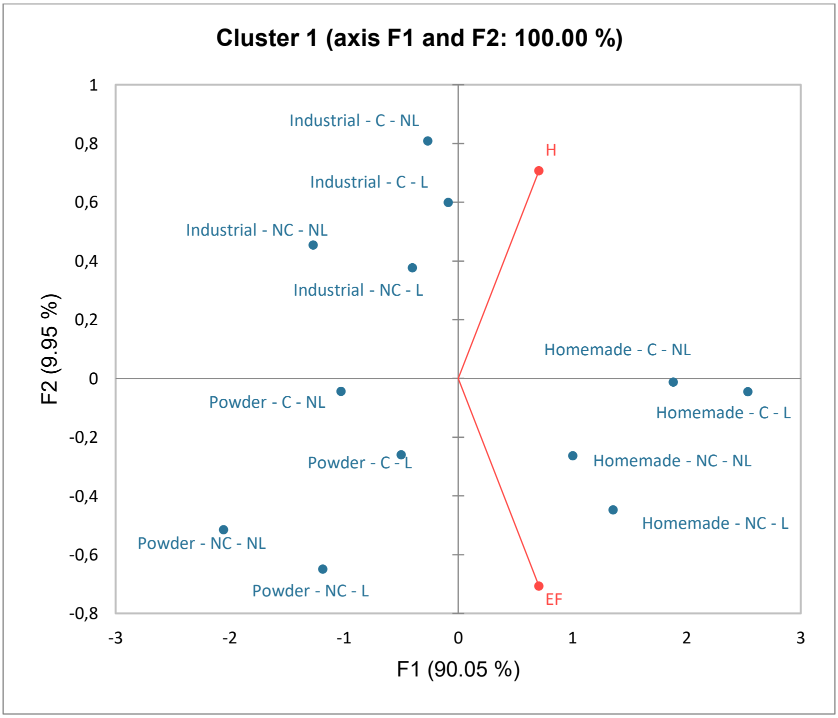

Figure 6. Principal component analysis for cluster 1. Relation between attributes and environmental friendliness and healthiness perception of Uruguayan consumers. C: fibre origin claim; NC: no fibre origin claim; L: logo presence; NL: no logo presence.

For Cluster 1 environmental friendliness perception was higher for the homemade type. It seems the information did not improve the perception, as the options without it were considered environmentally friendly. None of the products were considered as healthy options. These people are regular consumers of dairy desserts, 43% eating more than once per month and 28% eating more than once per week.

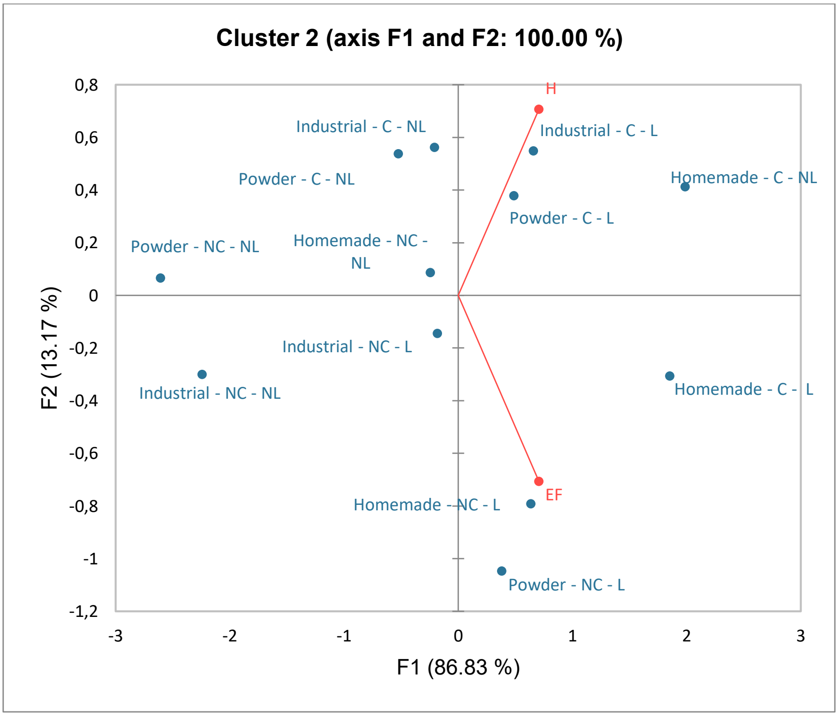

Figure 7. Principal component analysis for cluster 2. Relation between attributes and environmental friendliness and healthiness perception of Uruguayan consumers. C: fibre origin claim; NC: no fibre origin claim; L: logo presence; NL: no logo presence.

Cluster 2 perceived as environmentally friendly the homemade product with logo presence, and also the powdered product with logo and no information.

While the three type of products with information were regarded as healthy, the industrial type without information, regardless of logo presence, was considered not healthy. This cluster is formed mainly by people who are regular consumers of dairy desserts (55% consumes more than once per week), with people aged between 45 and 60 years.

