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International Degree of Doctor in Food Science

Tiger nut powder as ingredient for obtaining gluten free foods based on noodle processing and extrusion technology

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CERTIFICA:

Que la presente Tesis Doctoral, titulada " **Tiger nut powder as ingredient for obtaining gluten free foods based on noodle processing and extrusion technology"**, ha sido realizada bajo su dirección en el Departamento de Ciencia de Alimentos del Instituto de Agroquímica y Tecnología de Alimentos, por NICOLA GASPARRE; y que, habiendo revisado el trabajo, considera que reúne las condiciones necesarias para optar al grado de Doctor Internacional en Ciencias de la Alimentación.

Y para que así conste a los efectos oportunos, se expide el presente escrito.

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This Ph.D. thesis has been carried out at the Institute of Agrochemistry and Food Technology (IATA) belonging to the Spanish National Research Council (CSIC). Part of the experimental work has been performed at the Western Regional Research Center (WRRC) within the Agricultural Research Service (ARS) of the United States Department of Agriculture (USDA) located in Albany, California.

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List of original publications

The present thesis is based on the following publications:

1. Gasparre N, Betoret E, Rosell CM (2019) Quality Indicators and Heat Damage of Dried and Cooked Gluten Free Spaghetti. Plant Foods Hum Nutr 74 (4):481-488. doi:10.1007/s11130-019-00765-3

2. Gasparre N, Rosell CM (2019) Role of hydrocolloids in gluten free noodles made with tiger nut flour as non-conventional powder. Food Hydrocolloids 97. doi: 10.1016/j.foodhyd.2019.105194

3. Gasparre N, Pan J, da Silva Alves PL, Rosell CM, De J Berrios J (2020) Tiger Nut (Cyperus esculentus) as a Functional Ingredient in Gluten-Free Extruded Snacks. Foods (Basel, Switzerland) 9 (12). doi:10.3390/foods9121770

Book chapter:

1. Gasparre N, Rosell CM (2020) Snacking: ingredients, processing and safety. Cereal-Based Foodstuffs: The Backbone of Mediterranean Cuisine. Ed. Fatma Boukid, 2020. Springer Nature Switzerland AG. Sent for publication.

Volli, e volli sempre, e fortissimamente volli.

Vittorio Alfieri

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Resumen

Tubérculos y raíces siempre han jugado un papel crucial en la dieta humana. Desde el Paleolítico forman parte de las dietas de los cazadores-recolectores y hoy en día también están añadiendo heterogeneidad a los hábitos alimentarios. Dirigiendo la mirada hacia España, se encuentra un tubérculo comestible poco conocido, pero muy apreciado en la Comunidad Valenciana, la chufa.

La chufa es un rizoma tuberoso natural de una planta comestible perenne similar a la hierba (*Cyperus esculentus L.*). Actualmente se cultiva principalmente en el Este de España (Comunidad Valenciana) y en África Occidental (Mali, Burkina Faso, Níger, Nigeria). En el pasado, fue ampliamente utilizado como alimento para animales. Hoy en día, los tubérculos de chufa se consumen principalmente crudos, tostados y, además, en España, se emplean primariamente para producir una bebida llamada horchata de chufa.

La composición de la chufa depende en gran medida de la variedad. De hecho, se han reportado diferencias significativas en la composición de la chufa del área de Valencia y África Occidental. Los tubérculos de la zona de Valencia tienen un valor energético estimado que oscila entre 400 y 413.8 kcal/100 g. Los carbohidratos son el componente principal que representa 45.05 g/100 g (materia seca). El almidón representa el 29.9% (materia húmeda). En la chufa valenciana, la cantidad de fibra dietética es de aproximadamente 9.31 g/100 g (materia seca) y el 99.8% de la cual está constituida por fracción insoluble. Una característica peculiar de la chufa es el contenido de grasa. Los ácidos grasos monoinsaturados son los más abundantes (72.61 g/100 g de ácidos grasos). En las muestras españolas, el contenido de proteínas es de unos 8.45 g/100 g (materia seca). El contenido total de cenizas es de alrededor de 1.97 g/100 g (materia

seca); el calcio y el fósforo son los minerales más abundantes y en el tubérculo se pueden encontrar cantidades menores de magnesio, hierro, zinc y cobre.

La creciente demanda de alimentos saludables y de origen vegetal está impulsando la chufa hacia una apreciación sobresaliente entre más y más consumidores. Así, la chufa además de como alimento ha comenzado a utilizarse para la obtención de aceites y cosméticos, almidón para aplicaciones industriales y harinas. Se han realizado numerosos estudios para valorizar los subproductos del proceso de producción de la *horchata* como potenciador nutricional y tecnológico de nuevos alimentos funcionales. Esta línea de investigación se ha aplicado principalmente para la mejora de la calidad de productos cárnicos, chips de trigo, galletas y pasta fresca a base de huevo.

El perfil nutricional señalado anteriormente y la ausencia natural de gluten han contribuido a la creciente atención que ha surgido en torno a la chufa, convirtiéndola en un candidato potencial para el desarrollo de productos sin gluten. La expansión del mercado de productos sin gluten ha motivado numerosos estudios para identificar el valor nutricional y el estado de salud de las personas que padecen la enfermedad celíaca. Los resultados han evidenciado que los productos libres de gluten son calóricamente más densos que los obtenidos con harina de trigo. Los alimentos sin gluten también son más ricos en contenido de grasas, especialmente grasas saturadas. En cuanto al contenido de proteínas, la principal ingesta en la dieta sin gluten proviene de origen animal, ya que la mayoría de las harinas de cereales son pobres en compuestos nitrogenados.

Está bastante claro que una dieta sin gluten desequilibrada puede llevar al sujeto celíaco a un estado nutricional perjudicial. Debido a esto, recientemente, la investigación alimentaria tiende hacia la mejora nutricional de los productos sin gluten. Para hacer frente a este importante desafío de salud pública, se han comenzado a tener en cuenta las materias primas no convencionales, como los cereales integrales sin gluten, los pseudocereales y las legumbres. Dada la composición nutricional de la chufa previamente mencionada, podría ser ingrediente interesante para la producción de alimentos sin gluten. Los pocos estudios encontrados en la literatura científica contemplando esta posibilidad se han centrado en el uso de la harina de chufa en panificación, producción de galletas y pasta fresca al huevo.

Gracias a su accesibilidad, sabor, durabilidad y facilidad de almacenamiento pasta y fideos representan los alimentos que forman parte de la dieta de los consumidores de todo el mundo. La pasta tradicional a base de trigo se diferencia de los fideos principalmente en las materias primas utilizadas para su producción. Los fideos de trigo se elaboran normalmente con harina de trigo blando e incluyen sal en sus formulaciones. Otros tipos de fideos incluyen harinas de arroz, maíz, legumbres o diferentes almidones en sus formulaciones. Por el contrario, en la pasta tradicional la sémola de trigo duro es el ingrediente principal. En cuanto a su proceso de producción, la pasta y los fideos comparten dos tecnologías principales: extrusión y laminado. Debido a su versatilidad y flexibilidad, el proceso más utilizado para la producción de pasta y fideos es la extrusión.

La industria alimentaria de cereales ha recurrido universalmente a esta tecnología porque permite crear productos de diversas formas, volúmenes y consistencia. Los principales beneficios de este proceso están representados por su adaptabilidad, eficiencia energética y producción a bajo costo. A través de una serie de pasos (mezcla, amasado, cocción, formación) la extrusión conduce a través de la combinación controlada de materiales y energía a un alimento dando lugar a operaciones de reorganización y plastificación. A lo largo de este proceso termo-mecánico, las principales modificaciones fisicoquímicas están a cargo del almidón y de las proteínas. De hecho, la extrusión promueve la gelatinización del almidón y la desnaturalización de las proteínas. Por ello, las harinas de cereales y otros vegetales se transforman en productos de valor añadido como pastas, fideos y snacks. A diferencia del proceso convencional de elaboración de la pasta, donde la función del gluten es de suma importancia, la producción de pasta sin gluten se basa completamente en la capacidad de gelatinización y retrogradación del almidón. Durante la cocción ocurren dos fenómenos principales: la gelatinización del almidón y la coagulación de las proteínas. En la pasta y fideos a base de trigo, la red de proteínas atrapa los gránulos de almidón gelatinizado evitando la pérdida de sólidos y dando un producto cocido con una pegajosidad superficial reducida y una textura apreciable. En el caso de la producción de fideos de arroz, se requiere calor para permitir la gelatinización del almidón. El proceso más empleado para la obtención de fideos de arroz comprende una primera etapa de mezcla de ingredientes (agua, harina de arroz y / o almidón) con la formación de una masa muy consistente. Luego, se extruyen en forma de fideos que se vierten directamente en agua hirviendo provocando la gelatinización del almidón. El enfriamiento en agua fría (5°C) favorece la retrogradación del almidón y la formación de la estructura. A diferencia de la pasta de trigo, la gelatinización y retrogradación del almidón son los pasos cruciales en la extrusión de los fideos de arroz. Este biopolímero actúa como agente estructurante gracias a la capacidad de sus macromoléculas (amilosa y amilopectina) para recrear nuevos reajustes durante la retrogradación.

A pesar de los numerosos estudios realizados con pasta sin gluten y del uso de diferentes materias primas para el enriquecimiento nutricional de esos productos, existe muy poca información sobre la calidad de los alimentos de pasta sin gluten comercializados. Además, debido a su alto contenido en grasa, el uso de harina de chufa en extrusión resulta ser muy limitado.

Objetivos y Metodología

El objetivo principal de esta investigación fue extender el uso de la chufa como ingrediente alimentario mediante el diseño de alimentos sin gluten basados en la tecnología de extrusión y procesamiento de pasta / fideos.

Para alcanzar el objetivo principal se definieron los siguientes objetivos específicos:

1. Evaluación de mercado de los productos de pasta sin gluten identificando indicadores de calidad relacionados con el desempeño tecnológico y la calidad nutricional.

El logro de este objetivo comprendía el análisis nutricional y tecnológica de las pastas sin gluten adquiridas en el mercado internacional comparándolas con un homologo preparado a partir de trigo duro. Se analizó el comportamiento durante la cocción a través de la evaluación del tiempo óptimo de cocción, absorción de agua, índice de hinchamiento y perdidas de solidos durante la cocción. La composición química de la pasta seca y cocida fue evaluada para conocer el aporte nutricional real al consumir pasta sin gluten.

El estudio del color y de la textura completaron el marco relativo a la calidad sensorial y tecnológica.

Dado que, durante la elaboración de la pasta sin gluten, específicamente durante el secado a altas temperaturas, se podrían producir compuestos indeseables, la evaluación del daño térmico fu llevada a cabo a través de la cuantificación de las furosinas en muestras antes y después de la cocción. Hasta ahora, en literatura científica no se han encontrado estudios sobre el daño por calor en la pasta sin gluten antes y después de la cocción.

2. Elaboración de fideos a base de harina de chufa sin gluten aplicando conocimientos sobre ingredientes y procesamiento, con especial énfasis en el papel de los hidrocoloides como agentes estructurantes e hidratación de los productos finales.

Con ese propósito, se seleccionaron los fideos como una posible matriz alimentaria para ser elaborados con chufa. Dada la ausencia de gluten en la harina de chufa, fue necesario identificar un agente estructurante que ayudara a alcanzar la consistencia adecuada de los fideos. Para el diseño de productos sin gluten, los hidrocoloides se han ampliamente usados principalmente debido a su comportamiento como espesantes, emulsionantes, estabilizadores, agentes espumantes, mejoradores de la retención de agua y textura. En la mayoría de los casos estos productos suelen elaborarse de forma empírica mezclando ingredientes sin gluten sin conocer su función en la matriz alimentaria. Para ello y para proporcionar una cierta extensibilidad, se utilizó una selección de diferentes hidrocoloides (goma guar, goma xantana, inulina y carboximetilcelulosa con distintas viscosidades) para la fabricación de fideos sin gluten basados totalmente en harina de chufa. Debido a su diferente capacidad de retención de agua, los efectos de los hidrocoloides sobre la hidratación en la fabricación de fideos sin gluten ciertamente merecían ser investigados a fondo, ya que el agua tiene un papel crucial en la definición de la consistencia de la masa de fideos. Por este motivo, la evaluación de dos niveles de hidratación distintos, constantes habitualmente empleados en la literatura y adaptados a las solicitudes de la matriz alimentaria, permitió comprender las diferencias tecnológicas encontradas entre los distintos conjuntos de muestras. Las masas fueron sometidas a ciclos de calentamiento y enfriamiento para evaluar las propiedades termo-mecánicas y estudiar el efecto de las distintas hidrataciones e hidrocoloides. Las muestras de fideos fueron evaluadas desde un punto de vista nutricional y tecnológico mediante el estudio del comportamiento en cocción y el análisis de los atributos de color y textura.

3. Comprender el mercado de los snacks y el estado del arte en el desarrollo de productos para definir el posible uso de chufa en el diseño de nuevos alimentos de conveniencia.

Actualmente, los snacks representan un segmento de alimentos en constante crecimiento debido a que está profundamente asociado al comportamiento de los clientes. Por ello, se llevó a cabo un minucioso examen de los snacks a base de cereales originarios de la zona mediterránea. La cuenca mediterránea es reconocida por su rica historia gastronómica y los efectos beneficiosos asociados a ella. En toda la zona mediterránea, "snack" se refiere principalmente a una pequeña ración de comida consumida en un corto período de tiempo (comer estando de pie) entre las comidas principales (desayuno, almuerzo y cena), con el fin de reducir la sensación de hambre hasta la siguiente comida regular. La rica gastronomía que caracteriza a la zona mediterránea se extiende también al concepto de snacks. Sin embargo, a pesar de la variedad de alimentos que se pueden consumir como snacks, se analizaron los snacks basados en cereales. Específicamente, en el área mediterránea, una variedad de granos como trigo, maíz, avena y arroz se han utilizado típicamente como ingredientes para snacks, y en una proporción bastante menor otros como centeno, sorgo y mijo. Además, en los últimos quince años, la industria alimentaria ha comenzado a incorporar pseudocereales y legumbres al maíz y al arroz para producir snacks sin gluten, uno de los nichos alimentarios en crecimiento. Se analizaron sus procesos de producción, interacciones de ingredientes, características de calidad y aspectos de seguridad, con especial énfasis en el proceso de extrusión por su importancia en el mercado de los snacks. Asimismo, se presentó un panorama de las tendencias que impulsan la innovación en snacks, especialmente en relación con la búsqueda de ingredientes alternativos y nuevas tecnologías aplicadas a esta categoría de alimentos que es muy propensa a adaptarse a un futuro en constante cambio.

4. Explorar el uso de la chufa como ingrediente de alimentos extruidos, adaptando recetas y ajustes de proceso para superar el inconveniente del contenido de grasa de la chufa.

Para alcanzar este objetivo se elaboraron snacks extruidos sin gluten a partir de harina de chufa, arroz y fibra soluble evaluando el efecto de la extrusión sobre las cualidades físicas y microestructurales, la composición nutricional y la capacidad antioxidante.

Se llevaron a cabo estudios preliminares para obtener una harina de chufa con un adecuado tamaño de partícula para la extrusión. Para superar el problema relacionado con su alto contenido de grasa y fibra, se usaron dos molinos diferentes y hielo seco. Se evaluó la composición química y la viscosidad aparente de las muestras antes y después de la extrusión para poder averiguar el efecto de la incorporación de harina de chufa y del proceso termo-mecánico.

El análisis de los parámetros físicos como la relación de expansión, diámetro, densidad aparente, porosidad y textura permitió caracterizar los productos extruidos clasificándolos según los criterios de aceptabilidad.

A través de la evaluación de la microestructura se explicaron las diferencias entre las muestras debidas principalmente al distinto contenido de harina de chufa.

Los compuestos fenólicos solubles totales y la capacidad antioxidante total fueron cuantificados para evaluar el aporte de la harina de chufa en términos de enriquecimiento nutricional.

Resultados y Discusión

1. Evaluación de mercado de los productos de pasta sin gluten identificando indicadores de calidad relacionados con el desempeño tecnológico y la calidad nutricional.

Los resultados del análisis de las formulaciones de las pastas sin gluten mostraron que las harinas de maíz y arroz predominaron entre los ingredientes de las muestras sin gluten, mientras que se utilizaron mono y diglicéridos de ácidos grasos en 3 muestras. Otros ingredientes menores fueron la quinua, el arroz integral y la harina de mijo. La comparación con su homólogo a base de trigo mostró una deficiencia en el contenido de proteínas y cenizas en la mayoría de las muestras crudas y cocidas.

La evaluación de los diferentes indicadores de calidad (tiempo óptimo de cocción, perdidas de sólidos, absorción de agua y índice de hinchamiento) permitió analizar las diferencias entra las pastas de trigo y las sin gluten. Las muestras sin gluten mostraron mayor tiempo óptimo de cocción que el control de trigo. Se observaron diferencias significativas entre la cantidad de pérdidas de sólidos durante la cocción obtenida con las distintas muestras. En general, las muestras con emulgentes mostraron menores perdidas que las muestras solo compuestas por maíz, estas últimas fueron las que liberaron más sólidos en el agua de cocción. Esta falta de retención de componentes fue causada por la ausencia de gluten que es responsable del atrapamiento de sólidos. Por lo tanto, la pasta sin gluten no tuvo una estructura cohesiva, la cual mejoró en muestras que contenían emulgentes. La textura estuvo muy influenciada por el tipo de harina, y solo el maíz proporcionó mayor resiliencia, elasticidad y firmeza.

Las estrategias tecnológicas para simular la red de gluten y las altas temperaturas (> 75 a 100°C durante 2 a 3 h) alcanzadas para el secado de las pastas pueden favorecer la reacción de Maillard y la liberación de melanoidinas como productos finales. En las primeras etapas de esta reacción, se pueden desarrollar compuestos de Amadori (fructosil-lisina, lactulosil-lisina, y maltulosil-lisina), que generan furosinas (ε-N-furoilmetil-L-lisina). Estos compuestos se han definido como buenos marcadores del daño por calor durante el procesamiento de la pasta, así como un indicador del valor nutricional y de la seguridad de estos productos. La pasta sin gluten mostró un contenido de furosinas más bajo que el control a base de trigo y el proceso de cocción tendió a aumentar el nivel de furosinas en todas las muestras analizadas.

2. Elaboración de fideos a base de harina de chufa sin gluten aplicando conocimientos sobre ingredientes y procesamiento, con especial énfasis en el papel de los hidrocoloides como agentes estructurantes e hidratación de los productos finales.

Relativamente a los resultados de los fideos obtenidos a partir de harina de chufa, cuando el nivel del agua se mantuvo constante, las muestras con hidrocoloides tuvieron mayor consistencia. Esto se explicó por su gran habilidad para ligar el agua. De hecho, después del ajuste del agua, las diferencias disminuyeron. La goma xantana y la goma guar fueron los hidrocoloides que requirieron mayor cantidad de agua para corregir la consistencia de la masa. Como resultado, las masas con diferente reología generaron fideos frescos y cocidos que diferían en términos de propiedades de textura. Los hidrocoloides fueron los responsables del aumento de diámetro, mejorando la dureza y la firmeza. La mejora de la estructura causó la reducción de las pérdidas de sólidos durante la cocción. Por lo tanto, los fideos preparados con harina de chufa, con el 0.5% de goma xantana y un nivel de hidratación optimizado presentaron mejores rendimientos de cocción y atributos de textura final. El color se vio afectado de manera diferente por los hidrocoloides, observando una disminución de la luminosidad, aunque solo significativa cuando se adaptó la hidratación y en presencia de goma guar, goma xantana y carboximetilcelulosa A15. Además, los parámetros relativos a las tonalidades amarillas y rojas fueron significativamente influenciadas por los hidrocoloides, particularmente, la goma xantana tuvo los valores más altos en términos de tonalidad roja, mientras que la goma guar condujo a una menor tonalidad roja pero mayor tonalidad amarilla. La dureza y la firmeza se vieron afectadas significativamente por el nivel de hidratación e hidrocoloides. En general, la dureza, la fuerza necesaria para lograr una deformación, fue más alta en los fideos con hidratación constante, después de ajustar el agua se obtuvieron fideos más suaves. Cuando se agregaron hidrocoloides, la dureza aumentó, especialmente cuando se utilizó carboximetilcelulosa A15 (independientemente de la hidratación) y goma guar. La firmeza, la fuerza requerida para cortar el fideo, fue mayor en fideos con hidratación adaptada, excepto los que contienen inulina. La goma xantana fue el hidrocoloide que confirió mayor firmeza. La adhesividad, la fuerza necesaria para superar las fuerzas atractivas entre la superficie del fideo y la superficie de los dientes, fue significativamente reducida en presencia de hidrocoloides, excepto en aquellos que contenían goma xantana e hidratación constante. La masticabilidad o la energía necesaria para disgregar y reducir los fideos en un estado adecuado para la deglución aumentaron significativamente en todas las muestras con hidrocoloides.

3. Comprender el mercado de los snacks y el estado del arte en el desarrollo de productos para definir el posible uso de chufa en el diseño de nuevos alimentos de conveniencia.

Para expandir la aplicación de la chufa a otros productos GF, el enfoque se dirigió a los snacks. Actualmente, representan un segmento de alimentos en constante crecimiento debido a que está profundamente asociado al comportamiento de los clientes. Fueron considerados los principales snacks a base de cereales principalmente del área mediterránea, explicando su proceso de producción, interacciones de ingredientes, características de calidad y aspectos de seguridad, con especial énfasis en el proceso de extrusión por su importancia en el mercado de snacks. El análisis realizado sobre el mercado existente de snacks reflejó la continua innovación en este tipo de productos, sobre todo dirigida a la mejora nutricional y la seguridad alimentaria.

4. Explorar el uso de la chufa como ingrediente de alimentos extruidos, adaptando recetas y ajustes de proceso para superar el inconveniente del contenido de grasa de la chufa.

Últimamente se están procesando nuevas harinas alternativas mediante la tecnología de extrusión para el desarrollo de nuevos snacks expandidos sin gluten. En el caso de la chufa, las formulaciones diseñadas incluyeron diferentes niveles de la harina del tubérculo (10, 30, 50, 70%) como ingrediente no convencional y arroz y fibra soluble para mejorar la expansión. Su incorporación, produjo una disminución de la viscosidad máxima y de la viscosidad mínima como resultado de la cocción por extrusión que proporcionó una gelatinización del almidón. Los diámetros, las relaciones de expansión, las densidades reales y los volúmenes de poros disminuyeron progresivamente como consecuencia del aumento del contenido de chufa en las muestras. Las formulaciones conteniendo 50 y 70% de harina de chufa obtuvieron los valores más bajos en términos de diámetro y relación de expansión. En general, con el aumento de la harina de chufa en las formulaciones se observó un aumento de la densidad aparente de los extruidos. En particular, entre el control y la muestra con el 10% de harina de chufa hubo un ligero aumento que fue aún más pronunciado entre las muestras con el 30 y 70% de chufa. El análisis de la estructura de la superficie de los extruidos mostró que, en general, la chufa contribuyó a hacer la superficie más suave y menos uniforme. Se observó un impacto de la harina de chufa en las características de textura de las muestras extruidas; de hecho, a medida que aumentaba su incorporación, el contenido de almidón disminuía y se notó una reducción de la firmeza. Esta sección del estudio confirma la idoneidad de la incorporación de harina de chufa (10%) para el desarrollo de snacks sin gluten como una nueva alternativa para los celíacos. Desde el punto de vista nutricional, el uso de chufa para la producción de snacks sin gluten fue responsable del aumento del contenido de cenizas, proteínas y grasas con respecto al control del arroz. El contenido total de fenoles solubles y la capacidad antioxidante total aumentaron con la introducción de harina de chufa, mientras que la alta presión, fuerza y temperatura alcanzadas durante la extrusión provocaron su disminución.

Conclusiones

La investigación realizada a través de los diferentes capítulos permite concluir que la harina de chufa podría utilizarse como ingrediente alternativo en el diseño de fideos sin gluten y productos extruidos, después de definir los agentes estructurantes y los ajustes de extrusión adecuados, respectivamente.

En particular, se pueden destacar las siguientes observaciones finales:

- Hubo diferencias significativas en la composición nutricional entre los espaguetis sin gluten y la pasta de trigo duro. En general, las muestras sin gluten en ambas etapas (secas y cocidas) fueron más pobres en proteínas y cenizas con respecto a la pasta de trigo. En cuanto a los indicadores de calidad, las muestras sin gluten mostraron un comportamiento significativamente diferente y no pudieron relacionarse con ingredientes específicos. Sin embargo, la pasta hecha con harina de maíz requirió una cocción más prolongada y tuvo altos valores de pérdida de cocción, pero resultó en una pasta más resistente y elástica. El contenido de furosinas en la pasta seca sin gluten fue mucho más bajo que en la pasta de trigo, y esas diferencias aumentaron enormemente después de la cocción.

- Los fideos sin gluten podrían elaborarse con harina de chufa, prestando especial atención a la cantidad de agua utilizada en el proceso de producción y al tipo de hidrocoloide agregado, ya que juegan un papel crucial en la reología de la masa y la calidad de los fideos frescos y cocidos. En general, los fideos de chufa sin gluten elaborados con goma xantana y cantidad adaptada de agua mostraron la mejor calidad considerando las pérdidas de cocción más bajas obtenidas y su mayor firmeza.

- El segmento de mercado de snacks representó una buena oportunidad para lanzar productos innovadores con mayor calidad nutricional y seguridad alimentaria. Los comportamientos del cliente y la introducción de nuevas tecnologías alimentarias representan un muy buen activo para triunfar en el segmento de snacks. En particular, la inclusión de nuevos ingredientes que conducen a productos novedosos con texturas atractivas y percepciones sensoriales.

- La harina de chufa podría usarse para producir nuevos snacks extruidos sin gluten, lo que brinda una alternativa atractiva para los consumidores con enfermedad celíaca. El análisis de la viscosidad aparente mostró que la inclusión de chufa, en las harinas formuladas a base de arroz, aumentó la temperatura de inicio y retrasó la viscosidad máxima en las harinas extruidas. La adición progresiva de harina de chufa en las formulaciones promovió una reducción en el diámetro, la relación de expansión, la densidad real y el volumen de poro total en los extruidos, mientras que aumentaron sus densidades aparentes. Además, la incorporación de chufa fue responsable de un aumento en el contenido de cenizas, proteínas y fenoles totales, lo que es un valor agregado al snack desarrollado. Los extruidos con el 10% de harina de chufa en la formulación mostraron el mejor perfil de textura general.

Abstract

Tiger nut (*Cyperus esculentus L.*) is a sweet tuber mostly cultivated in Eastern Spain (Valencia) and in west Africa. Its nutritional profile stands out for the high fiber and unsaturated fat content; moreover, the moderate protein amount might make it suitable for the nutritional enrichment of gluten free foods. The objective of this thesis was to extend the applications of tiger nut as gluten free (GF) ingredient in noodles making and snacks. The study of marketed GF pasta revealed the nutritional inadequacy and lower cooking performances compared with their gluten containing homologous. The quantification of furosine content was handy for a better understanding of the heat damage caused during the production process. At this point, tiger nut flour was utilized for the design of gluten free noodles. The effect on noodles physicochemical characteristics leaded by different hydrocolloids (guar gum, xanthan gum, inulin and carboxymethyl cellulose) and by differing hydration levels revealed the importance in the selection of the most appropriate strengthening agent. Specifically, samples with 0.5% xanthan gum and adjusted hydration level exhibited the best cooking behavior and high final firmness. Owing to the growing extruded snacks success, tiger nut flour was blended with rice and soluble fiber to evaluate its technological adequacy for the extrusion-cooking process. Apparent viscosity, structural and surface characteristics of the gluten free snacks were deeply influenced by tiger nut addition. An improving of the nutritional profile, total soluble phenolics content and the total antioxidant capacity were found in the samples with tiger nut. Overall, it was possible to obtain GF foods containing tiger nut powder by applying different processing alternatives.

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Abbreviations

TN	Tiger Nut
GF	Gluten Free
MR	Maillard Reaction
FUR	Furosine
OCT	Optimal Cooking Time
CL	Cooking Loss
WA	Water Absorption
SI	Swelling Index
TPA	Texture Profile Analysis
PCA	Principal Analysis Component
XG	Xanthan Gum
GG	Guar Gum
CMC	Carboxymethyl cellulose
SF	Soluble Fiber
RVA	Rapid Visco Analyzer
SEM	Scanning Electron Microscope
ER	Expansion Ratio
BD	Bulk Density
TSP	Total Soluble Phenolics
TAC	Total Antioxidant Capacity

1. Introduction

Tubers and roots have always played a crucial role in human diet. Ever since Paleolithic, they have been part of the hunter–gatherers' food preferences and nowadays they also add heterogeneity to the eating habits [1]. Turning the analytical gaze towards Spain, there is a little-known edible tuber, which is widespread in the traditional food of the Valencian Community, the tiger nut.

Tiger nut (TN) (Figure 1 and 2) is a naturally occurring tuberous rhizome of an edible perennial grass-like plant (*Cyperus esculentus L.*) [2]. Despite his wrinkled crunchy texture, it does not fall within the nut group. Currently is widely cultivated mainly in Eastern Spain (Valencian Community) and in West Africa (Mali, Burkina Faso, Niger, Nigeria). It has ancient origin; in fact, in V century BC, Herodotus of Halicarnassus called TN as "*biblo*" in his work "*Histories*". Subsequently, in IV century BC Theophrastus described TN in "*Historia Plantarum*" as an eatable plant grown in the Nile Valley closely related to the papyrus from which a round shape and soft consistence fruit was obtained [3]. It is unknow how TN arrived in Europe. Some authors link it to the Arabs that during the Middle Age introduced it in Spain and some others agree that it was already being grown in Valencia long before that. In 1795, Antonio José Cavanilles y Palop, through his book "*Observaciones sobre la Historia Natural, Geografía, Agricultura, población y frutos del Reyno de Valencia*", explained in detail the TN cultivation in the Spanish towns of *Alboraya* and *Almácera* [4].

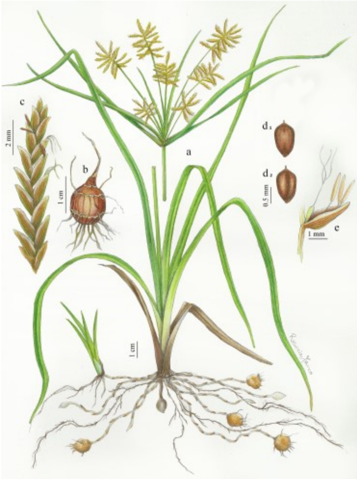

Figure 1: *Cyperus esculentus L.*: (a) habit of the flowering plant; (b) mature tuber; (c) spikelet; (d1) achene: dorsal view; (d2) achene: ventral view; (e) details of flower and rachilla. Figure adapted from Follak et al. [5]

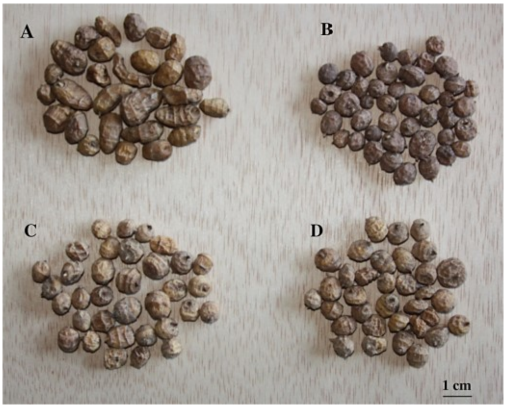

Figure 2: Comparative appearance of tiger nuts from Valencia (A); Niger (B) and Burkina Faso (C, D). Figure adapted from Codina-Torrella et al. [2].

Depending on the framework and owing to its invasive capability and ecological plasticity, TN is classified as a crop or as a weed [6]. In the past, it was widely used as animal feed. Today, TN tubers are primarily consumed raw, roasted and moreover in Spain, they are mainly employed to produce a cold milky-looking beverage named "*horchata de chufa*". In 2018, the consumption of Spanish TN from Valencia north area (*Horta Nord*) was about 2,9000,000 kg, 13% more than the previous harvest year [7]. Most of the TN produced in this area is regulated and protected by the designation of origin "*Chufa de Valencia*" identifiable by the distinctive logos (Figure 3 and 4) [8].

Figure 3: Designation of origin logo "Chufa de Valencia"

Figure 4: Designation of origin European logo "Chufa de Valencia"

1.1 Nutritional Value of Tiger Nut

TN composition is greatly dependent on the variety; in fact, it has been reported significant differences in the proximate composition of TN from Valencia area and Western Africa [2].

Nevertheless, the following explanation is referred to the TN cultivated in Valencian area. TN tubers from Valencia area have a length varying from 10 to 14.37 mm and a thickness from 8.21 to 9.05 mm [2]. Generally, they have an estimated energy value ranging from 400 to 413.8 kcal/100 g [9]. As reported in table 1, carbohydrates are the major component accounting for 45.05 g/100 g (dry matter) [2]. Starch represents 29.9% (wet matter) [10]. In Valencian TN, dietary fiber amount is about 9.31 g/100 g (dry matter) [2] and 99.8% of which consists of insoluble fraction [11]. Considering its high fiber content, TN could be an ally against several diseases including colon cancer, coronary heart disease, obesity, diabetes and gastrointestinal disorders [12] [13] [14]. Sucrose content is of particular relevance, in fact its quantity is about 13.03 g/100 g [10].