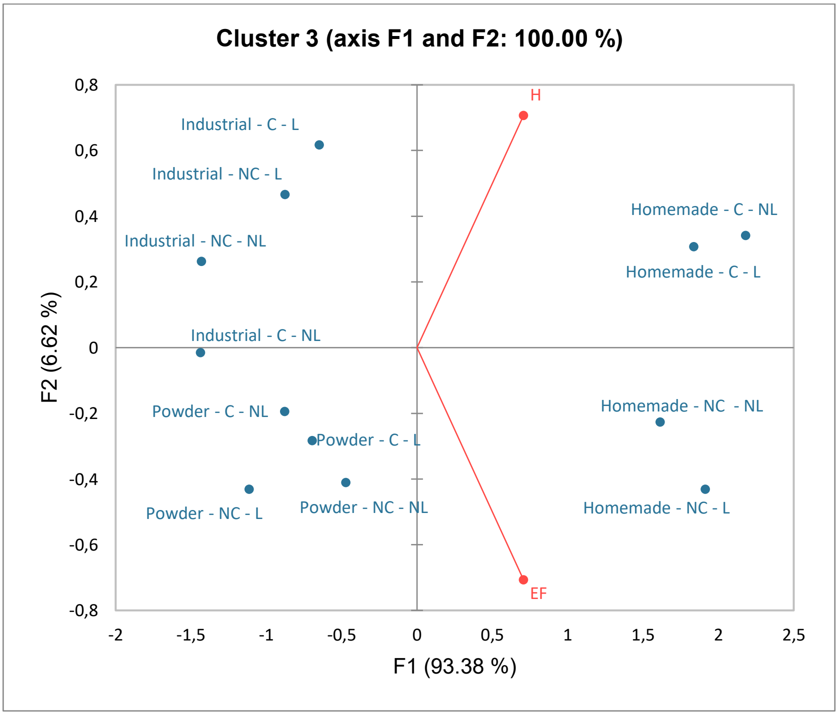

Figure 8. Principal component analysis for cluster 3. Relation between attributes and environmental friendliness and healthiness perception of Uruguayan consumers. C: fibre origin claim; NC: no fibre origin claim; L: logo presence; NL: no logo presence.

The third cluster perceived as healthier the homemade category with information. On environmental friendliness, the industrial type was the least environmentally friendly, and only the homemade options without information were considered friendly. This group is formed by people who are rare dairy desserts consumers (30% never eats, 28% less than once per month), aged between 17 and 45 years old and with a medium high income.

4. Conclusion

Product category was the most important attribute in purchase intention for Uruguayan and Spanish populations, being the home-made product preferred by both. Product category also affected the healthiness and environmental friendliness perception, probably due to differences on perceived processing level. The information about fibre origin had a positive impact on the perceived healthiness, while the logo related to sustainability had impact in purchase intention and environmental friendliness perception. The homemade product with a logo was the one perceived as healthiest and environmentally friendliest by both populations. The allegation “orange fibre” also increased these perceptions, although not the purchase intention in Spain.

Although Spanish consumers perceived the products with fibre claim and logo as healthier and environmentally friendlier, this did not lead to a positive impact in purchase intention. Purchase intention is related to healthiness in both populations, but only the Uruguayan relate it to environmental friendliness.

Cluster analysis showed three groups of Spanish consumers, two who consider more important the logo presence than the fibre origin claim. Within these two, one of the groups has a higher purchase intention in the industrial category, while the other prefers the industrial type. In the case of Uruguay, the three clusters analysed consider more important the fibre origin claim than the logo presence.

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Conflicts of Interest: "The authors declare no conflict of interest."

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Incorporating an upcycled orange fibre on flan formulation. Impact on sensory properties

Perez-Pirotto, Claudia [a, b], Hernando, Isabel [b], Cozzano, Sonia [a], Curutchet, Ana [a], Arcia, Patricia [a,c]

[a] – Departamento de Ingeniería, Universidad Católica del Uruguay. Montevideo 11600, Uruguay.

[b] - Food Microstructure and Chemistry Research Group, Department of Food Technology, Universitat Politècnica de València, Valencia 46022, Spain

[c] - Latitud Latu Foundation. Montevideo 11500, Uruguay.

En preparación para envío a *LWT - Food Science and Technology*

Abstract

Fruits by-products are a valuable source of ingredients, in the formulation of what is known by “upcycled foods”. Orange pomace, a by-product of orange juice industry, is a dietary fibre source. In this work, a powdered ingredient with soluble fibre obtained from orange pomace was used as replacement of inulin in the formulation of source of fibre “flan” like puddings. Four different formulations were analysed using Flash Profile and instrumental texture: 100% inulin, 70% inulin: 30% orange fibre, 30% inulin: 70% orange fibre, 100% orange fibre. The replacement of 30% of pudding’s total fibre with the new ingredient helped to improve the texture and general appearance of the dessert. Greater percentages imparted non desirable flavour attributes, such as bitterness and acidity. The use of this ingredient as a replacement of commercial inulin in the formulation of source of fibre puddings is possible. However, further research is needed to reduce the off flavours.

Keywords

Flash profile, by-product, texture analysis, product development, inulin.

1. Introduction

Dessert is a sweet food consumed mainly after meals (Riquelme et al., 2022). Semisolid dairy desserts are usually formulated with milk, sugar, aroma and thickeners, such as starch or gelatine (Dello Staffolo et al., 2017). Among these products is flan, which consists of a milk-based dessert, traditionally prepared with eggs, milk, and sugar. Some alternative formulations of flan-type puddings replace the eggs with gelling agents such as starch or hydrocolloids (Abd El-Fattah et al., 2019).

New trends demanding healthier products force the industry to formulate products with a better nutritional profile (Djaoud et al., 2020), such as fibre enriched. This would be an alternative to address the “fibre-gap”, a problem derived of current lifestyle, where people eat more processed foods and therefore, less fibre than needed. As a result, an increase of non-transmittable chronic diseases has been seen, as reported by (Chutkan et al., 2012; Jones, 2014; O’Keefe, 2019).

Dairy desserts may be fortified with fibre, being the inulin widely used due to its high solubility compared with other fibres (Kalyani Nair et al., 2010). Inulin is a fructose polymer (linked by a β(2,1) bond) commonly found in chicory root and other plants. Its degree of polymerization varies between 2 and 60. It has a neutral taste, is colourless and does not provide any viscosity. It is therefore regarded as an invisible way of adding fibre to foods. Although adding fibres such as inulin may help close the fibre gap, these do not have any phytochemicals associated. Fibre derived from fruits, however, has embedded bioactive compounds with antioxidant capacity that can be absorbed in the small intestine, fermented in the large intestine and also help keep an antioxidant environment in the intestinal lumen (Palafox-Carlos et al., 2011).

In the search for new fibre source, the use of a by-product, with high fibre content in its composition and bioactive compounds may also be an alternative for fibre fortification, as well as contributing to the reduction of food waste (Perez-Pirotto, Cozzano, et al., 2022; Tagliani et al., 2019). In this sense, upcycled foods have been emerging across the world. These are those that are produced using ingredients that would otherwise be wasted, such as fruit peels (Bhatt et al., 2021; Goodman-Smith et al., 2021).