A peculiar characteristic of TN is the fat content. The Valencian variety is characterized by a 35.21% (dry matter) [2]. Monounsaturated fatty acids are the most abundant (72.61 g/100 g of fatty acids), while saturated fatty acids content amounts to 19.01 g/100 g of fatty acids and the polyunsaturated class represents 8.39% of total fatty acids (Figure 5) [15]. Under a fatty acids profile point of view, TN oil can be considered like olive oil. In fact, in TN, oleic acid is the predominant fatty acid (72.39%) followed by palmitic (15.76%), linoleic (8.22) and stearic (2.57%) acids [15]. Plenty of oleic acid could impart a pivotal role to TN in the prevention of particular forms of cancer, blood pressure control, cardiovascular diseases, cell membrane fluidity and immune response [16]. Several phytosterols naturally occur in TN: β-sitosterol (601 μg/g), stigmasterol (195 μg/g), campesterol (140.1 μg/g), α-tocopherols (4.6 μg/g) [15]. In the Spanish samples, protein content is about 8.45 g/100 g (dry matter) where the prevalent fraction corresponds to the water-soluble fraction of albumin and non-protein nitrogen (91.93%) while 4.72% is covered by the insoluble residue [2]. Total ash content is around 1.97 g/100 g (dry matter) [2]; calcium and phosphorus are the most plentiful minerals, and a smaller quantity of magnesium, manganese, iron, zinc, and copper can be found in the tuber [17].

Certain studies about TN quercetin and vitamin E reported possible cellular protection effects against free radicals and inhibition of the proliferation at the G1 phase of cancer cells [18] [19]. Research carried out by Oyedepo, F [20] highlighted the hepato-protective activity of TN against carbon tetrachloride action in rats. An *in vitro* study demonstrated the TN anti-inflammatory and immuno-stimulatory properties in apolipoprotein deficient mice [21]. Those studies confirmed the healthy contribution of tiger nut.

The presence of some antinutritional compounds like tannins and phytates has been reported in Valencian tubers. These compounds are largely recognized to decrease the absorption of some nutrients in the intestinal section. For example, lipase and amylase inhibition by tannins, causes a decrease of protein absorption while some mineral absorption (magnesium and calcium) may be decreased by the phytates chelating action [2]. Other than the antinutritional action, these compounds may also provide an antimicrobial activity [10]. Samples from Valencia contains 0.08 and 16.27 g/100 g (dry matter) of tannin and phytate, respectively. In addition to these, another study highlighted the presence of alkaloids and saponins in TN tubers [18]. Authors reported that soaking and roasting significantly reduce the antinutritional compounds content in TN tubers [18].

Table 1: Nutritional composition of Tiger Nut tuber from Valencia

Moisture (g/100 g)	Carbohydrate (g/100 g)	Fiber (g/100 g)	Fat (g/100 g)	Protein (g/100 g)	Ash (g/100 g)	Reference
8.66 ± 0.04	45.05 ± 3.13	9.31 ± 0.09	35.21 ± 3.07	8.45 ± 0.20	1.97 ± 0.01	Codina-Torrella et al. [2]

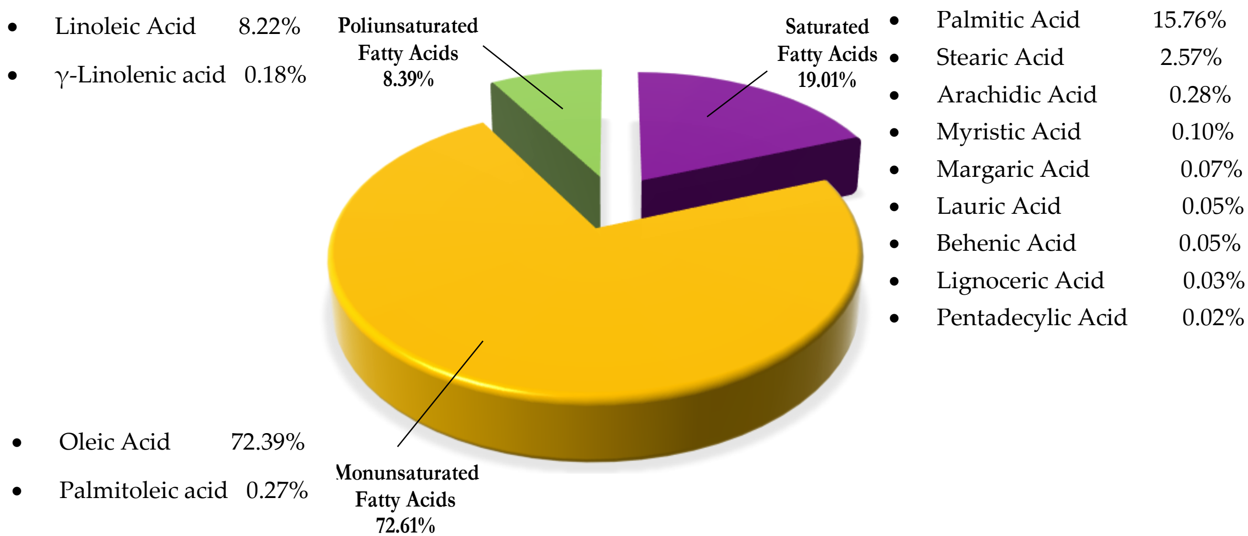

Figure 5: Tiger Nut from Valencia fatty acids profile as reported by Lopéz-Cortés et al. [15]

1.2 Tiger Nut as Food Ingredient in New Products Development

The growing request for healthy and plant-based foods is propelling TN towards an outstanding appreciation among more and more consumers. Thus, TN is no longer used only as a feeding stuff [22] but new products TN-based have started to be created. TN have been exploited to give birth to oil and cosmetic products [23], starch for industrial applications [24] and alternative flours [25]. A substantial number of studies have been carried out to valorize "*horchata*" process by-products as a nutritional and technological booster for new functional foodstuffs elaboration. The result of "*horchata*" production is represented by a residual paste that can be pressed and filtered until obtain a solid and liquid waste. Solid waste is principally characterized by a high level of insoluble fiber while polyphenolic compounds and prebiotic activity defined the fluid co-product [10]. This research line has been applied mainly for quality enhancing of meat products. Indeed, TN fiber was integrated at increasing levels (5%, 10% and 15%) in the blends for pork burger production, resulting to be acceptable by the panelists [26]. A better control of lipid oxidation was observed when TN solid residue was employed (1% to 2%) in dry-cured sausages production [27]. Liquid co-product was used as a water replacer in pork burgers improving cooking yield and texture properties [28]. In beef burger, the replacement of beef fat by TN oil (50 and 100%) allowed the achievement of lower total and saturated fat product with similar texture as the control one [29]. Regarding baked goods, Alava et al. [30] studied the effect of TN solid co-product enrichment (5, 10, 20%) into wheat chip production. Reduced hardness and color changing were the main differences observed, while the sensory test obtained acceptable score in all the samples. Several authors have utilized TN in the form of powder for novel products elaboration characterized by improved nutritional and physiochemical properties. Omoba et al. [31] divulged that biscuits made by pearl millet and TN flour showed a high total phenolic compounds and antioxidant activity and resulted acceptable after the sensory test.

Non-conventional TN powder has been successfully employed to understand its effects in pasta products as well. In fresh wheat pasta eggs-based dough, TN flour (20 and 40%) and xanthan gum modified the rheological characteristics. Due to a weaker and less elastic gluten network formation, pasta seemed to have less consistent structure and lower hardness compared to the wheat control. This breakable structure caused an easier hydration capacity during cooking which provoked a reduction of the optimal cooking time. The xanthan gum action markedly improved the rheological behavior of the dough. The thermal study of the sample with highest TN concentration showed two phase transitions, one associated to wheat starch and the other one probably related with TN starch or amylose-lipid complexes [32]. TN added (20 and 40%) to dry wheat pappardelle enhanced the nutritional profile in terms of dietary fiber, mineral content, oleic and linoleic acids. Textural qualities such as elasticity, hardness, softness and homogeneity were lower in the sample containing the highest TN amount. Therefore, xanthan gum gave higher firmness to the samples, which was even maintained after cooking. Water absorption and cooking loss resulted to be higher in the sample with highest TN content that led to the most appreciated sample during the sensory test [33]. Albors et al. [34] confirmed the trend found in the previous analyzed study. Generally, fresh egg tagliatelle made by durum wheat semolina and containing highest replacement TN level (30%) had higher stickiness, cooking loss, water absorption and swelling index, and lower texture attributes (firmness, hardness and cohesiveness). Sample with TN was higher in fiber, fat content and in terms of color they resulted darker. Precisely this last factor associated with textural characteristics seemed to be the main cause of the overall sensorial acceptance.

1.3 Tiger Nut as a Novel Ingredient in Gluten Free products

Nutritional profile above pointed out, and the natural absence of gluten have contributed to the growing attention that have sprung up around TN, making it a potential candidate in the gluten free (GF) products manufacturing.

Nowadays, the life-long adherence to a gluten free diet continues to be the mainstay in the treatment of celiac disease [35]. Gluten is composed by a prolamin and glutelin combination that represents about 80% of the total protein of cereals like wheat, rye, barley and oat. Gliadin (prolamin) are ethanol-soluble while glutenin (glutenin) dissolve in weak acid solutions [36]. In cereal processing, gluten three-dimensional protein network is developed when these two proteins are hydrated and subject to mechanical force during mixing and kneading. In fact, they play a pivotal role in the food industry since they are responsible for the doughs rheological properties. Gliadin gives plasticity and glutenin brings elasticity and strength to the dough. Their synergy provides the peculiar characteristics to staple food such as pasta, bread and other baked products [37]. Owing to its well-known properties, such as water binding and viscosity yielding, gluten is widely used also as a food additive [38].

However, some peptides from both gluten proteins seem to be the celiac disease trigger factors even though an efficient epitopes classification is still missed [39]. Some authors have reported that some peptides from α and ω-gliadin would cause the gluten-related disorders in adults while the ones from glutenin and γ-gliadins would be responsible for the adverse effects on children [40][41].

Usually, celiac disease refers to an autoimmune chronic enteropathy which affects genetically predisposed subjects [42]. Small intestine mucosa is the first involved organ with the appearance of structural alterations, described as villous atrophy (villi flattening) and crypt hyperplasia (crypts elongation) (Figure 6) with consequent signs of malabsorption [35]. Nevertheless, celiacs may have either extraintestinal symptoms or may remain asymptomatic [43]. Celiac disease registered prevalence is approximately equal to 1% of the general population even though most cases are still unknown [44]. Currently the disease is rising in fact, in the United States, between the years 1975 and 2000, prevalence increased fivefold [45].

Gluten-related disorders group also includes other different pathological manifestations known as non-celiac gluten sensitivity or non-celiac wheat sensitivity, dermatitis herpetiformis, wheat allergy and gluten ataxia [46].

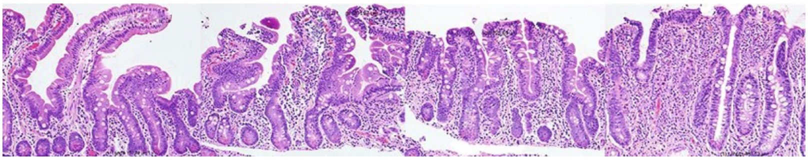

Figure 6: Mucosal sections. Normal villi from far-left side to villous atrophy in far-right side. Figure adapted from Lindfors et al. [35]

Up to now, the efforts made by food industry have permitted to bring GF products closer to the regular ones, mainly in terms of technological quality.

Consumers' buying patterns are changing principally due to the adopting of new healthy diets that lead to follow GF diet under different beliefs. Therefore, GF global market is experiencing a real boom nowadays. In fact, global GF products market size was estimated at USD 21.61 billion in 2019 and is projected to reach USD 43.65 billion by 2027 growing at a CAGR of 9.2% during the forecast period of 2020 to 2027 [47]. Reaching an income of USD 5.64 billion, bakery products was the large segment in 2019 and, following the predictions, it is expected to expand at a CAGR of over 10.2% over the forecast period [48].

Simultaneously to this expansion of GF products market, several studies has been conducted to investigate their nutritional value and the health status of the people suffering for celiac disease. In a study carried out by Lasa et al. [49] about comparison between GF products (flours, breads, bakery products, pasta, cereals, cookies, and snacks) and their analogues gluten containing ones, no differences in terms of energy value were found in most of them. Nevertheless, GF flours, breads, and pizza dough resulted calorically denser than the wheat-based ones, because GF products are mainly starchy foods [50,51]. GF foods are also richer in fat content, especially saturated fats. This can be explained because the GF formulations frequently include lipid-based ingredients like animal or vegetable oils and emulsifiers (mono- and diglycerides of fatty acids) for the palatability improving [52]. The lack of whole grain cereals in GF pasta ingredients was mirrored by a lower amount of final product dietary fiber [53]. However, recently, the introduction of non-conventional flours (pseudocereals) and hydrocolloids in the GF formulations, turned to improve the patterns of GF pasta and GF bakery products [54]. Regarding protein content, the main intake in GF diet comes from animal source since most common cereal flours are poor in nitrogenous compounds [55].

It is quite clear that an unbalanced GF diet may drive the celiac subject to a harmful nutritional status. Due to this, recently, food research is moving towards the nutritional improvement of GF products. In order to meet this important public health challenge, unconventional raw materials such as whole meal GF cereals, pseudocereals and pulses have started to be taken into account [56][57][58]. Few studies in scientific literature took up the subject of TN in GF products processing.

In the research lead by Aguilar et al. [59], TN flour, TN milk and TN milk by-product were used as substitutes (5%) of soy flour in GF bread formulations maize starch based. TN milk breads had the softest crumb, highest loaf-specific volume and it was the most appreciated by the tasters. No differences were observed between breads made by TN flour and the ones with soy flour; nevertheless, they showed different color and crumb structure. TN milk by-product produced hard and dark crumb breads that received the lowest score in terms of consumers' preferences.

The effect of the shortening levels reduction (5, 2.5 and 0%) and emulsifier (2, 1 and 0%), were investigated in chickpea and TN flours, separately and combined for GF breadmaking [60]. TN flour led to a reduced bread specific volume and darker crumb. In the sample composed by a blend of TN and chickpea flour, the absence of emulsifier did not affect the crumb texture thanks to the interactions between chickpea protein and tiger nut lipids.

Increasing TN flour concentrations (0, 5, 10, 15, 20, 25%) were incorporated in rice flour GF breads baked in infrared–microwave combination and conventional ovens [61]. Samples with TN reached the gelatinization at higher temperatures. Breads with 10% TN conventionally baked had similar texture, volume and color to the ones subjected to an infrared–microwave baking, that contained 20% TN.

TN flour has been used as ingredient (10, 20, 30%) for GF maize-based biscuits preparation [62]. From a nutritional point of view, TN flour helped to boost fiber and ash content. Thermal analysis revealed a decrease of the onset gelatinization and peak temperature of the blend due to TN flour addition. Samples containing TN flour had lower hardness and resilience, while the set of biscuits with 20% of TN flour appeared to have the best technological quality in terms of shape, cross section structure, hardness and surface appearance.

1.3.1 Gluten Free Pasta-like Products

Pasta and noodles have been part of worldwide consumers' diets for a long time thanks to their accessibility, tastiness, durability and their easy storing. The global pasta and noodles market was valued at USD 59.6 billion in 2016 and is expected to grow at a CAGR of 3.6% from 2018 to 2025 when it will reach USD 81.7 billion [63]. Traditional wheat-based pasta differs from noodles principally in raw materials utilized for their production. Wheat noodles are usually made by fine wheat flour and they include salt in their formulations [64]. Other types of noodles include rice, corn, pulses, or different starches in their formulations. On the contrary, traditional pasta is free from salt added and wheat durum semolina is the main ingredient. Regarding their production process, pasta and noodles share two principal technologies: extrusion and sheeting [64]. Because of its versatility and flexibility, extrusion has been not employed only to produce pasta-like products, but also in the manufacturing of breakfast cereals, snacks, pre-cooked flours, cereal-based baby food, and texturized proteins [65]. To better understand the extrusion application in the gluten free pasta field, it is only right to clarify a little the main steps of traditional pasta production. Durum wheat semolina is mixed by water until reach a 33-34% moisture content [66]. After 10-20 min of mixing, formed dough is conveyed into the vacuum extruder. Here, a rotating screw forces the dough towards the head press and by passing through a die, pasta takes the final shape. In this process, the interaction between glutenin and gliadin and the generation of the disulphide bonds are crucial for the gluten network development [67]. Obtained fresh pasta is dried until get a final 12.5% moisture content. After drying, gluten consistently takes position around the starch granules which still remain in the native form. During cooking two principal phenomena occur: starch gelatinization and protein coagulation. Indeed, protein network entraps the gelatinized starch granules preventing solid loss and giving a cooked product with reduced surface stickiness and appreciable texture [68]. Often, traditional extrusion pasta is named "cold" because the temperatures applied never exceed 35-50 °C in order to protect gluten [66]. In the case of rice noodles production, heating is required to allow the starch gelatinization. The most employed process to obtain rice noodles comprises a first stage of mixing ingredients (water, rice flour or/and starch) with a formation of a very consistent batter. Then, it is extruded in the form of noodles directly into boiling water causing the starch gelatinization and after a few minutes they are soaked immediately in cold water (5 °C) to promote the starch retrogradation. To improve the final quality a blasting cold air (-30 °C) may be applied [64]. In the last case, unlike wheat pasta, gelatinization and retrogradation of starch are the crucial steps in the extrusion of rice noodles. This biopolymer works as structuring agent by exploiting the ability of its macromolecules (amylose and amylopectin) to re-create new readjustments during retrogradation [68]. Based on these cycles of dough heating and cooling, the modern food industry set up the most utilized technology in GF pasta making. The global gluten-free pasta market was valued at $909.8 million in 2017 and is expected to reach $1,289.2 million by 2025, increasing at a CAGR of 4.5% from 2018 to 2025 [69]. GF pasta line differs from the traditional ones for the presence of steam injection into the extruder barrels and for the higher temperatures (more than 100 °C) adopted during the process [70]. GF pasta produced in that way resulted better in terms of texture and flavour compared with samples containing the same ingredients but processed by conventional extruder [71]. Among the technological way to improve the final quality of pasta-like products, annealing and heat moisture treatment must be considered. Annealing consists in the starch heating in excess of water (more than 50% w/w) above glass transition temperature but below gelatinization temperature (for rice 50-60 °C) [72] while heat-moisture treatment applies higher temperature (100-120 °C) for rice under limited moisture content (10-30%) [73]. During these physical processes, starch amorphous areas become glassier and the amylopectin double helices are arranged in a more organized manner [74]. Due to the inhibition of granule swelling, decrease of amylose leaching, and increase of paste stability, texture and cooking performances of rice noodles resulted enhanced [75]. The use of annealing is advisable for fresh rice noodles that require a soft texture while heat-moisture treatment that improve tensile strength and gel hardness is proper for dried noodles manufacturing [70]. Anyway, modern industry utilizes pregelatinized flours instead of the previous described heat treatments that are considered more expensive to manufacture pasta-like products. Moreover, pregelatinized flours can be used in the same conventional press for wheat durum semolina pasta, adopting some tricks such as the greater water amount required compared to the one needed for the traditional pasta making [76]. Some studies reported the obtaining of noodles with improved textural and cooking qualities following to the use of pregelatinized rice and corn flours, highlighting the importance of the gelatinization level and the cereal variety [77][78]. Gluten substitution in GF food pasta-like products design is still one of the most important challenges for cereals food technologists. Besides, GF pasta is generally intended for consumers that in most cases, have already consumed the conventional one and they are familiar with its qualities. Due to this, users' expectations regarding structure, texture and flavor are closely related to the ones of the containing gluten products [79].

Alongside the physical treatment above pointed out, GF flours from cereals/pseudocereals/pulses, hydrocolloids, proteins enzymes and emulsifiers have been utilized to emulate the cohesiveness and elasticity of the gluten-including pasta and noodles (Figure 7).

Naturally gluten free flours represent the first choice in GF products development plan. Rice and corn constitute the main ingredients, but lately flours from pseudocereals, such as quinoa, amaranth, buckwheat and sorghum and millet have been utilized with satisfactory results [80][81]. Successively also the impact of faba bean, lentil and chickpea flours in GF pasta making has been studied [82][83][84].

Appreciated for their water holding capacity, aging delay, textural improvement capability and gelling properties, hydrocolloids are polymers largely used in the GF food industry. In pasta making their interactions with water molecules and starch contribute to give viscosity to the GF matrix creating a continuous structure that allows texture improving, rehydration increasing and adhesiveness and cooking losses reduction [85]. The effects of utilizing carboxymethyl cellulose, xanthan gum, guar gum, locust bean gum have been amply studied [86][87][88].

Recent studies have reported a quality enhancing of GF pasta following to the addition of some emulsifiers such as mono and diacylglycerol, lecithin, and distilled glyceryl of monostearate. This is due to their amphiphilic nature fitting between two physical phases reducing surface tension and originating dispersion [89]. In extrusion process, emulsifiers behave as a lubricant providing firmer consistency and a less sticky pasta since they limit starch swelling and leaching phenomena during cooking [68].

Non-gluten protein can constitute another way forward to simulate the gluten performance in the dough. The introduction of egg albumen, rice bran, whey and protein concentrates was responsible for an improvement of cooking and firmness of GF pasta [90].

Enzymatic solutions have been adopted to explore the effects on GF pasta and noodles. Microbial transglutaminase action can visibly provide for a high-quality product with better sensory and color attributes and reduced the *in vitro* starch hydrolysis [91] [92].

In the last decade, non-conventional flours employing in GF pasta and noodles is representing an alternative approach for the nutritional and sensorial improvement of these products. In the study leaded by Padalino et al. [93] the outcome of yellow pepper flour addition (15%) for the GF spaghetti corn-based was evaluated. Results revealed that yellow pepper pasta contained higher fiber amount than the control while cooking loss and hardness were lower. Unripe plantain flour was blended (30%, 25%, 20% and 15%) with chickpea and maize flour to manufacture GF spaghetti. All the samples showed higher protein, fat, and ash content while carbohydrates amount was lower than the wheat control. Regarding cooking behavior, samples under study reported higher losses [58]. In quinoa and potato starch tagliatelle, egg protein was used as egg protein replacer [94]. It was observed that the higher the lupine flour content the higher was the firmness of the pasta.

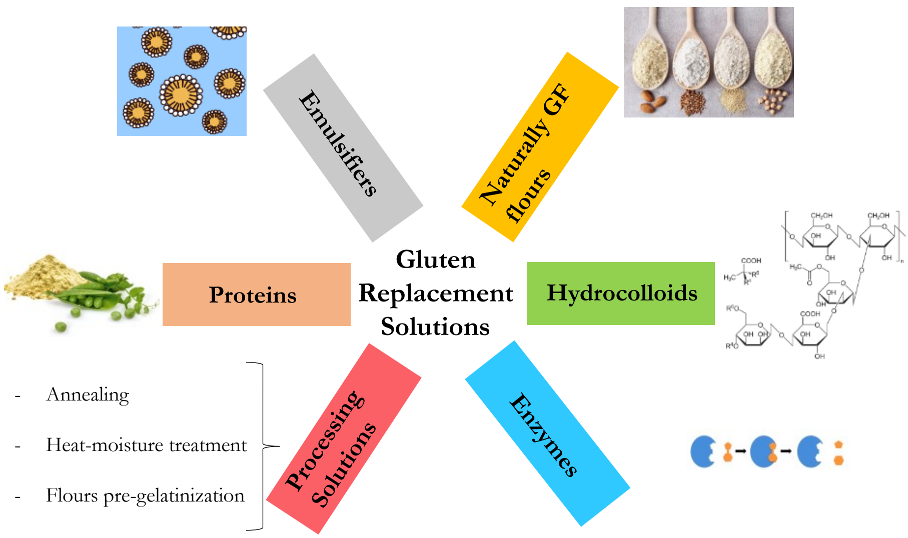

Figure 7: Principal gluten replacement solutions in pasta-like products

The state of the art indicates that despite the great potential of TN as food ingredient in GF products, only that has been exploited in the production of baked goods. Nevertheless, TN use in other type of foods has been scarcely explored. Recently, TN has been applied even in GF pasta products. Indeed, the combination of fenugreek, chickpea and TN flours allowed to obtain high fiber GF fresh pasta eggs-based [95]. Fenugreek was added into the basis formula (50% chickpea flour and 50% TN flour) at different levels (0, 2.5, 5, 7.5, 10%) replacing TN flour, improving pasta cooking performances.

No studies have been reported about the extrusion in GF pasta and noodles making. Nevertheless, some more authors have carried out research in the cooking-extrusion field for GF snacks preparation, to understand the effects deriving by TN flour integration. Increasing TN flour (up to 100%) blended with high quality cassava flour and a mixture of several spices resulted in an increased content of protein, ash, fat, and dietary fiber, although as the TN fraction rose, expansion ratio decreased [96]. Samples with more TN had higher levels of antinutrient (tannin, phytate, saponin, oxalate and alkaloids) that significantly ($P < 0.05$) decreased after extrusion process [97].

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2. Objectives

The main objective of this research was to extend the use of TN as food ingredient by designing GF foods based on pasta/noodle processing and extrusion technology.

To reach the main objective, the following specific objectives were defined:

1. Market evaluation of the GF pasta products identifying quality indicators related to technological performance and nutritional quality.

   This objective comprises the analysis of gluten free pasta acquired in the international market to investigate their performance during cooking and the real nutritional intake when consuming cooked GF pasta. Likewise, the heat damage induced during pasta making was assessed by quantifying the furosine (FUR) content in dried and cooked GF pasta.

2. Building gluten free TN based noodles applying knowledge on ingredients and processing, with particular emphasis on the role of hydrocolloids as structuring agents and pasta hydration.

   With that purpose, the role of a selection of hydrocolloids with varied chemical structure (GG, XG, inulin and two types of CMC with diverse viscosity) were evaluated on the quality characteristics of TN-noodles. Moreover, the role of hydration level was assessed by using two distinct hydrations: constant hydration usually applied in literature and adapted hydration depending on the food matrix requirements.

3. Understand the snacks markets and the state of the art in snacks development in order to define possible use of TN in designing new convenience foods.

   For that purpose, an updated search of the snacks' types, their productions and characteristics was carried out, identifying the market trends and the current scientific goals.

4. Explore the use of TN as ingredient of extruded foods, adapting recipes and process settings for overcoming the drawback of the TN fat content.

   To reach this goal GF extruded snacks containing TN were produced evaluating the effect of extrusion on the physical and microstructural qualities, the nutritional composition and antioxidant capacity.

This thesis has been structured in different chapters that correspond to specific scientific publications. The thesis compiles four peer-reviewed scientific publications and one book chapter. A schematic representation of the research performed in this thesis is presented in Figure 8.

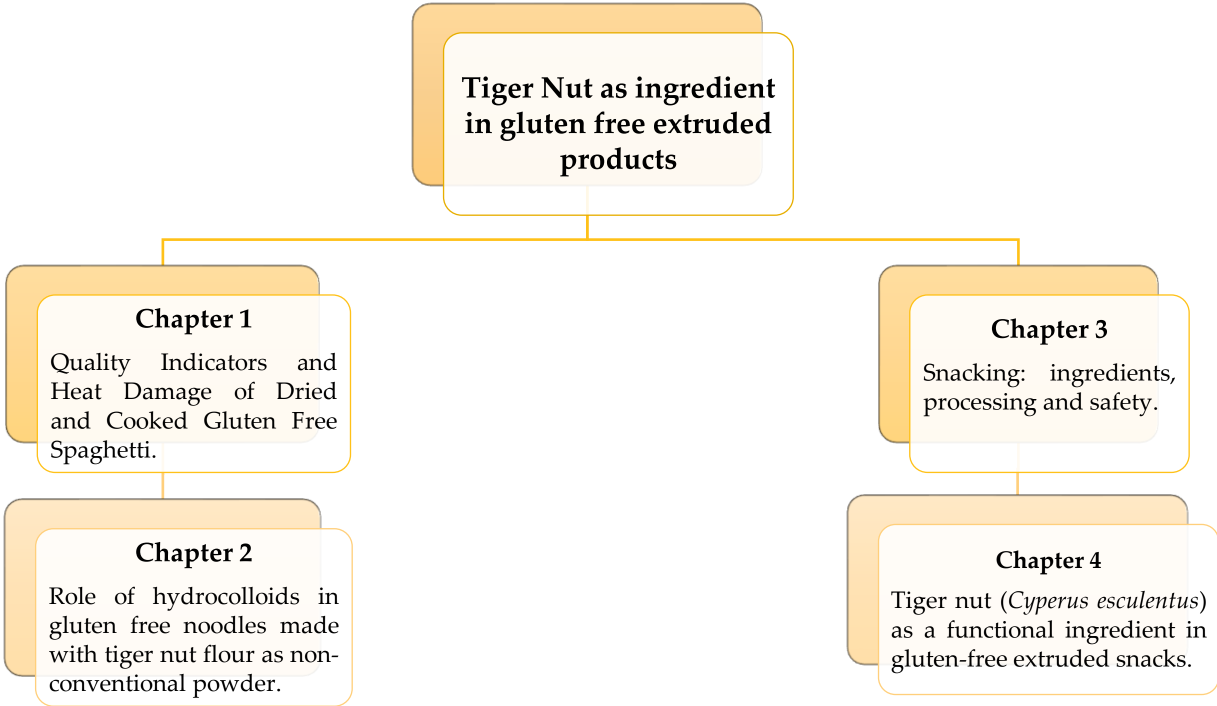

Figure 8: Schematic representation of the work carried out in this thesis

3. Results I

Quality Indicators and Heat Damage of Dried and Cooked Gluten Free Spaghetti

Nicola Gasparre, Ester Betoret and Cristina M. Rosell

Declaration: This chapter was written by author Nicola Gasparre that co-designed the study and performed the experimental work. Ester Betoret carried out the experimental work of analyzing furosines and wrote that part. Cristina M. Rosell designed and supervised the study and reviewed the manuscript.

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Quality Indicators and Heat Damage of Dried and Cooked Gluten Free Spaghetti

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Abstract

The quality and safety indicators of commercial dried gluten free (GF) pasta were analyzed to investigate, for the first time, the real nutritional intake through the chemical composition and the heat damage during processing by quantification of furosine. Eight samples of GF spaghetti were compared with wheat spaghetti. Dried and cooked GF pasta had lower protein and ash content than wheat spaghetti. GF samples composed solely by corn flour had higher optimal cooking time. Samples with emulsifier showed lower losses during cooking. Considering their composition, no trend could be established to explain textural behavior. Samples constituted merely by corn showed the highest resilience and elasticity. Spaghetti constituted only from corn and rice showed the highest firmness. The furosine content in dried samples ranged between 19 – 134 mg FUR/100 g proteins and in cooked samples ranged between 48 to 360 mg FUR/100 g proteins. Furosine content of GF pasta was in general lower than in wheat pasta, and those differences were even enlarged when comparing them after cooking. The results of PCA indicated it was possible to discriminate GF pasta regarding their technological and nutritional behavior.

Keywords: gluten free pasta; cooking quality; heat damage; furosine.

3.1 Introduction

Consumption of gluten free (GF) products has become trendy because of growing number of diagnosed celiac patients and the adoption of gluten free diets not related to pathologies. Among the non-gluten foods, pasta is one of the most consumed due to its convenience, palatability, and long shelf-life [1]. Although gluten pasta could be considered a simple processed food, it gets more complex when going to GF pasta. A good quality pasta is characterized by adequate performance during cooking, which depends on the raw materials, particularly the quantity and quality of proteins and starch that give rise to the formation of a gluten network that traps the starch granules [2]. In the case of GF pasta, to develop a pseudo gluten network, the industry uses different ingredients (flour/starch from corn, rice, tapioca, pseudo cereals and legumes) and may add emulsifiers, hydrocolloids or adopt nonconventional pasta-making processes [1]. Consequently, market offers a great range of GF pasta, which in opposition to what happens with gluten pasta, shows great variability. Nevertheless, the integration of the different ingredients might lead to complete different pseudo gluten network with rather diverse performance during cooking, and in turn, pasta quality characteristics. Similarly, scarce attention has been paid to their nutritional quality. Previous studies have pointed out the deficient nutritional pattern of GF breads [3,4], but information regarding the GF pasta is almost non-existent.

The diverse composition of GF pasta might respond different to process, particularly to pasta drying, which is a decisive stage for ensuring its microbiological stability by keeping final water content lower than 12.5 %. The High Temperature-Short Time system (> 75 to 100 °C for 2 to 3 h) usually applied for pasta drying allows producing gluten pasta with excellent cooking properties [5], but with a concomitant increasing risk of occurring Maillard reaction (MR) and the release of melanoidins as final products [6]. In early stages of MR, Amadori compounds (fructosyl–lysine, lactulosyl–lysine, and maltulosyl–lysine) are formed, which generate furosine (FUR) (ε-N-furoylmethyl-L-lysine) that has been established as a good marker of heat damage during pasta processing, as well as an indicator for the nutritional value and safety of the pasta products. Moreover, in the last years, some studies have stressed the toxic consequences of FUR on the biological system, specifically its adverse effects on mice liver and kidney [7].