In the other hand, in addition to the nutritional benefits, dietary fibre has some technological advantages such as increasing water and oil holding capacity and gel forming ability, that could be helpful in the development of dairy desserts (Dello Staffolo et al., 2017)

In this context, on the development of new products with added fibre, it is important to understand how this ingredient may change sensory aspects of the product. Fortifying foods may change its characteristics, as some may impart a coarse texture and strong flavour, as well as a dry mouthfeel (Mudgil et al., 2017). Work has been done with fibre fortified yogurts, where the fibre addition gave products with a more compact texture and a particulate mouthfeel (Kieserling et al., 2019). Ardabilchi Marand et al. (2020) found that fortifying yogurts with flaxseed at high levels (5%) gave graininess and bad mouth-feel, produced by the insoluble powder particles. Ares et al. (2009) studied the addition of high-amylose maize starch in dairy dessert formulation and found that this addition may cause roughness, rough after feel and changes in taste, as well as a decrease in creaminess and melting.

To evaluate changes on sensory profile of new developed product, flash profile may be a promissory approach. Flash profile was developed as a variant of Free Choice Profiling, in which each panellist uses its own list of attributes (free-choice terms) and ranks the sample according to the intensity of those attributes. It is a fast method that does not need a common vocabulary, in which panellists are presented with all the samples at the same time. As a result, it provides relative sensory positioning of the whole product set (Liu et al., 2018; Santos et al., 2013).

The aim of this work is to investigate the effect on sensory properties of replacing inulin with an upcycled orange fibre in a flan type pudding. Sensory properties were evaluated through flash profiling. Instrumental texture measurements were also carried out to investigate the effect of inulin substitution in texture parameters.

2. Materials and methods

2.1. Orange fibre extraction

For orange fibre's extraction, the extrusion + hot water (EHW) procedure described by Perez-Pirotto, Cozzano, et al. (2022) was used. Briefly, orange pomace was dried in a convection oven at 55˚C for 72 hours and grinded to a particle size of less than one millimetre. Sample was afterwards humidified to obtain a moisture content of 15% and left overnight to reach equilibrium.

The equilibrated sample was extruded in a single screw extruder, at 230 rpm and 129˚C. Extruded sample was then milled until particle size was less than one millimetre, and hot water extraction was performed. For this purpose, the milled sample was mixed with water (1:16.6 m/V) and kept at 75˚C for 45 minutes in a mechanically agitated boiler. The slurry was then centrifuged at 9500 rpm and 4˚C and supernatant was concentrated under vacuum until solids content was 15±2˚Bx. Whey protein isolate (WPI-Provon 292, Glanbia Nutritionals Inc.) was added to this concentrate (8% soluble solid basis) and kept under magnetic stirring at 60˚C until completely dissolved. Finally, the sample was spray dried in a laboratory spray drier (Buchi B290). Powder was kept in laminated bags in the dark at room temperature until use.

2.1 Desserts formulation

Desserts were prepared with different relation orange dietary fibre/inulin, so they could be labelled as source of fibre according to European Union regulation. According to previous works, total fibre content on Orange fibre ingredient is 20% (Perez-Pirotto, Cozzano, et al., 2022). Four different formulations were tested (Table 1).

For preparation, milk was heated to 37˚C in a Thermomix. An aliquot was separated to dissolve the gelatine and the vanilla flavour. Sugar, inulin, and orange fibre were mixed and added to the heated milk. The mixture was heated to 80˚C under agitation and once temperature was reached the heating was stopped. The sample was kept under agitation for five minutes. The gelatine was dissolved in milk and gently heated until dissolved. Vanilla flavour was added to this mixture and the whole set was added to the initial preparation. The mixture was poured into 30 cc plastic glasses with lids and kept under refrigeration at 4˚C for 24 hours until flash profile evaluation.

Table 1. Dessert formulations to be claimed as "source of fibre".

	100%In	**70% In: 30% OF**	**30% In: 70% OF**	**100% OF**
Inulin	3	2.1	0.9	0
Orange fibre	0	4.5	10.5	15
Sugar	9.63	9.63	9.63	9.63
Gelatine	1.2	1.2	1.2	1.2
Vanilla flavour	0.2	0.2	0.2	0.2
Whole milk *	85.97	82.37	77.57	73.97

* Whole milk was adjusted to keep constant the percentage of the other ingredients, while varying the orange fibre and inulin content. In: Inulin; OF: Orange fibre.

2.3. Flash profile

Sensory characterization of the desserts was conducted through a Flash Profile test. Fourteen people with experience in sensory analysis participated in the test, recruited among students and employees of the Universitat Politécnica de Valencia. Flash profiling (FP) was performed in one unique session, as it does not demand a specific participant training stage. Prior to the evaluation session, the researcher explained the procedure to each panellist and gave him/her a printed example of how to carry out the test. In the evaluation step, after tasting and observing the samples, each panellist generated his/her own list of attributes to describe the differences among the four desserts. No indication was given regarding the number of attributes that should be proposed. The panellists then ranked each sample on an ordinal scale for each attribute they had individually proposed (ties were allowed). The evaluation was individual, and each panellist was presented with the whole sample set simultaneously. The samples (30 mL) were served at consumption temperature (5.0 ± 0.5 °C) in small transparent plastic cups coded with random three-digit numbers. Mineral water and crackers were provided for rinsing the mouth between samples.

2.4. Texture analysis

For texture measurements, a texture analyser was used (TA.XT2, Stable Micro Systems, Surrey, England). A P/0.5 Delrin flat-tipped cylindrical probe was inserted into the flan at a rate of 2.0 mm/s to a penetration distance of 12 mm. 5 replicates by sample were done. The hardness, consistency, and adhesiveness were recorded.