The aim of this study was to analyze the quality indicators of commercial gluten free pasta (cooking behavior, color, texture properties), to investigate for the first time the real nutritional intake through the chemical composition of commercial cooked GF pasta and to evaluate the heat damage by quantification of FUR in dried and cooked GF pasta.

3.2 Materials and Methods

A total of 8 commercial GF spaghetti samples, manufactured by different European companies, were purchased in the market. Samples were labeled with different codes for identification, GF1, GF2, GF3, GF4, GF5, GF6, GF7, GF8, respectively. A wheat pasta was also included as a reference and labeled as W. Information contained in the package was recorded (Table 1). Chemical reagents and solvents of analytical grade were purchased to Sigma–Aldrich (Madrid, Spain). Standard of furosine was from Extrasynthese (Genay, France). Hydrochloric acid (37%) and methanol were purchased from Merck (Darmastadt, Germany). Ultra-pure water was obtained using a Mili-Q water purification system (Millipore, U.S.A.).

3.2.1 Cooking quality indicators

Optimal cooking time (OCT) was evaluated according to AACC official method [8] with some modifications. Spaghetti (5 g) were cooked in 60 mL of boiling water and every 30 s spaghettis were squeezed between two Petri dishes to determine the OCT that corresponded to the disappearance of the white core (ungelatinized starch). For each sample, two measurements were performed.

Cooking loss (CL), the organic material lost during cooking, was analyzed. For that, the cooking water was dried to constant weight in an air oven at 65 °C. The remnant (solid loss) was weighted and expressed as percentage following equation 1. The mean value was a result of three replicates.

$$\text{CL (\%)} = \frac{\text{Solid loss (g)}}{\text{Raw Pasta (g)}} \times 100 \qquad \text{[Equation 1]}$$

Water absorption (WA) was determined weighting five pieces of cooked spaghetti (5 cm long) and following the equation 2 as reported by Tudorică et al. [9]:

$$\text{WA (\%)} = \frac{\text{Cooked (g)} - \text{Raw Pasta (g)}}{\text{Raw Pasta (g)}} \times 100 \qquad \text{[Equation 2]}$$

Three measurements were performed for each analysis, and the mean values were calculated.

Swelling index (SI) was determined following the same steps of WA, five pieces of cooked spaghettis (5 cm long) were weighted and dried during 24 h to constant weight in air oven at 65°C. SI was determined following the equation 3:

$$SI\ (\text{g water/g dry pasta}) = \frac{\text{Cooked Pasta (g)} - \text{Cooked Pasta after drying (g)}}{\text{Cooked Pasta after drying (g)}} \quad \text{[Equation 3]}$$

Each sample was analyzed in triplicate and mean value was noted.

Color of dried and cooked spaghetti was measured by a Minolta Chromameter (Model CR-400, Minolta Co., Osaka, Japan) using CIELAB coordinates. *L** for the lightness (0 - 100), *a** for the green – red (negative values relate to green while the positive ones refer to red) and *b** for blue – yellow *b** (negative values relate to blue while the positive ones refer to yellow). From these parameters, the cylindrical coordinates hue or hue angle (h_{ab}) and Chroma (C^*_{ab}) were obtained following the equations 4 and 5 and the mean value came from five measurements.

$$C^*_{ab} = \sqrt{(a*)^2 + (b*)^2} \quad \text{[Equation 4]}$$

$$h_{ab} = \tan^{-1}\left(\frac{b*}{a*}\right) \quad \text{[Equation 5]}$$

3.2.2 Pasta characterization

Dried and cooked spaghetti samples were analyzed for moisture content, crude protein by Dumas Combustion Principle using 6.25 as conversion factor, total fat and ash following the ICC standard methods [10]. Total carbohydrates data were obtained by subtraction from 100 the moisture, protein, fat, and ash values. Results are the average of two determinations and are expressed as g/100 g.

Using the Texture Analyzer (TAHDi/500, TAHD Co., Stable Micro System, Surrey, UK) the texture profile analysis (TPA) was studied. The instrument was equipped with a load cell of 30 Kg and P/36 cylinder probe (36 mm Ø) and the following settings were used: pre-test speed, test speed and post-test speed was 2 mm/s, and trigger force was 10 g. The distance was 10 mm with 75% strain and holding time between compressions was 2 s. Five pieces (5 cm long) of cooked spaghetti were submitted to a double compression and Hardness, Adhesiveness, Chewiness and Resilience were measured using five replicates; data were calculated using the software of the instrument. Following the official method of the AACC International [8], firmness (g) (the maximum force necessary to cut spaghetti) and work of shear (g·s) (the area under the curve of force/time graphic) were investigated using a Texture Analyzer (TAHDi/500, TAHD Co., Stable Micro System, Surrey, UK) fitted with a plastic cutting tool. Each sample was analyzed in triple and data were calculated by software instrument. Elastic limit (g) and elasticity (mm) of spaghetti were determined by a Texture Analyzer equipped with a load cell of 30 Kg and with tensile grips (ref. A/SPR, Stable Micro Systems), following certain conditions: test speed was 3 mm/s and initial distance was 100 mm. Five measurements were performed for each sample and data calculated by the instrument software.

3.2.3 Furosine determination

Furosine was determined in dried and cooked spaghettis. Previously to furosine determination, cooked samples were lyophilized for 24 h at -70 °C and 933.26 Pa with a Virtis SP Scientific equipment (Pennsylvania, United States). Samples were hydrolyzed with 8 N HCl at 110°C for 24 h. One milliliter of the hydrolysate was filtered in a 0.22 μm cellulose filter. Chromatographic determination of FUR in pasta samples was performed on a HPLC instrument (HP Series 1050, USA) with a DAD detector (1040A) following the method of Li et al. [11]. The separation of FUR was accomplished on a C18 column (4.6 x 150 mm, 5 μm, Hewlett-Packard) and two mobile phases consisting of 0.1% trifluoroacetic acid (TFA) in water (A) and 0.1% TFA in acetonitrile (B). A gradient elution, 1-21% B at 0-25 min and 21-1% B at 25-30 min, was applied. A cleaning process with 1% tetrahydrofuran (THF) during 5 min was performed after each sample analysis. The flow rate was maintained at 1 mL/min. The identification of FUR was carried out by DAD spectra and the quantification was performed by an external standard method. The content of FUR was expressed as mg of compound in 100 mg of proteins. The analyses of each sample were carried out in duplicates.

3.2.4 Statistical analysis

Data were analyzed by One-Way analysis of variance (ANOVA) and multiple sample comparison, which was performed by using Statgraphics Centurion XVII (Statpoint Technologies, Warrenton, USA). Fisher's least significant differences (LSD) test was used to describe means with 95% confidence. Principal Component Analysis (PCA) was also performed to significantly differentiate (P <0.05) among samples.

3.3 Results and Discussion

3.3.1 Chemical composition

Most of the commercial samples of GF spaghetti analyzed in this study contained a mixture of corn and rice flours in their formulations; only two GF samples were composed only by corn, as shown in Table 1. Quinoa flour was present in two of the samples and other ingredients included brown rice and millet flour. Despite their coincidence in ingredients, there was significant difference in their nutritional composition (Table 2). Dried and cooked GF pasta had lower protein and ash content than wheat pasta, except for GF2, likely due to the presence of millet in the formulation as it has higher mineral content than corn or rice [12]. Conversely, dried GF pasta contained higher amount of fats than wheat pasta, excepting GF3, but those differences were greatly reduced in the cooked pasta. Differences among dried and cooked pasta confirms that cooking promoted significant loses of nutrients. When proximate composition (Table 2) of dried and cooked pasta was compared, it was observed that ash and carbohydrates were released to the cooking water, resulting in higher protein content in the cooked pasta. Cooking affected the fat content of the GF pasta in different way, increasing it in GF7 but decreasing in GF1, GF5 and GF6.

Table 1: Ingredients in commercial gluten free pasta as indicated in the label.

Code	Ingredients
GF1	White Corn Flour, Yellow Corn Flour, Rice Flour, Water, Emulsifier: E-471 Mono and Diglycerides of fatty acids
GF2	Corn Flour, Millet Flour, Rice Flour, Cane sugar syrup
GF3	Corn Flour, Rice Flour, Corn Starch, Quinoa Flour, Emulsifier: E-471 Mono and Diglycerides of fatty acids
GF4	Corn Flour
GF5	Corn Flour
GF6	Corn Flour, Rice Flour, Brown Rice Flour, Quinoa Flour, Emulsifier: E-471 Mono and Diglycerides of fatty acids
GF7	Corn Flour, Rice Flour
GF8	Corn Flour, Rice Flour, Emulsifier: E-471 Mono and Diglycerides of fatty acids
W	Durum Wheat Semolina

Table 2: Proximate composition (d.b.), furosine content (d.b.) and color of the pasta samples before and after cooking.

Sample	Moisture (%)	Ash (%)	Protein (%)	Fat (%)	Carbohydrates (%)	Furosine (mg/100 g of protein)	Hue angle	Chroma
Dried								
GF1	10.02 ± 0.13 b	0.31 ± 0.01 a	8.37 ± 0.04 e	1.17 ± 0.19 cd	80.14	24 ± 2 ab	-1.52 ± 0.00 c	57.25 ± 4.92 cd
GF2	9.89 ± 0.18 b	0.89 ± 0.00 f	9.90 ± 0.02 g	1.08 ± 0.22 c	78.23	19 ± 1 a	1.53 ± 0.00 e	55.61 ± 0.79 bc
GF3	11.52 ± 0.08 de	0.42 ± 0.00 c	7.71 ± 0.02 d	0.68 ± 0.00 a	79.67	134 ± 2 g	-1.56 ± 0.00 a	51.85 ± 0.43 a
GF4	11.26 ± 0.17 cde	0.37 ± 0.00 b	5.42 ± 0.03 b	1.42 ± 0.12 d	81.52	66 ± 3 d	1.57 ± 0.00 f	64.81 ± 1.52 g
GF5	11.88 ± 0.53 e	0.43 ± 0.00 c	5.29 ± 0.01 a	1.05 ± 0.02 bc	81.35	95 ± 6 e	1.51 ± 0.00 d	62.66 ± 2.28 fg
GF6	11.22 ± 0.81 cde	0.49 ± 0.00 d	8.86 ± 0.07 f	1.19 ± 0.01 cd	78.24	49 ± 5 c	-1.55 ± 0.00 b	60.44 ± 1.46 ef
GF7	10.48 ± 0.16 bc	0.63 ± 0.02 e	7.12 ± 0.19 c	0.95 ± 0.15 abc	80.82	30 ± 2 b	1.57 ± 0.00 f	64.80 ± 1.54 g
GF8	10.85 ± 0.14 cd	0.64 ± 0.01 e	7.00 ± 0.07 c	0.95 ± 0.04 abc	80.56	50 ± 2 c	-1.53 ± 0.00 c	59.85 ± 0.74 de
W	8.78 ± 0.20 a	0.90 ± 0.01 f	13.78 ± 0.03 h	0.76 ± 0.19 ab	75.78	121 ± 1 f	1.53 ± 0.01 e	53.62 ± 1.51 ab
Cooked								
GF1	65.62 ± 0.18 b	0.18 ± 0.03 a	8.92 ± 0.01 e	0.87 ± 0.02 ab	24.41	84 ± 0 bc	-1.43 ± 0.01 d	34.48 ± 3.60 c
GF2	66.32 ± 0.07 bc	0.61 ± 0.05 d	10.81 ± 0.06 g	1.12 ± 0.21 bcd	21.14	71 ± 7 b	-1.49 ± 0.00 b	30.32 ± 0.80 b
GF3	64.57 ± 0.18 a	0.31 ± 0.01 bc	8.24 ± 0.04 d	0.85 ± 0.19 ab	26.03	198 ± 7 e	-1.47 ± 0.02 bc	16.53 ± 2.18 a
GF4	66.81 ± 0.04 cd	0.17 ± 0.05 a	5.97 ± 0.01 b	1.17 ± 0.09 cd	25.88	131 ± 6 d	-1.48 ± 0.00 b	42.49 ± 1.21 d
GF5	69.78 ± 0.13 f	0.24 ± 0.10 ab	5.63 ± 0.03 a	0.82 ± 0.05 a	23.53	48 ± 1 a	-1.49 ± 0.01 b	40.52 ± 3.05 d
GF6	67.32 ± 0.78 d	0.31 ± 0.01 bc	9.32 ± 0.10 f	0.70 ± 0.03 a	22.36	79 ± 5 bc	-1.45 ± 0.03 cd	17.34 ± 2.36 a
GF7	67.38 ± 1.08 d	0.38 ± 0.02 c	7.44 ± 0.05 c	1.32 ± 0.12 d	23.48	77 ± 6 b	-1.43 ± 0.05 d	16.53 ± 1.28 a
GF8	68.17 ± 0.03 e	0.39 ± 0.01 c	7.56 ± 0.10 c	0.90 ± 0.14 ab	22.99	91 ± 10 c	-1.43 ± 0.03 d	29.85 ± 2.95 b
W	67.15 ± 0.06 d	0.60 ± 0.02 d	14.46 ± 0.01 h	0.92 ± 0.04 abc	16.87	360 ± 4 f	-1.52 ± 0.00 a	29.77 ± 0.48 b

Mean ± standard deviation Different letters within the same parameter for dried or cooked pasta differ significantly ($P< 0.05$)

Different cooking indicators (OCT, CL, WA, SI) of the cooking quality were evaluated (Table 3). GF samples had higher OCT than the wheat control, indicating that GF pasta required longer cooking to gelatinize all the starch granules, particularly those composed solely by corn flour (GF4 and GF5). Significant variability was observed among the amount of solid losses during cooking (CL). Considering that lower values are associated to better consistency and low stickiness [13], which in wheat pasta are related to the development of a stable and strong network of coagulated gluten proteins that entraps the starch granules [14], GF matrixes gave diverse network. In general, samples with emulsifier (GF1, GF3, GF6, GF8) showed lower CL, and samples only composed by corn (GF4 and GF5) had higher CL. Indeed, emulsifiers can interact with starch and/or protein [15] to form a good structure reducing the cooking losses. As described by Schoenlechner et al. [15], the use of emulsifiers (1.2 %) reduced cooking loss of gluten-free pasta made with quinoa, amaranth and buckwheat flour blends. Great variability was also observed in the water absorption (WA) that ranged from 49 % (GF2) to 107 % (GF7). In fact, there was a strong negative correlation between WA and CL (r = -0.73, $P < 0.001$). Regarding SI, or rather the capacity of protein-starch-fiber network to retain the water, the highest value was obtained with GF5 that only contains corn flour. Results agree with those reported by Foschia et al. [16]. Results related to the pasta color (Table 2) show that in the dried group, GF4 and GF7 had the highest values of Hue Angle and with GF5 they had the highest Chroma values. Among the cooked samples GF1, GF7 and GF8 were found to be the samples with Hue Angle highest values. Nevertheless, cooking affected the Chroma parameter, in fact all the cooked samples had lower values that the ones observed for the dried group. GF4 and GF5 had the highest Chroma parameter, related to yellowness, which could be expected owing to the solely presence of corn flour in their composition.

3.3.2 Texture properties

Texture properties of cooked pasta was evaluated (Table 3). Hardness, a required force to get a compression of spaghetti strands, is a good index of the pasta consistency. GF7 showed the highest consistency followed by GF6 and GF8. Hardness values were within the range obtained by Motta Romero et al. [17], when making pasta with proso millet flour and hydrocolloids (4480 – 8454 g). A negative weak correlation (r = -0.5125; P < 0.05) was found between hardness and the protein content in cooked sample, thus when the proteins amount increased, hardness decreased. Adhesiveness, related to the stickiness of pasta, also significantly varied within samples, showing GF8 the lowest value and GF4 was the stickiest owing to its highest adhesiveness. No trend could be established to explain that behavior considering the composition. In general, chewiness of GF pasta was higher than that of wheat pasta, thus high energy was required for swallowing GF spaghetti. Resilience of GF pasta was higher than that of wheat pasta, except for GF7 and GF8. GF4 and GF5, made with corn flour, showed the highest resilience or capacity to recuperate the initial state after compression force. GF5 also showed the highest firmness, which is related to good quality pasta. Firmness values were close to those reported for wheat-based spaghetti (174-183 g) [18] and much lower than those reported by Bouasla et al. [19] when gluten free pasta was enriched with different amount and kind of legumes flours (199.50 - 326.50 N). The work of shear showed the same tendency of firmness. Elastic limit, measured as the resistance (g) to extension was higher in GF1, GF4, GF5 and GF8 had the lowest value. A strong positive correlation ($r = 0.8366$; $P < 0.05$) was observed between elastic limit and resilience, namely samples with high capacity to recuperate the state before compression had more resistance to tensile force. Elasticity (mm) was higher in pasta made simply by corn flour (GF4 and GF5).