2.5. Statistical analysis

One way analysis of variance (ANOVA) with Tukey post-hoc test ($p<0.05$) was performed on texture results. The data analysis for the flash profile was performed using generalized Procrustes analysis (GPA). This reduces the scale usage effects, delivers a consensus configuration, and allows the comparison of the proximity between terms that are used by different assessors to describe the test sample. An individual matrix for each participant (product x attributes) was built to enter the product rankings. The average sensory configuration is displayed on a score plot, together with the correlations of all individual attributes with the factorial axes. Therefore, if an attribute appears is used more than once by panellists, it will appear several times in the loading plot. To facilitate the interpretation of the data, a Hierarchical Cluster Analysis was performed on the attributes' coordinates obtained from GPA (Lassoued et al., 2008).

3. Results and discussion

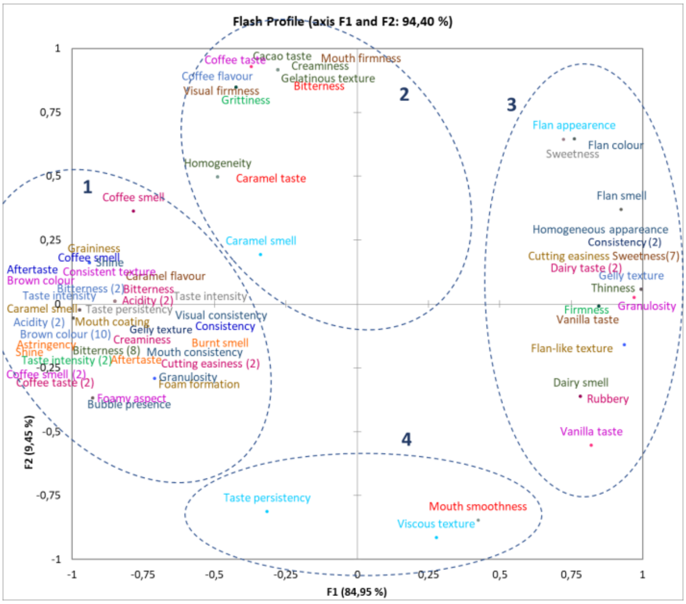
3.1. Flash profile

A total of 46 different terms were generated, from which 17 were texture terms, 14 were taste terms, 10 were general appearance terms and 5 were odour terms. Although there was higher diversity in texture terms, the most evaluated parameters of the desserts were taste ones (43% of mentions).

Figures 1A and B show the biplots obtained by GPA analysis from the flash profile. The first two principal axes accounted for 94.40% of the variability of the results. The first factor explains 84.95% of the experimental data's variability. This axis separates the desserts formulated with more than a half of fibre coming from inulin from those with higher percentages of orange dietary fibre. The dessert formulated with 100% orange fibre (100% OF) and the one formulated with 70% Orange fibre and 30% inulin (30% In:70% OF) where close to each other, while the other two were more apart among them.

The second factor (9.45% of data variability) separates mainly the dessert formulated with 70% inulin from the others, although the separation between the four of them is not as big as in the X-axis.

A)



B)

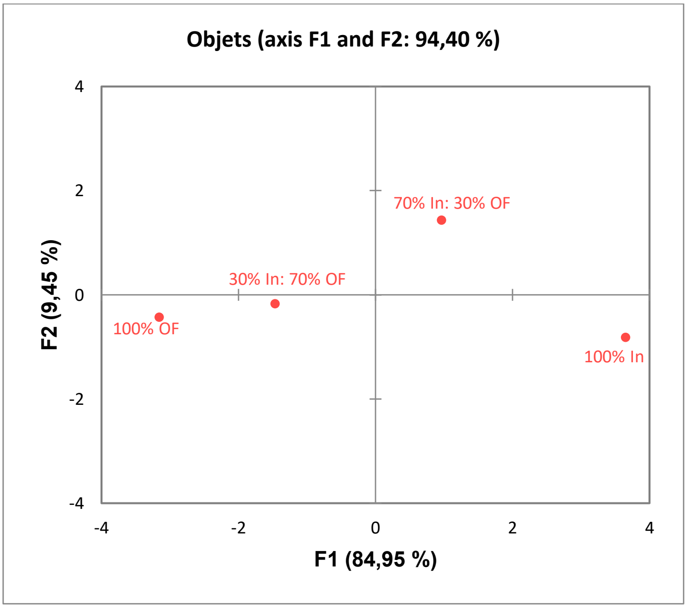


Figure 1. A) Representation of the terms used to describe the sample; number in parentheses following an attribute represent the times the same attribute superimposed in the same place in the original GPA plot; and B) representation of

the dessert samples in the first two dimensions of the GPA from data of the flash profile.

A Hierarchical Cluster Analysis was performed to facilitate the interpretation of the different descriptors used, revealing four different groups of sensory terms. These are represented in figure 1A, with the dotted lines. As can be seen in that figure, these groups not necessarily represent each one a product. However, it seems that cluster 3 refers to the products with less orange fibre added.

F1 seemed to oppose attributes such as bitterness, brown colour and coffee/burnt smell with sweetness, vanilla taste, and dairy smell. This is due to higher percentages of inulin substitution with orange fibre, which provides the brown colour and burnt smell. Bitterness is probably due to orange flavour, as well as some burnt compounds which could result in bitterness (Hofmann, 2005; Wuttipalakorn et al., 2009). As vanilla flavour was added in equal amount in all formulations, it seems that orange fibre masked its taste and therefore its intensity is higher on those with lower content of this ingredient.

100% OF was perceived as bitter, brownish, with more consistence, while the 100% In was regarded as the sweetest, with dairy smell, more homogeneous and a with a gelatinous structure. Other parameters that described those flans with higher amount of orange fibre were coffee-like descriptors, such as coffee smell and taste, probably due to their toasted smell, which was also regarded as “burnt smell" and “caramel taste”.

The whole substitution of inulin with orange fibre gave a bitter, acid, and brown product, which was not considered like the commonly known flan. On the other hand, the product that had 30% of the fibre content provided by the orange fibre was the most like this kind of flan-type desserts.

Also, the products with 70 and 100% fibre content from orange fibre were regarded with higher aftertaste and taste persistency, which could be due to the strong flavour of the ingredient. These also presented higher bubble formation or foaming, which is related to the foaming capacity of this ingredient, as described by Perez-Pirotto, Moraga, et al. (2022).

Considering all the information provided by the GPA plot, 100% In and 30% OF were perceived as more like what is commonly expected of a flan, than those with higher amounts of OF, where bitter compounds were very evident.

3.2. Texture analysis

Firmness is defined as the maximum force required to compress the sample; in eating process, it is the force that is needed to compress the sample between the palate and the tongue (Cardarelli et al., 2008). Consistency refers to the work of penetration. Thus, it measures the resistance offered by the sample to penetration process. Adhesiveness is the necessary energy required to disrupt attractive forces between food surface and other materials (Lobato et al., 2009). Texture analysis results are shown in Table 2.

Table 2. Texture parameters

Formula	Firmness (N)	Consistency (N.s)	Adhesiveness (N.s)
100 In	0.91 ± 0.02^{a}	2.39 ± 0.10^{d}	-0.17 ± 0.04^{a}
70 In:30 OF	0.84 ± 0.06^{b}	3.90 ± 0.10^{a}	-0.55 ± 0.06^{b}
30 In:70 OF	0.82 ± 0.01^{b}	3.30 ± 0.09^{b}	-0.88 ± 0.07^{d}
100 OF	0.75 ± 0.02^{c}	2.65 ± 0.04^{c}	-0.69 ± 0.08^{c}

Values in the same column with different letters are significantly different according to the Tukey test ($p<0.05$). In: Inulin; OF: Orange fibre.

The dessert formulated with 100% inulin was the harder, and hardness decreased when the OF content increased. In this work, the lowest consistency was seen in the 100% In, and highest with 30% OF. Adhesiveness was highest in 70% OF formulation.

Gurditta et al. (2019) tried different percentages of sucrose substitution with different dietary fibers in an Indian cottage cheese-based dessert and found their hardness increased when increasing the fibre content. In our case, the fibre content was kept constant, but the source was changed. Hardness decreased when decreasing the percentage of inulin used. As the gelatine used was the same amount for all the samples, as orange fibre content increases and the milk content decreases, it could be expected to get a harder dessert (gelatine has less amount of liquid to gel). That was not the case, indicating that the orange fibre may hinder the places in which the milk bonds with gelatine and prevents gelation. According to (Wang et al., 2019) the addition of fruit by-products in yogurt increased their firmness due to the interactions between those particles and milk protein matrix. In this case, however, there are no insoluble particles that could help increase the hardness. Also, orange fibre may disrupt the gel and decrease firmness (Narender Raju & Pal, 2014).

Despite the lower hardness, the lower values are consistent with what is reported for an egg-formulated flan in bibliography (Abd El-Fattah et al., 2019), who found hardness for that type of dessert was 0.5 N. The general texture and appearance of those desserts formulated with higher amounts of OF are more similar to what is expected in this kind of desserts, as the one with 100% inulin was too similar to gelatine itself, as described by flash profiling.

4. Conclusions

The sensory profile of the product with a replacement of orange fibre in 30% of the total fibre content was described like what is commonly known as flan. The use of orange fibre in percentages higher than 30% of total fibre content gave the products non desirables flavours like burnt, bitter and taste persistent. Nevertheless, with respect to texture characteristics, these were improved with the use of orange fibre instead of inulin, granting less firmness and moderate consistency, attributes expected in a flan-type pudding. The use of orange fibre as an alternative to fibre enrichment on flan formulation is possible. To reach higher levels of orange fibre incorporation it may be necessary to investigate how to keep the off flavour to a minimum.

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Discusión general de resultados

La presente tesis se centró en la obtención de un ingrediente en polvo, que pudiera utilizarse en la formulación de alimentos para enriquecer su contenido de fibra. Para ello, y en un marco de economía circular, se partió del subproducto de la industria citrícola y se ensayaron cuatro tecnologías de extracción no contaminantes diferentes: agua caliente; extrusión + agua caliente; jet cooker; jet cooker + agua caliente.

En la figura 1 se presenta un diagrama del trabajo realizado.

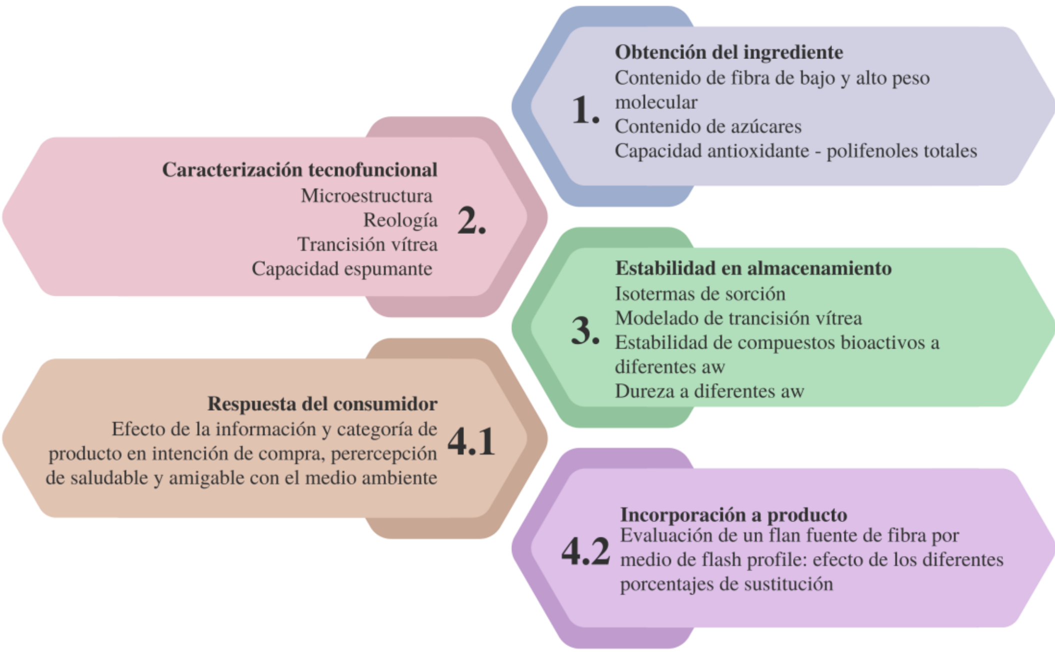

Figura 1. Diagrama descriptivo del trabajo realizado

En la primera etapa, luego de obtener los ingredientes, se caracterizó la composición de los polvos obtenidos según la tecnología de extracción aplicada. La apariencia de los polvos se muestra en la figura 2.