Table 3: Cooking quality indicators and texture parameters.

Sample	OCT (min)	CL (%)	WA (%)	SI (g water / g dry pasta)				
GF1	14.00 ± 0.71 **d**	3.22 ± 0.66 **a**	106 ± 3 **f**	1.68 ± 0.04 **ab**				
GF2	12.75 ± 0.35 **bc**	20.56 ± 1.22 **f**	49 ± 2 **a**	1.74 ± 0.05 **bc**				
GF3	13.25 ± 0.35 **cd**	8.21 ± 0.37 **d**	84 ± 8 **cd**	1.61 ± 0.04 **a**				
GF4	15.50 ± 0.71 **e**	29.82 ± 0.94 **g**	68 ± 6 **b**	1.81 ± 0.08 **cd**				
GF5	16.00 ± 0.71 **e**	19.95 ± 0.10 **f**	75 ± 4 **bc**	2.06 ± 0.08 **e**				
GF6	14.25 ± 0.35 **d**	4.83 ± 0.46 **b**	93 ± 6 **de**	1.83 ± 0.03 **cd**				
GF7	13.75 ± 0.35 **cd**	12.06 ± 0.20 **e**	107 ± 5 **f**	1.75 ± 0.04 **bc**				
GF8	11.75 ± 0.35 **ab**	6.98 ± 0.72 **c**	93 ± 7 **de**	1.88 ± 0.02 **d**				
W	11.25 ± 0.35 **a**	5.67 ± 0.20 **b**	94 ± 6 **e**	1.83 ± 0.10 **cd**				
	Hardness (g)	**Adhesiveness (g·s)**	**Chewiness (g)**	**Resilience**	**Firmness (g)**	**Work of Shear (g·s)**	**Elastic Limit (g)**	**Elasticity (mm)**
GF1	5408 ± 348 **a**	7.8 ± 0.7 **c**	3747 ± 553 **cd**	0.42 ± 0.01 **cd**	231 ± 4 **e**	28 ± 1 **cd**	42 ± 2 **f**	36 ± 5 **ab**
GF2	6560 ± 735 **bc**	7.9 ± 0.6 **c**	4317 ± 786 **de**	0.39 ± 0.02 **c**	166 ± 9 **b**	24 ± 1 **bc**	38 ± 2 **e**	36 ± 4 **ab**
GF3	6254 ± 655 **abc**	7.6 ± 0.8 **c**	3295 ± 512 **bc**	0.35 ± 0.02 **b**	155 ± 8 **b**	26 ± 5 **bcd**	31 ± 2 **c**	56 ± 6 **c**
GF4	6927 ± 844 **cd**	9.5 ± 0.7 **d**	5007 ± 434 **e**	0.44 ± 0.01 **d**	201 ± 10 **cd**	21 ± 3 **ab**	42 ± 1 **f**	73 ± 8 **e**
GF5	6331 ± 487 **abc**	7.6 ± 0.3 **c**	3843 ± 543 **cd**	0.43 ± 0.02 **d**	268 ± 6 **f**	28 ± 1 **d**	43 ± 2 **f**	64 ± 3 **d**
GF6	7795 ± 873 **de**	6.2 ± 0.4 **b**	3735 ± 558 **cd**	0.34 ± 0.02 **b**	161 ± 6 **b**	26 ± 1 **cd**	35 ± 2 **d**	34 ± 4 **a**
GF7	8366 ± 875 **e**	6.1 ± 1.0 **ab**	4936 ± 443 **e**	0.30 ± 0.03 **a**	110 ± 8 **a**	18 ± 3 **a**	25 ± 1 **b**	34 ± 3 **a**
GF8	7990 ± 533 **de**	5.1 ± 0.7 **a**	3224 ± 401 **ab**	0.30 ± 0.05 **a**	190 ± 9 **c**	22 ± 3 **ab**	20 ± 1 **a**	38 ± 3 **ab**
W	5615 ± 598 **ab**	7.8 ± 0.5 **c**	2121 ± 239 **a**	0.29 ± 0.02 **a**	205 ± 4 **d**	28 ± 0 **cd**	31 ± 3 **c**	41 ± 4 **b**

Mean ± standard deviation. Different letters within the same parameter differ significantly ($P < 0.05$). OCT: Optimal cooking time; CL: Cooking loss; WA: Water absorption; SI: Swelling index.

3.3.3 Furosine Content

Furosine were measured to assess possible heat damage during pasta drying. The statistical analysis showed significant differences ($P < 0.05$) in FUR content in both pasta samples: dried and cooked (Table 2), indicating high variability in drying conditions and raw materials in all 9 pasta samples. No information has been previously reported regarding the content of FUR in GF pasta. Values of FUR in dried pasta ranged between 19 – 134 mg FUR/100 g proteins, with the highest values of FUR obtained for samples GF3 and GF5 (134 and 95 mg FUR/100 g proteins, respectively). Much research has been dedicated to demonstrate the influence of drying cycles on FUR content in wheat pasta, ranging from 45 up to 209 mg/100 g of protein for pasta from artisanal processes (short temperatures long times), while in industrial ones (high temperatures short times) FUR reached values from 390 up to 562 mg/100 g of proteins [5, 20]. In addition, recipes can also impact the FUR content of wheat spaghetti. In fact, in those enriched with common bean, values of FUR ranged between 25 – 77 mg /100 g of proteins with higher values obtained in products with higher bean content (30 %) and higher drying temperatures (80 ºC) [17]. In lupine enriched spaghetti (0-20 %) FUR values ranged between 200 – 300 mg /100 g of protein after drying at 60 ºC for 18-20 h [21]. Cooking process significantly increased the FUR content ($P <$ 0.05) except in GF5, in which unexpectedly the FUR content significantly decreased. However, some authors have pointed out that Amadori compounds, which are formed in the early stages of the MR, are not stable and they are easily converted to intermediate and final products, depending on the progress of the MR [22]. In fact, GF5 had the highest cooking time.

According to Giannetti et al. [20] high intensity heat treatments can promote a dramatic progression of MR leading to FUR underestimation. The values obtained in cooked samples ranged between 48 - 360 mg FUR/100 g proteins with an increase in the content of FUR of around 38%. The highest FUR content was obtained for wheat pasta. Despite the toxicity of FUR, no data on the content of FUR in pasta after cooking are available in the literature. Only FUR content of fresh wheat egg pasta after double pasteurization (1: 95 ºC for 1.5 min; 2: 70 – 93 ºC for 60 - 90 min) were reported in the range 11.12 – 87.61 mg/ 100 g of proteins [23].

3.3.4 Principal Component Analysis

To understand the possible role of specific GF ingredients in the quality of the resulting pasta, all the parameters were subjected to a principal component analysis. The results of PCA indicated that 34% and 23% of the variation was explained by principal components 1 (PC1) and 2 (PC2), respectively (Figure 1). GF2 and GF3 were the samples closer to wheat durum pasta for protein and ash content, furosine and work of shear. GF1, GF4 and GF5, behaved similar regarding cooking performance (OCT, CL, SI), texture (adhesiveness, resilience, firmness, elastic limit, elasticity) and color (Hue angle of dried samples and Chroma in cooked stage). GF6, GF7 and GF8 had in common water absorption. Therefore, it was possible to discriminate GF pasta regarding their technological and nutritional behavior.

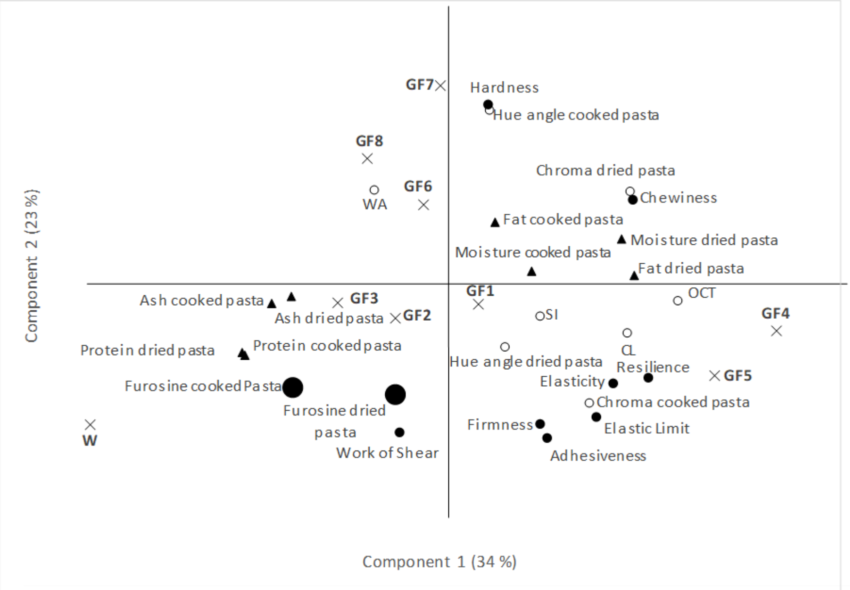

Figure 1: Score plot from a principal component analysis of the combination of components weight (chemical composition▲, cooking quality indicators ○, texture parameters• and furosine content●)

3.4 Conclusions

There were significant differences in the nutritional composition between the gluten free spaghetti and the durum wheat pasta. In general, gluten free samples in both stage (dried and cooked) were poorer in protein and ash with respect to the wheat pasta. Regarding quality indicators, GF samples showed significantly different behavior, and they could not be related to specific ingredients. Nevertheless, pasta made with corn flour required longer cooking and had high cooking loss values but resulted in more resilient and elastic pasta. The furosine content in dried GF pasta was majorly lower than that in wheat pasta, and those differences were greatly magnified after cooking. In the recent years, food technology applied to gluten free pasta production has taken great steps forward. Nevertheless, looking at the results, today nutritional improvements and new technologies approaches are needed to assure a nutritional balanced food.

Acknowledgements

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4. Results II

Role of hydrocolloids in gluten free noodles made with tiger nut flour as non-conventional powder

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Declaration: This chapter was written by author Nicola Gasparre that co-designed the study and performed the experimental work. Cristina M. Rosell designed and supervised the study and reviewed the manuscript.

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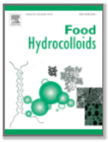


Role of hydrocolloids in gluten free noodles made with tiger nut flour as non-conventional powder

Nicola Gasparre, Cristina M. Rosell

Abstract

Gluten free noodles have been made from a variety of ingredients, but very often without applying ingredients and process knowledge. Aim of this study was to build up gluten free noodles using tiger nut flour, a selection of hydrocolloids (guar gum, xanthan gum, inulin and carboxymethyl cellulose) and considering the impact of dough hydration in noodles making. Dough rheology, fresh noodles characteristics, cooking quality indicators and noodles quality after cooking were evaluated. Results showed that hydration level significantly affected dough rheology during mixing, heating, and cooling, with a significant (*P*< 0.05) impact on hardness and firmness of fresh noodles and adhesiveness and firmness of cooked noodles. Hydrocolloids type significantly affected the characteristics of fresh and cooked noodles, but the extent of their effect was dependent on the hydration level applied during noodles making. In general, hydrocolloids increased dough consistency, resulting in fresh noodles with higher diameter, hardness and firmness, trend that was maintained after cooking, reducing cooking losses. In particular, gluten free tiger nut noodles made in the presence of 0.5% xanthan gum and adjusting the amount of water showed the best performance, with low cooking losses and high firmness.

Keywords: Gluten free, noodles, Tiger nut, Hydrocolloids, hydration.

4.1 Introduction

The design and development of gluten free (GF) foods is attracting very much attention, as a consequence of their increased consumption linked to whatever motivation (intolerances, lifestyle and so on). GF bread is the food that has prompted more extensive research trying to understand the matrixes, interactions among polymers and the impact of process conditions on the quality of the fresh bread [1] [2]. However, there are other widely consumed gluten free foods that are usually empirically designed by mixing gluten free ingredients without understanding their inner functionality.

Among the worldwide most consumed GF foodstuffs are noodles. They are a staple food owing to its variety, versatility, taste, and price [3], representing a viable alternative for gluten intolerant patients. Noodles can be produced from various grain flours such as wheat, rice, buckwheat, and corn, following three steps: raw material mixing (flour, water, salt), dough sheeting and cutting [3]. Nevertheless, in the GF field, noodles are generally produced through extrusion or sheeting of a rice batter [4]. The versatility of those technologies has stimulated the development of multitude of noodles, incorporating ingredients for nutritional enrichments, like pseudocereals and legumes [5] [6], or looking for healthy patterns [6]. Lately, extensive research has been reported adding alternative raw materials that provide functional properties [7], but their role within the process requires additional understanding. Tiger nut (*Cyperus esculentus* L.) (TN) is a specie of herbaceous plant that produces an edible sweet tuber and it is widely cultivated in Spain, Burkina Faso, Mali, Niger and Nigeria [8]. It has been used to produce a milky beverage (h*orchata*) and for feeding fish and farm animals [8]. From a nutritional point of view, TN is characterized by a good amount of fiber and omega-6 fatty acids [9][10]. These components could play a key role in the prevention of some diseases such as coronary heart disease, colon cancer, diabetes and obesity [11]. For this reason, TN is attracting great interest in human nutrition. In fact, many researches tried to exploit those properties developing food applications like TN oil [12], flours and biscuits [8][13] and fresh egg pasta [14]. In addition, the natural absence of gluten makes TN a potentially useful raw material for the manufacture of foods intended to consumers with specific nutritional needs. Despite its great potential, as mentioned above, the number of researches aimed to the development of TN based gluten free foods is very limited in literature, which include the use of up to 25% TN flour to improve the nutritional and functional quality of GF breads [15] or extruded snacks mixed with cassava flour [16]. Therefore, no previous study has been focused on developing GF noodles using TN as major ingredient. To make GF noodles, the lack of gluten must be counteracted with ingredients that overcome the loss of extensibility. Hydrocolloids represent a group of water-soluble polysaccharides with different chemical structure widely used in the food industry as gelling agents and thickeners, emulsifiers, stabilizers, foaming agents, improvers of water retention and texture [17]. Some studies have already emphasized the hydrocolloids role on GF noodles, mainly proposing the use of xanthan gum (XG), guar gum (GG) and carboxymethyl cellulose (CMC) to improve their texture and cooking quality [18].

The objective of this study was to build gluten free noodles applying knowledge on ingredients and processing. With that purpose, a gluten free flour or powder with described healthy properties (TN flour) was chosen, and a selection of hydrocolloids with varied chemical structure (GG, XG, inulin and two types of CMC with diverse viscosity) were evaluated. Moreover, the role of hydration level in noodles making was assessed by using two distinct hydrations: constant hydration usually applied in literature and adapted hydration depending on the food matrix requirements.

4.2 Materials and Methods

4.2.1 Materials

Milled TN was kindly granted by Món Orxata (Alboraia, Valencia, Spain). TN powder had 8.62% moisture, 48.29% carbohydrates, 26.27% total fat, 7.67% protein, 7.31% total fiber, 1.84% ash content. GG from Cargill (Spain), XG food grade from Jungbunzlauer (Ladenburg, Germany), inulin (Fibruline® XL) from Cosucra Groupe (Warcoing, Belgium), while CMC (Methocel A15 and Methocel A4M) were generously provided by Dow Pharma & Food Solutions (La Plaine Saint Denis, France).

4.2.2 Noodles preparation

Commercial TN powder was ground using a laboratory mill (IKA Eurostar M 20, Staufen, Germany) equipped with a water-cooling jacket, applying 3 cycles for 10 s, with a pause of 10 s between cycles. Particle size of the resulting powder was measured by laser diffraction (Mastersizer Scirocco 2000, Malvern Instruments Ltd., Worcestershire, UK), displaying d(0.50) and d(0.90) of 553,06 μm, and 1337.87 μm, respectively.

For noodles making, preliminary studies were run to determine the amount of water that allowed preparing a dough with TN powder with appropriate consistency to be extruded. In a control sample TN powder and distilled water (8:2, w:w) were mixed using a laboratory mixer (IKA Eurostar 40, Staufen, Germany) for 2 min at 100 rpm. Resulting dough was then extruded using a syringe (ø 3 mm) and noodles were poured directly into one liter of boiling water. The water was standardized following the official method (AACC official method 66-50.01). After ± 30 s, noodles were drained and cooled at ± 25°C for 5 min before drying at 40°C for 2 hours in a vacuum oven (NÜVE EV 018, Ankara, Turkey). Noodles containing hydrocolloids were prepared adding 0.5% (w/w in solid basis) of GG, XG, I, CMC A15 or CMC A4M, following the same protocol described above to obtain those samples referred as constant hydration level. Other set of samples, named adapted water hydration, was prepared having same consistency (similar Mixolab torque during the mixing-heating-cooling cycle) than the control. The amount of water needed to reach same torque during mixing was determined in preliminary assays carried out with the Mixolab®. Three batches were prepared for each type of noodles.