Figura 2. Apariencia de los polvos tras el secado, de izquierda a derecha: agua caliente; extrusión + agua caliente; jet cooker; jet cooker + agua caliente.

Los resultados mostraron que los tratamientos lograron extraer fibra en diferentes proporciones. El uso de agua caliente a escala piloto permitió obtener resultados similares a los reportados en bibliografía en escala laboratorio. El uso de vapor mediante el jet cooker no logró mejorar sustancialmente el contenido de fibra extraído, aunque se evidenció un mayor rompimiento de los enlaces glicosídicos, lo que se tradujo en la obtención de un polvo con mayor contenido de azúcares. La combinación de ambos tratamientos (jet cooker + agua caliente) logró un aumento del contenido de fibra de alto peso molecular. Esta fibra pudo haberse generado como consecuencia de enlaces que fueran debilitados en la etapa de pasada por jet cooker y se terminaran de romper en el proceso posterior de extracción en agua. La extrusión, por otro lado, logró duplicar el contenido de fibra extraído respecto al agua caliente. A su vez, fue el tratamiento que menos azúcares liberó. Contrariamente a lo que se esperaba, no se liberaron los ácidos urónicos y azúcares de las pectinas.

En cuanto a la capacidad antioxidante y contenido de polifenoles, los valores aumentaron con los tratamientos más agresivos térmicamente. Esto podría deberse a la formación de compuestos antioxidantes vía reacciones de Maillard, o a la liberación de estos compuestos, que están normalmente unidos a la fibra, durante el procesamiento térmico.

Una vez que se estudió la composición de los polvos se estudiaron las propiedades tecno funcionales de los ingredientes obtenidos. Todos fueron solubles en agua y forman espumas en solución acuosa. Las soluciones formadas presentaron comportamiento newtoniano en todos los casos, pero se observó un aumento de la viscosidad aparente en la solución con el polvo obtenido mediante la extrusión respecto a la del agua, siendo 25 veces mayor. Justamente, este ingrediente, de todos los tratamientos ensayados, fue el de mayor contenido de fibra soluble, por lo que es posible que fuera este componente el responsable del aumento en la viscosidad. En la capacidad espumante también fue el tratamiento con resultados más interesantes, ya que su espuma fue la más estable de las cuatro muestras estudiadas. Esto pudo deberse tanto al aumento de la viscosidad de la fase continua como al contenido de polisacáridos: estos recubren las burbujas de aire, formando un film flexible que mejora la formación de espuma.

Dado que todos los polvos se obtuvieron mediante el secado de soluciones acuosas, la temperatura de transición vítrea fue baja. Esto se debe a que esas soluciones tienen principalmente azúcares y compuestos de bajo peso molecular disueltos. Aunque se utilizó proteína de suero aislada como agente encapsulante para evitar el "caking" del producto, en los polvos con mayor contenido de azúcares se observó una coalescencia de los polvos, fruto de los puentes formados por el agua circundante. Este fenómeno fue observable tanto a nivel microscópico como macroscópico, donde se evidenció que aquellos polvos con temperatura de transición vítrea más baja tienden a estar más aglomerados, y los grumos son más compactos. En la microscopía electrónica de barrido, se observó coalescencia de las partículas, sobre todo en el caso del jet cooker.

Una vez finalizada la caracterización de los ingredientes, se estudió su estabilidad en el almacenamiento. Se almacenaron en cámaras con diferentes actividades de agua a temperatura ambiente por 15 semanas, y se estudiaron las isotermas de sorción y transiciones vítreas, junto con el efecto de la humedad relativa en la capacidad antioxidante, contenido de polifenoles, propiedades mecánicas y color. Las isotermas obtenidas se ajustaron al modelo de Henderson con un muy buen ajuste ($R^2>0.90$), lo cual es esperable en productos con alto contenido de azúcar. El polvo obtenido mediante el jet cooker no se encontró en estado vítreo al almacenarse a temperatura ambiente a ninguna de las actividades de agua ensayadas. Este resultado pudo deberse a su composición, su contenido de fibra es menor al de otros tratamientos, y su contenido de azúcares es el mayor. Por ello, su peso molecular probablemente sea menor, y su temperatura de transición vítrea, por lo tanto, también. Los otros tratamientos sí permitieron encontrar condiciones de almacenamiento que permiten que el polvo se conserve en el estado vítreo al almacenarse a 20˚C. En temperaturas de refrigeración (5˚C), el polvo obtenido mediante la extrusión + agua caliente se mantendría en el estado vítreo si se almacena a una humedad menor al 30.9%. En los otros casos deben realizarse modificaciones a los procesos de obtención del ingrediente para mejorar la humedad del producto final, dado que en todos los casos esta es mayor al contenido crítico de agua que asegura la estabilidad. La humedad del ingrediente debería ser menor a 4.71% en el caso de jet cooker + agua caliente, 4.24% en agua caliente y 3.31% en el caso de jet cooker. Para ello podrían tomarse varios caminos. En primer lugar, se podría aumentar la temperatura de secado, con el consiguiente efecto en los compuestos bioactivos y propiedades sensoriales. También se podría ensayar secar con una velocidad de alimentación menor, lo que logrará un tiempo de residencia mayor dentro de la cámara de secado y por lo tanto un polvo más seco. Por último, también se podrían realizar cambios en el proceso de encapsulación. Podrían utilizarse otros compuestos de mayor peso molecular, así como también incrementar su concentración, y de este modo aumentar el peso molecular de la mezcla. Debido a la relación entre este último y la temperatura de transición vítrea, si se agrega más cantidad se elevaría su peso y por lo tanto aumentaría su T_g, asegurando que el polvo se encuentra en estado vítreo a temperatura ambiente.

Respecto a la textura y apariencia de los polvos, el obtenido mediante extrusión + agua caliente fue el de apariencia más homogénea. Se mantuvo sin aglomerarse por más tiempo, como se evidencia tanto a simple vista como en el ensayo de textura (la mayor dureza se da a actividades de agua mayores). Se observó también un pardeamiento mayor conforme aumenta la actividad de agua, que se traduce en una mayor diferencia de color con la muestra inicial. Al aumentar la humedad relativa del medio, el producto adsorbe agua y esto genera dos cosas: una mayor movilidad de las moléculas, que se traduce en mayor velocidad de reacción y, por tanto, de pardeamiento, y también una saturación mayor del color debido al contenido mayor de agua.

Finalmente, debido a que el interés principal de la obtención de este tipo de ingredientes es su uso para la fortificación en fibra de otros alimentos, se estudió cual sería el impacto de su incorporación en la elaboración de flanes. Por un lado, se estudió cómo comunicar a los consumidores sobre la incorporación de fibra y su origen, y posteriormente el impacto sensorial de agregar el ingrediente a los productos. Para lo primero, se estudió el efecto de la información de la fibra en dos poblaciones diferentes; consumidores uruguayos y españoles, cuando se usa en diferentes categorías de un mismo producto. Se tomó en consideración el hecho de que el efecto del procesamiento de alimentos podría influir fuertemente en la percepción de los consumidores. Por ello se consideraron diferentes categorías de un flan: un producto listo para consumo, un polvo para preparar el postre en casa y un flan casero. En la figura 3 se muestran las imágenes utilizadas. Junto a las imágenes se presentó información relacionada con el origen del enriquecimiento de fibra ("con fibra de naranja") e información relacionada a la sostenibilidad, a través de la presencia de un logo, que intenta comunicar el uso de un subproducto (figura 4).

Figura 3. Categorías de producto utilizadas.

Figura 4. Logo

Los consumidores fueron consultados por su intención de compra, percepción de amigable con el medio ambiente y de saludable. En general, estas tres variables de respuesta fueron más altas para la población uruguaya que para la española. Respecto a la categoría de producto, el casero fue el que presentó mayor intención de compra, en ambas poblaciones. Respecto a la información sobre el origen de la fibra, la población española lo consideró más relevante en la intención de compra y en la percepción de saludable que la población uruguaya.

Mientras que los uruguayos consideraron saludable el producto casero únicamente, los españoles también consideraron el producto industrial como una alternativa saludable. Respecto a la percepción de amigable con el medio ambiente, ambas poblaciones coincidieron en encontrar únicamente el producto casero relevante en esta variable. Sería interesante estudiar si eso se debe a la categoría del producto en sí mismo, o si se refiere al tipo de envase que presentan, ya que este producto es el único que no tiene ninguno asociado y por lo tanto podría considerarse "menos contaminante". Otro aspecto interesante para profundizar es cómo el consumidor español presentó una intención de compra mayor para aquellos productos sin la información sobre el origen de la fibra, a pesar de que los consideraron menos saludables y amigables con el medio ambiente. Por lo tanto, aunque el producto informado se perciba más amigable con el medio ambiente y saludable, a la hora de la compra eso no es determinante y el consumidor está más dispuesto a comprar el producto sin información.

Finalmente, se incorporó el ingrediente con mayor contenido de fibra soluble (obtenido por el tratamiento extrusión + agua caliente) en la formulación de un flan “fuente de fibra”. En este estudio se trabajó con inulina como fibra “control” y se sustituyeron diferentes porcentajes de la fibra total del producto con el ingrediente obtenido, para poder mantener el etiquetado “fuente de fibra”. La evaluación sensorial se realizó por medio de la metodología de “flash profile”, lo que permite una evaluación rápida y sin necesidad de entrenamiento de panel. Del análisis de estos datos, se observó que el flan con un 30% de sustitución de inulina por la fibra de naranja, es descrito de forma similar al flan sin fibra de naranja (100% inulina). Esto es interesante, ya que muestra que los evaluadores no encontraron mayor diferencia entre estos productos, mostrando un potencial de sustitución a estos niveles sin perjuicio de cambios en atributos sensoriales de impacto. Los productos con mayor porcentaje de fibra de naranja añadida fueron calificados con atributos como “quemado”, “amargo” y “ácido”. Estos fueron claramente aportados por el ingrediente, en el que el proceso de obtención favoreció las reacciones de caramelización y con ello los consiguientes compuestos aromáticos. Los sabores amargo y ácido también pueden haber sido aportados por la propia cáscara de naranja. Por esto, se hace evidente la necesidad de ajustes en el proceso, de forma de disminuir el tiempo de sometimiento a altas temperaturas y con ello los productos de la caramelización que otorgan sabores extraños al producto. De este modo, también se podría mejorar el color del producto para lograr obtener un ingrediente con un color más claro, y no asociado a quemado. Por otro lado, también podría evaluarse cómo eliminar o reducir el sabor amargo del ingrediente y así lograr un sabor más neutro y ampliar su potencial de uso.

Respecto a la apariencia y textura (Figuras 5 y 6), visualmente se observó que el agregado de la fibra de naranja aumentó el brillo del postre y mejoró el corte, disminuyendo la textura gelatinosa y aumentando la cremosidad. El flan con mayor contenido de fibra de naranja presenta una apariencia más cremosa, mientras que el flan formulado con inulina únicamente tiene un corte más gelatinoso.

Figura 5. Apariencia de los flanes luego de desmoldados. De izquierda a derecha: 100% inulina; 70% Inulina:30% fibra de naranja; 30% fibra de naranja: 70% inulina; 100% fibra de naranja.