4.2.3 Rheological behavior

Dough (75 g) prepared as previously described was placed into the Mixolab® bowl (CHOPIN Technologies, France). Rheological behavior was recorded using the protocol Chopin+, with the following settings: mixing for 8 min at 30°C, heating from 30°C to 90°C at the rate of 4°C/min, hold at 90°C for 7 min, cooling to 50°C at 4°C/min and finally held at 50°C for 5 min [19]. The dough consistency obtained with the control sample was used as a target consistency and the amount of water required to reach that consistency with each recipe was determined. That water absorption was applied to obtain the set of noodles referred as adapted water hydration. Parameters recorded from the plots included: consistency during mixing (Nm), minimum torque during heating stage (Nm) and maximum torque during cooling stage (Nm). Two assays were performed for each sample, and mean values calculated.

4.2.4 Extrusion force

The force needed to extrude the dough (8 g) through the 12 mL syringe (ø 3 mm) was quantified using a Texture Analyzer (TAHDi/500, TAHD Co., Stable Micro System, Surrey, UK) equipped with a load cell of 30 kg and P/36 cylinder probe (36 mm Ø) and the following settings: pre-test speed and test speed was 1 mm/s, post-test speed was 10 mm/s, and trigger force was 10 g. The maximum peak of a compression test was identified as extrusion force. Three measurement were carried out for a single dough and mean value was recorded.

4.2.5 Cooking quality indicators

Optimal Cooking Time (OCT) was evaluated according to AACC official method [20] with some modifications. Noodles (5 g) were immersed in boiling water (60 mL), one piece of noodles (5 cm) was taken out every 30 s and squeezed between two Petri dishes to visually observe the time of disappearance of the white core (ungelatinized starch). The OCT was achieved when center core just disappears, thus complete starch gelatinization. Two measurements were performed for each sample and mean values recorded.

Cooking loss (CL) was evaluated according to AACC official method [20] with some modifications. After reaching OCT, noodles were drained in a Büchner funnel and rinsed with 50 mL of distilled water for 30 s. Cooking and rinse waters were combined and the total volume annotated. An aliquot of this water (1 mL) was dried during 24 h up to constant weight in air oven (J. P. Selecta 2000210, Barcelona, Spain) at 65°C. After 1 h in a desiccator, the residue (Solid loss) was weighted and calculated CL as percentage using the Equation 1. Three measurements were performed for each sample, and mean value was calculated.

$CL_{(g/100\ g)} = (\text{Solid loss}_{(g)} \times \text{Final cooking water}_{(mL)}) / (\text{Raw Noodles}_{(g)}) \times 100$ [Equation 1]

Drained noodles were allowed to cool down to room temperature for 10 min in a sealed Petri dish. Then, five pieces of noodles (5 cm long) were weighed and water absorption (WA) expressed in percentage was determined following equation 2:

$WA_{(g/100\ g)} = (\text{Cooked Noodles}_{(g)} - \text{Raw Noodles}_{(g)}) / (\text{Raw Noodles}_{(g)}) \times 100$

[Equation 2]

Three measurements were performed for each analysis, and mean values calculated.

Five pieces of drained noodles (5 cm long) were weighed and dried for 24 h to constant weight in an air oven (J. P. Selecta 2000210, Barcelona, Spain) at 65°C. Swelling index (SI) expressed as grams of water per gram of dry noodles was determined following the procedure described by Cleary and Brennan [21] and calculated as indicated in equation 3:

$$SI_{(g\ water/g\ dry\ noodles)} = (Cooked\ Noodles_{(g)} - Cooked\ Noodles\ after\ drying_{(g)}) / (Cooked\ Noodles\ after\ drying_{(g)})$$

[Equation 3]

Three measurements were performed for each sample, and the mean values were calculated.

Diameter of uncooked and cooked GF noodles was measured with a digital caliper. Five measurements were performed for each sample and mean value reported.

Color of uncooked and cooked GF noodles was measured using a Minolta Chromameter (Model CR-400, Minolta Co., Osaka, Japan) following CIELAB scale: *L** (lightness 0-100), *a** (positive values measure redness, negative values measure greenness) and *b** (positive values measure yellowness, negative values measure blueness). Each data represents the mean of five measurements.

Texture profile analysis (TPA) was carried out in a Texture Analyzer (TAHDi/500, TAHD Co., Stable Micro System, Surrey, UK) equipped with a load cell of 30 kg and P/36 cylinder probe (36 mm Ø) and with the following settings: pre-test, test and post-test speed was 2 mm/s and the trigger force was 10 g. Distance was set up at 30 mm with 75% strain and 2 s the holding time between compressions. Five strands (5 cm long) of fresh and cooked noodles were submitted to a double compression test and Hardness (g), Adhesiveness (g·s), Chewiness and Resilience were measured with the software instrument. Five replicates were evaluated for each experimental value reported.

Firmness, expressed as the maximum force (g) necessary to shear a strand of noodles, and work of shear (g·s) corresponding to the area under the curve of force/time graphic, were investigated using a Texture Analyzer (TAHDi/500, TAHD Co., Stable Micro System, Surrey, UK) as described by the official method 66-50.01 (AACC 2001). Samples were analyzed in triple and Data were calculated with the software instrument and the average of three replicates used.

4.2.6 Statistical Analysis

Experimental data were expressed as a mean ± standard deviation and statistically analyzed by multifactor analysis of variance (MANOVA) using Statgraphics V.7.1 (Bitstream, Cambridge, MN). Fisher's least significant differences test was used for assessment of significant differences among experimental mean values with 95% confidence.

4.3 Results and discussion

4.3.1 Rheological behavior

Mixolab® has been traditionally applied to record bread doughs performance during mixing when subjected to a heating-cooling cycle. In the present study, Mixolab® was used to record the rheology of a GF noddle dough made from TN powder. Therefore, plots (Figure 1) displayed completely different patterns than the ones observed either with gluten matrixes or non-gluten flours. Fresh doughs were directly placed into the bowl, recording initial consistency that showed a slight decay as mixing proceeds, indicating some loss of stability. A pronounced dough softening was observed during heating stage and no increase in consistency, usually attributed to starch gelatinization, was observed owing to the low amount of starch or limited amount of water. Dough consistency only exhibited a steady increase during the cooling stage.

In GF noodles, hydrocolloids are needed to provide extensibility. But, considering their diverse ability to bind water, it was assumed that water would have a crucial role in defining noodles dough consistency and the quality of fresh noodles. Nevertheless, usually constant amount of water is applied when producing rice noodles, and slightly varied when changing the flour. Considering that a non-cereal flour was used and the diverse characteristics of the hydrocolloids, dough rheology was tested applying different hydrations till reaching the same consistency in all the blends for obtaining noodles (Figure 1). Two types of CMC were selected due to their great variation on viscosity (CMC A15 15 mPa·s and CMC A4M 4000 mPa·s). Firstly, doughs were prepared using the same amount of water, which were referred as constant hydration (Figure 1 A), and secondly, doughs were prepared adjusting the level of water to hydrate all ingredients, leading to doughs with the same consistency (same torque during mixing with a Mixolab) than the control sample, those were referred as constant consistency (Figure 1 B). The incorporation of hydrocolloids to doughs resulted in doughs with higher consistency when the level of water was kept constant (Figure 1 A), due to their water retention ability. Those differences were minimized when the water level was adapted, allowing hydrocolloids hydration, being GG followed by XG the hydrocolloids that required higher amount of water to adjust consistency (Figure 1 B). Parameters used to quantify rheological behavior are showed in Table 1, where type of dough hydration and presence of hydrocolloids were identified as main factors of variance. Statistical analysis confirmed the significant ($P < 0.05$) effect of hydration on the dough rheology during mixing, heating and cooling stages, but no significant effect was observed due to the type of hydrocolloid because differences were eliminated when constant consistency was applied. Nevertheless, the analysis of variance within the samples group with constant hydration showed a significant ($P < 0.05$) effect of hydrocolloid on the consistency (Nm) during the Mixolab® stages. When constant hydration (20 g water/100 g powder) was used for making doughs, the addition of hydrocolloids, except for inulin, significantly increased dough consistency during mixing and cooling stages with respect to control (in the absence of hydrocolloid). XG and GG were the hydrocolloids that mostly increased consistency during mixing and cooling stages, indicating their higher absorption of water. The enhancement of consistency during cooling promoted by all hydrocolloids, with exception of inulin, suggested higher interaction between hydrocolloids and leached amylose, which might indicate that gums were preferentially placed on the surrounding solution instead of being located at the external side of the granules [22].

Figure 1: Mixolab® curves representing the rheological behavior of the noodle's doughs. A. Doughs obtained with the same hydration; B. Doughs obtained with adapted hydration level to keep constant consistency. Solid black line displays the temperature gradient applied during the rheology assessment.

Figure 1 A

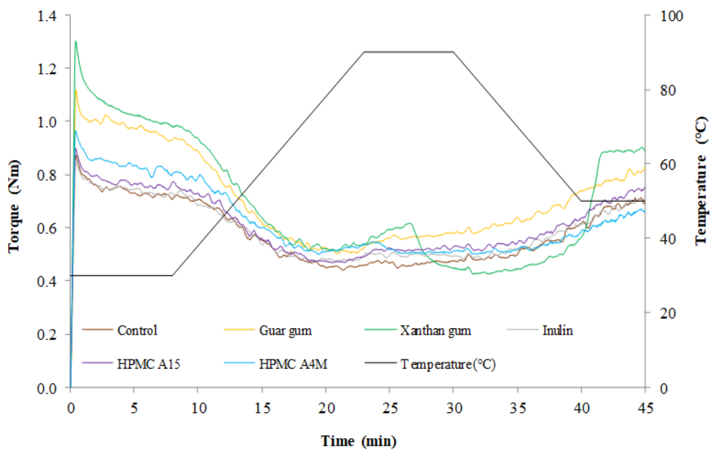
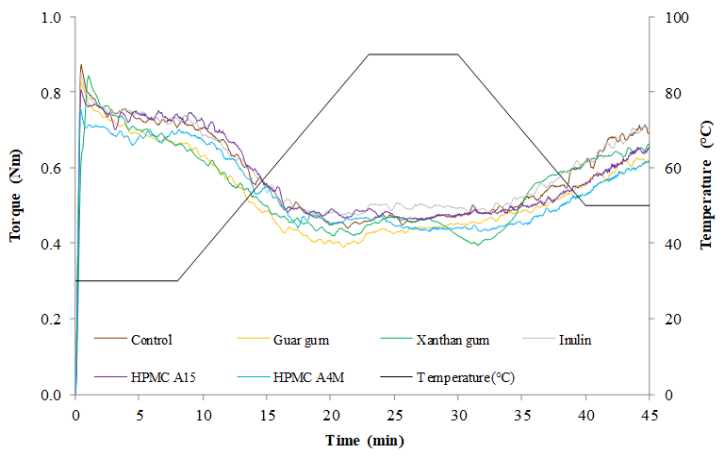


Figure 1 B



To obtain a constant consistency, doughs required higher amount of water with the subsequent reduction in the torque (Nm), apart from inulin that did not affect consistency (Table 1). After adjusting water levels, no significant differences were observed due to hydrocolloids along mixing, heating, and cooling stages.

Extrusion force required to obtain noodles were also evaluated, and significant differences were observed due to the hydration level and the type of hydrocolloids (Table 1). Differences within hydrocolloids type were only significant when constant hydration was applied to obtain doughs, confirming that doughs containing XG or GG required higher extrusion force. Same findings were observed when GG was added (0 - 10%) to various flours (corn, potato, rice, and wheat) to produce extruded snacks, observing an increase of extrusion torque (Nm) with all the flours [23].

Table 1: Thermomechanical behavior of Tiger nut doughs with constant hydration and constant consistency

Hydration	Hydration level (g/100 g)	Hydrocolloid	Mixing Consistency (Nm)				Heating Consistency (Nm)				Cooling Consistency (Nm)				Extrusion Force (g)			
Constant	20.00	None	0.71	±	0.01	**ab**	0.44	±	0.03	**abc**	0.70	±	0.02	**de**	5371	±	175	**a**
	20.00	Guar gum	0.96	±	0.01	**e**	0.50	±	0.01	**c**	0.83	±	0.00	**g**	6453	±	135	**c**
	20.00	Xanthan gum	1.01	±	0.03	**f**	0.43	±	0.01	**ab**	0.89	±	0.02	**h**	6516	±	23	**c**
	20.00	Inulin	0.73	±	0.01	**b**	0.47	±	0.01	**bc**	0.71	±	0.01	**e**	5246	±	53	**a**
	20.00	CMC A15	0.77	±	0.01	**c**	0.46	±	0.01	**abc**	0.76	±	0.01	**f**	5967	±	236	**b**
	20.00	CMC A4M	0.82	±	0.03	**d**	0.50	±	0.01	**c**	0.66	±	0.01	**c**	6085	±	178	**b**
Adapted	20.00	None	0.71	±	0.01	**ab**	0.44	±	0.03	**abc**	0.70	±	0.02	**de**	5371	±	175	**a**
	26.00	Guar gum	0.70	±	0.01	**ab**	0.39	±	0.01	**a**	0.63	±	0.01	**ab**	5264	±	161	**a**
	24.00	Xanthan gum	0.70	±	0.01	**ab**	0.39	±	0.05	**a**	0.67	±	0.02	**cd**	5281	±	70	**a**
	20.00	Inulin	0.73	±	0.01	**b**	0.47	±	0.01	**bc**	0.71	±	0.01	**e**	5246	±	53	**a**
	21.06	CMC A15	0.73	±	0.01	**abc**	0.44	±	0.01	**abc**	0.66	±	0.00	**bcd**	5385	±	247	**a**
	21.30	CMC A4M	0.69	±	0.01	**a**	0.42	±	0.05	**ab**	0.62	±	0.01	**a**	5125	±	58	**a**
	***P*-value**	Hydration	0.00				0.00				0.00				0.00			
		Hydrocolloid	0.10				0.16				0.08				0.01			

*means with different letters within the same parameter differ significantly ($P < 0.05$)

4.3.2 Quality indicators of fresh noodles

It was possible to make noodles with TN powder, despite the absence of gluten and the particular characteristics of this small tuber. Characterization of GF TN noodles was carried out at the different stages of production, including fresh noodles and their quality after cooking. Regarding fresh noodles, the statistical analysis revealed that hydration level significantly affected the luminosity of the noodles (L^*), hardness, firmness and the work of shear; conversely hydrocolloids type significantly affected all the parameters evaluated with the exception of work of shear (Table 2). Therefore, on fresh noodles, the type of hydrocolloid was crucial for producing GF noodles. Going into detailed effects, it was observed that GG, XG and CMC A4M increased the noodles diameter, independently of the amount of water used for producing them. That increase confirmed the high ability of those hydrocolloids to retain water. Color was differently affected by hydrocolloids, observing a decrease of luminosity, although only significant when hydration was adapted and in the presence of XG, GG or CMC A15. Additionally, a^* and b^* were significantly ($P < 0.05$) influenced by hydrocolloids, particularly, XG had the higher values in terms of redness, whereas GG led to lower redness and yellowness. The parameters a^* and b^* were very close to those obtained with fresh durum pasta containing TN [24].