Figura 6. Corte de los flanes. De izquierda a derecha: 100% inulina; 70% Inulina:30% fibra de naranja; 30% fibra de naranja: 70% inulina; 100% fibra de naranja.

Al evaluar la textura de forma instrumental se observó que la firmeza del flan disminuye al aumentar el contenido de fibra de naranja. Aunque esto podría considerarse contradictorio, lo que se observó al aumentar el contenido de fibra es un producto más compacto, cremoso, que ofrece menos resistencia al corte (menos firme).

Por lo tanto, es posible el uso de fibra de naranja como alternativa al enriquecimiento de fibra en la formulación de flan. Sin embargo, para alcanzar niveles más altos de incorporación de fibra de naranja, será necesario investigar cómo mantener los “off flavor” al mínimo.

Conclusiones

- Fue posible obtener un ingrediente en polvo a partir del residuo de la industria citrícola mediante el uso de diferentes tecnologías de extracción no contaminantes. El contenido de fibra soluble en el ingrediente varió entre un 10 y 20% entre los tratamientos ensayados. Estos polvos presentan un elevado contenido de azúcares simples (glucosa, fructosa y sacarosa) y compuestos bioactivos que podrían proceder del subproducto o haberse formado en el proceso de extracción.

- El tratamiento de extrusión + agua caliente es el que obtuvo una mayor extracción de fibra soluble y menor contenido de azúcares. Esto indica que el proceso logra liberar fibra soluble, sin perjuicio de aumentar el contenido de azucares simples. Además, este tratamiento fue el que obtuvo mayor contenido de compuestos bioactivos. Se destaca el hecho de ser un procesamiento sencillo y sin uso de solventes contaminantes.

- Los ingredientes presentan propiedades tecno funcionales de aplicación industrial, como la capacidad espumante, aumento de la viscosidad sin cambio en el tipo de flujo, y alta solubilidad en agua. Esto hace que la incorporación a producto presente potencial no solamente desde el punto de vista nutricional sino también desde el punto de vista tecnológico.

- Respecto a la estabilidad en el almacenamiento, los polvos no serían estables en las condiciones en las que se obtuvieron, dado que el contenido de humedad es mayor al crítico según lo descrito por la relación entre la isoterma de sorción y la transición vítrea. Por ello, y para evitar o retardar las reacciones de deterioro, sería necesario mejorar el proceso de secado, o cambiar el encapsulante, para disminuir la humedad del producto final y aumentar la temperatura de transición vítrea.

- Al evaluar el potencial uso del ingrediente, y cómo comunicar a los consumidores sobre el origen de la fibra, así como transmitir un mensaje de sostenibilidad, se observó que las poblaciones uruguaya y española presentan comportamientos diferentes. Ambas poblaciones coinciden en que la mayor intención de compra es para el producto casero con logo; sin embargo, el efecto de las variables estudiadas (tipo de producto, información y logo) sobre la percepción de saludable y amigable con el medio ambiente es diferente. El producto casero con logo es el que tiene mayor percepción de saludable y amigable con el medio ambiente en ambas poblaciones. La alegación "fuente de fibra" aumenta también esas percepciones, aunque no la intención de compra en España.

- Para validar el nuevo ingrediente, Se formuló un flan fuente de fibra, con una sustitución del 30% de inulina por el ingrediente a base de naranja obtenido, logrando un producto similar a lo esperado de un flan y sin descriptores asociados a sabores no deseados. De hecho, se logra una mejora en la consistencia y en apariencia destaca el brillo obtenido.

- Al aumentar el contenido del ingrediente de naranja aumenta la percepción de sabores no agradables, cómo ácido y amargo. Por ello, será necesario trabajar en mejorar los procesos de extracción, ya sea disminuyendo temperaturas o tiempos (para reducir la formación de compuestos de caramelización), o estudiar pretratamientos para reducir o eliminar los componentes responsables de sabores amargos.

Conclusions

- It was possible to obtain a powder ingredient from orange industry by-product, through the use of different non contaminant extraction technologies. The ingredients' fibre content varied from 10 to 20% in the assayed treatments. These powders have a high content of simple sugars (glucose, fructose and sucrose) and bioactive compounds that could be from the by-product or be formed in the extraction processes.

- Extrusion + hot water treatment was the one with that extracted the highest amount of soluble dietary fibre, and least content of sugars. This indicates that the process is able to extract soluble fibre, without increasing the sugar content. Furthermore, this treatment was the one with the highest amount of bioactive compounds. This extraction procedure is simple and does not use contaminant solvents.

- The ingredients have techno functional properties of industrial application, such as foaming capacity, increasing viscosity without changing the flow behaviour, and high water solubility. Because of this, these ingredients have potential not only from the nutritional point of view but also from the technological one.

- Regarding storage stability, powders would not be stable in the conditions that these were obtained, as their moisture content is higher than the critical water content described by the sorption isotherm – glass transition relation. Because of this and to slow down the spoilage reactions, drying process should be improved or the encapsulant should be changed, to decrease the final moisture content and increase the glass transition temperature.

- When evaluating the ingredient's potential use, and how to communicate both the fibre's origin and a sustainability message, Spanish and Uruguayan populations exhibited different behaviours. Both populations have the highest purchase intention in the homemade product with a logo. However, the studied variables' effect (product's category, logo presence and fibre origin's claim) on environmental friendliness' and healthiness' perception is different. The homemade product, with the logo presence, is the one with the highest healthiness and environmental friendliness perception in both countries. The claim regarding the fibres' origin increases these perceptions, but not the purchase intention in Spain.

- To validate the new ingredient, a source of fibre flan-type dessert was formulated. The substitution of 30% of inulin content with the orange fibre ingredient gave a product similar to what is expected of this kind of desserts, without off flavours. In fact, there is an improvement in the dessert's consistency, and an increase in the shine of the product.

- When increasing the orange fibre content the off flavours perception increases, such as acid and bitter. Therefore, work must be done in extraction processes, whether decreasing temperatures or times (to reduce the caramelization processes) or studying pre-treatments to reduce or eliminate the bitter compounds.