Hardness and firmness were significantly affected by hydration level and hydrocolloids ($P<0.05$) (Table 2). In general, hardness, the force necessary to attain a given deformation [25], was higher in the noodles with constant hydration, after adjusting the water softer noodles were attained. When hydrocolloids were added, hardness increased especially when CMC A15 was used, independently of the hydration, and GG, but only when constant hydration was used. Thus, hydrocolloids improved the stiffness of GF noodles structure. Similar result was obtained by Padalino et al. [26] that reported an increase of hardness in GF spaghetti made by corn and oat when they were separately added (2 % of GG and CMC). Firmness, the force required to cut the noodle, was higher in noodles with adapted hydration, except for those containing inulin. XG was the hydrocolloid that conferred higher firmness. Adhesiveness, the work necessary to overcome the attractive forces between the surface of the noodle and the surface of teeth [25], was significantly reduced in the presence of hydrocolloids, excepting XG when it was added without adjusting the hydration level, which led to more adhesive noodles. Likewise, Chauhan et al. [27] described the decrease of adhesiveness when 0.5% or 1% of hydrocolloids (GG, acacia gum and tragacanth gum) were added to amaranth GF pasta. Chewiness or the energy needed to make noodles in a state suitable for swallowing [25] were significantly increased by all the hydrocolloids tested, but the extent of the impact was dependent on the hydration level applied to noodles. In fact, CMC A15 resulted in the highest chewiness when using constant hydration, despite its lower initial viscosity, but GG led to the highest value when adapted hydration was applied. Hydrocolloids also significantly increased the noodles resilience, except for XG when constant hydration was used. Analyzing the work required to shear, it significantly ($P < 0.05$) depended on the hydration level, samples group with constant hydration was characterized by lower results. GG, CMC A15 and CMC A4M after adjusting hydration led to noodles with the highest value in work of shear. Therefore, level of hydration significantly affected fresh noodles characteristics

Table 2: Diameter, color, and textural properties of fresh Tiger nut gluten free noodles

Hydration	Hydrocolloid	Diameter (mm)	Color			Firmness	
			L^*	a^*	b^*	Firmness (g)	Work of Shear (g·cm)
Constant	None	3.43 ± 0.06 a	44.40 ± 1.24 cd	5.70 ± 0.34 b	21.24 ± 0.31 de	32 ± 2 a	5.5 ± 0.4 ab
	Guar gum	3.57 ± 0.06 c	43.55 ± 1.70 bcd	4.09 ± 0.57 a	18.45 ± 0.16 a	35 ± 1 abc	5.0 ± 0.9 a
	Xanthan gum	3.57 ± 0.06 c	43.02 ± 1.27 bc	6.36 ± 0.51 c	21.65 ± 0.68 e	38 ± 1 abcd	5.6 ± 0.5 abc
	Inulin	3.47 ± 0.06 ab	43.20 ± 1.79 bc	5.59 ± 0.74 b	19.33 ± 0.49 b	33 ± 7 ab	5.8 ± 0.5 abc
	CMC A15	3.47 ± 0.06 ab	42.92 ± 1.03 abc	5.87 ± 0.41 bc	20.18 ± 0.22 c	36 ± 5 abcd	5.1 ± 1.3 a
	CMC A4M	3.53 ± 0.06 bc	45.19 ± 0.80 d	5.46 ± 0.40 b	20.79 ± 0.46 d	38 ± 2 abcd	6.8 ± 0.4 cd
Adapted	None	3.43 ± 0.06 a	44.40 ± 1.24 cd	5.70 ± 0.34 b	21.24 ± 0.31 de	32 ± 2 a	5.5 ± 0.4 ab
	Guar gum	3.53 ± 0.06 bc	41.20 ± 0.40 a	5.72 ± 0.18 b	20.87 ± 0.31 d	40 ± 2 cde	6.9 ± 0.5 d
	Xanthan gum	3.57 ± 0.06 c	42.44 ± 1.52 ab	5.70 ± 0.44 b	21.50 ± 0.30 e	44 ± 1 e	6.3 ± 1.0 bcd
	Inulin	3.47 ± 0.06 ab	43.20 ± 1.79 bc	5.59 ± 0.74 b	19.33 ± 0.49 b	33 ± 7 ab	5.8 ± 0.5 abc
	CMC A15	3.47 ± 0.06 ab	41.93 ± 1.41 ab	5.52 ± 0.37 b	20.11 ± 0.58 c	41 ± 3 de	7.5 ± 0.6 d
	CMC A4M	3.53 ± 0.06 bc	43.52 ± 1.92 bcd	5.52 ± 0.37 b	19.87 ± 0.29 c	38 ± 4 bcde	6.9 ± 1.0 d
P-value	Hydration	0.73	0.02	0.48	0.23	0.01	0.00
	Hydrocolloid	0.00	0.00	0.01	0.00	0.00	0.09

Hydration	Hydrocolloid	Texture profile analysis (TPA)			
		Hardness (g)	Adhesiveness (g·s)	Chewiness (g)	Resilience
Constant	None	8758 ± 534 a	93 ± 20 c	798 ± 144 a	0.19 ± 0.01 a
	Guar gum	12765 ± 658 de	39 ± 4 a	2086 ± 397 cde	0.25 ± 0.01 e
	Xanthan gum	10711 ± 559 b	114 ± 28 d	1433 ± 271 abc	0.19 ± 0.01 a
	Inulin	11920 ± 493 c	35 ± 5 a	2020 ± 563 cde	0.23 ± 0.01 cd
	CMC A15	13377 ± 652 e	22 ± 2 a	2495 ± 531 e	0.27 ± 0.02 f
	CMC A4M	10447 ± 678 b	83 ± 8 bc	1918 ± 666 cde	0.22 ± 0.01 bc
Adapted	None	8758 ± 534 a	93 ± 20 c	798 ± 144 a	0.19 ± 0.01 a
	Guar gum	10986 ± 767 b	79 ± 12 bc	2397 ± 663 cde	0.22 ± 0.02 bcd
	Xanthan gum	10349 ± 795 b	70 ± 7 b	1285 ± 244 ab	0.21 ± 0.01 b
	Inulin	11920 ± 493 c	35 ± 5 a	2020 ± 563 cde	0.23 ± 0.01 cd
	CMC A15	12190 ± 621 cd	26 ± 4 a	1837 ± 208 bcd	0.24 ± 0.02 de
	CMC A4M	10617 ± 662 b	38 ± 4 a	2027 ± 366 cde	0.24 ± 0.01 de
P-value	Hydration	0.01	0.19	0.61	0.41
	Hydrocolloid	0.00	0.00	0.00	0.00

*means with different letters within the same parameter differ significantly ($P < 0.05$)

4.3.3 Quality indicators of cooked noodles

Generally, good quality noodles shall be characterized by low cooking loss, high firmness, and absence of stickiness [28]. In this study other quality indicators related to cooking have been evaluated (Table 3). The level of hydration significantly ($P < 0.05$) affected the color of the cooked noodles, and hydrocolloids affected the OCT, CL and water absorption, as well. Hydrocolloids incorporation increased significantly ($P < 0.05$) the OCT, which might be expected considering water limitation, and in consequence the delay in the starch gelatinization. Same trend was described by Kaur et al. [29] when GG and XG were added (0.25% or 0.35%) to produce potato, corn and mung bean starch noodles, which has been recently explained by the protective behavior of the hydrocolloids towards starch granules that cause the increase in the gelatinization temperature onset [30]. On the other hand, hydrocolloids addition decreased ($P < 0.05$) the cooking loss, and those losses tended to be lower when adapted hydration was applied during the making process. The hydrocolloids improve the noodles' structure, preventing the release of solid matters into cooking water. The addition of XG allowed obtaining GF TN noodles that kept better their structure, and that effect was even greater when prepared adapting the hydration. Same action of gums on CL has been reported for GG, CMC and XG when making organic red jasmine rice noodles [31] and dried-naturally fermented rice noodles [18]. Therefore, present research shows that hydrocolloids are also able to held a tuber powder for producing noodles.

The great ability of hydrocolloids to bind water was also sustained by the WA results, which were significantly (P < 0.05) higher, whatever hydration was applied (Table 3), except for the GG when the adapted hydration was applied. Even after adjusting the hydration, the presence of hydrocolloids promoted higher water absorption. Other authors described same results obtained with rice noodles [18]. The effect of hydrocolloids was evident also in the SI, indicating that hydrocolloids significantly increased the amount of bonded water to starch and proteins, leading to lower released of soluble compounds, except in the case of inulin. As mentioned above, hydration and hydrocolloids had a significant (P < 0.05) effect on the color parameter L^*, a^* and b^* of cooked noodles. TN tubers have a brownish color, that is maintained in TN noodles. In general, noodles made with constant hydration had higher L^*, a^* and b^*. Noodles containing inulin were brighter, with reddish and brownish tone.

Hydration type significantly (P < 0.05) affected the adhesiveness, resilience and firmness, and hydrocolloids type significantly affected all the texture parameters evaluated (hardness, adhesiveness, chewiness, resilience, firmness, work of shear) (Table 3). Regarding hardness, the most pronounced effect was the softening effect of GG and CMC A15, but only when constant hydration was applied; and the same trend was observed in the noodles' resilience. Hardness of cooked samples (Table 3) are in line with the values obtained for other noodles prepared with different flours, like tartary buckwheat noodles that showed hardness from 5960 g to 7780 g [32]. In contrast with what was observed in fresh samples, in cooked noodles the presence of hydrocolloids increased the adhesiveness, although it was not significant in the case of inulin and CMC A15, and the former had significant effect when adjusting hydration. Consequently, gelling and starch gelatinization modified the impact of hydrocolloids on adhesiveness. Although hydrocolloids significantly affected chewiness, the effect was dependent on the type of hydrocolloids, being the impact greater when constant hydration was applied. It must be stressed the effect induced by GG, that reduced chewiness when noodles were prepared with constant hydration but when adjusting it, a significant increase in chewiness was obtained. Similarly, opposite effects were observed when adding XG depending on the hydration type applied during noodle making. XG induced a significant increase in chewiness when constant hydration, but no significant difference with the control was observed when using adapted hydration. As it was previously described with fresh noodles, firmness during shearing was significantly affected by hydration and hydrocolloid type, but the work required to cut noodles was significantly dependent on the type of hydrocolloids. Xanthan gum was the hydrocolloid that mostly affected the performance of noodles during shearing, especially when adapted hydration was used.

Table 3: Quality indicators, color, and textural properties of cooked Tiger nut gluten free noodles

Hydration	Hydrocolloid	Cooking quality indicators: OCT (min)	CL (g/100 g)	WA (g/100 g)	SI (g/g)	Color: *L**	*a**	*b**
Constant	None	4.75 ± 0.35 a	18.38 ± 0.32 f	82.12 ± 2.82 a	1.43 ± 0.08 bc	47.70 ± 1.71 cde	3.56 ± 0.44 bc	14.37 ± 0.42 a
	Guar gum	5.75 ± 0.35 b	17.96 ± 0.82 ef	110.73 ± 3.43 f	1.28 ± 0.15 a	46.31 ± 1.47 abc	4.08 ± 0.45 d	15.40 ± 0.35 b
	Xanthan gum	6.00 ± 0.00 b	13.68 ± 0.43 b	94.47 ± 2.77 cd	1.22 ± 0.06 a	48.58 ± 1.43 de	4.03 ± 0.42 d	15.58 ± 0.28 b
	Inulin	5.75 ± 0.35 b	16.52 ± 0.29 d	105.50 ± 7.15 ef	1.50 ± 0.06 c	50.25 ± 1.09 f	4.99 ± 0.10 e	17.59 ± 0.36 d
	CMC A15	5.50 ± 0.00 b	15.38 ± 0.69 c	103.45 ± 1.72 ef	1.22 ± 0.09 a	49.24 ± 1.25 ef	3.95 ± 0.44 cd	16.73 ± 0.36 c
	CMC A4M	5.50 ± 0.00 b	14.65 ± 0.95 c	93.68 ± 5.39 bc	1.22 ± 0.09 a	47.60 ± 0.52 bcd	3.91 ± 0.42 cd	17.07 ± 0.62 c
Adapted	None	4.75 ± 0.35 a	18.38 ± 0.32 f	82.12 ± 2.82 a	1.43 ± 0.08 bc	47.70 ± 1.71 cde	3.56 ± 0.44 bc	14.37 ± 0.42 a
	Guar gum	5.75 ± 0.35 b	13.06 ± 0.40 b	87.56 ± 6.75 ab	1.24 ± 0.06 a	46.05 ± 0.88 ab	3.57 ± 0.27 bc	15.36 ± 0.39 b
	Xanthan gum	6.00 ± 0.00 b	12.04 ± 0.70 a	101.37 ± 2.90 de	1.34 ± 0.08 ab	46.44 ± 0.92 abc	3.25 ± 0.30 ab	14.58 ± 0.18 a
	Inulin	5.75 ± 0.35 b	16.52 ± 0.29 d	105.50 ± 7.15 ef	1.50 ± 0.06 c	50.25 ± 1.09 f	4.99 ± 0.10 e	17.59 ± 0.36 d
	CMC A15	5.50 ± 0.00 b	17.43 ± 0.41 de	92.04 ± 2.21 bc	1.29 ± 0.02 a	45.32 ± 1.29 a	3.04 ± 0.23 a	14.40 ± 0.40 a
	CMC A4M	5.50 ± 0.00 b	14.82 ± 0.57 c	95.81 ± 0.93 cd	1.33 ± 0.04 ab	46.80 ± 0.96 abc	3.43 ± 0.38 ab	15.11 ± 0.15 b
P-value	Hydration	1.00	0.12	0.07	0.08	0.00	0.00	0.00
	Hydrocolloid	0.00	0.00	0.00	0.04	0.00	0.00	0.00

Hydration	Hydrocolloid	Texture profile analysis (TPA): Hardness (g)	Adhesiveness (g·s)	Chewiness (g)	Resilience	Firmness: Firmness (g)	Work of Shear (g·cm)
Constant	None	6556 ± 543 cde	96 ± 6 a	905 ± 80 c	0.15 ± 0.01 b	17.7 ± 1.4 a	2.1 ± 0.4 a
	Guar gum	5375 ± 907 ab	128 ± 2 d	600 ± 187 ab	0.12 ± 0.01 a	24.0 ± 0.5 bc	3.1 ± 0.2 ab
	Xanthan gum	7205 ± 1129 e	120 ± 9 cd	2132 ± 526 e	0.14 ± 0.01 b	30.3 ± 0.6 e	4.6 ± 0.3 c
	Inulin	6744 ± 537 cde	103 ± 12 abc	832 ± 24 bc	0.14 ± 0.01 b	22.1 ± 2.5 b	2.4 ± 0.3 a
	CMC A15	5289 ± 936 a	99 ± 13 ab	507 ± 88 ab	0.12 ± 0.02 a	25.5 ± 1.3 c	2.5 ± 0.4 a
	CMC A4M	7243 ± 638 e	122 ± 9 d	981 ± 134 cd	0.15 ± 0.01 b	26.1 ± 0.5 cd	2.3 ± 0.3 a
Adapted	None	6556 ± 543 cde	96 ± 6 a	905 ± 80 c	0.15 ± 0.01 b	17.7 ± 1.4 a	2.1 ± 0.4 a
	Guar gum	7439 ± 739 e	157 ± 8 e	1261 ± 362 d	0.14 ± 0.01 b	26.4 ± 1.8 cd	2.8 ± 0.5 a
	Xanthan gum	7084 ± 227 de	148 ± 14 e	959 ± 196 c	0.14 ± 0.01 b	33.9 ± 0.6 f	3.6 ± 0.3 b
	Inulin	6744 ± 537 cde	103 ± 12 abc	832 ± 24 bc	0.14 ± 0.01 b	22.1 ± 2.5 b	2.4 ± 0.3 a
	CMC A15	5965 ± 481 abc	116 ± 10 bcd	865 ± 47 bc	0.14 ± 0.01 b	29.5 ± 3.0 e	2.5 ± 0.2 a
	CMC A4M	6199 ± 789 bcd	148 ± 17 e	999 ± 298 cd	0.14 ± 0.01 b	28.2 ± 0.4 de	3.0 ± 0.2 ab
P-value	Hydration	0.23	0.00	0.78	0.03	0.00	0.76
	Hydrocolloid	0.01	0.00	0.00	0.01	0.00	0.00

*means with different letters within the same parameter differ significantly ($P < 0.05$)

4.4 Conclusions

Gluten free noodles could be made from TN flour, giving special attention to the amount of water used in the production process and the type of hydrocolloid added, because they play a crucial role on the dough rheology and the quality of fresh and cooked noodles. Hydration applied during noodle processing significantly affected the dough rheology during mixing, heating, and cooling, but also the luminosity, hardness and firmness of fresh noodles and the color and firmness of cooked noodles. Instead, hydrocolloids type affected properties of both fresh and cooked noodles. Overall, gluten free TN noodles made with xanthan gum and adapted amount of water showed the best quality considering the lowest cooking losses obtained and its higher firmness.

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5. Introduction Results III

Snacking: ingredients, processing and safety

In Cereal-Based Foodstuffs: The Backbone of Mediterranean Cuisine. Ed. Fatma Boukid, 2020. Springer Nature Switzerland AG. Sent for publication.

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Abstract

Urbanization has changed worldwide consumers' lifestyle and consequently their dietary habits. Consuming foods or snacking between main meals is becoming a growing practice in people's lives, initially for reducing the starving sensation but later on as mainstream meal. This upward trend is confirmed by the exponential increase in sales of the snacks industries. The term snack includes a great variety of small pieces foods. Nevertheless, this chapter will be focused on the cereals-based snacks originally from the Mediterranean area, as well as a reference to other initially autochthonous snacks that are becoming globally popular is included. In this chapter, main cereal-based snacks are considered, explaining their production process, ingredients interactions, quality characteristics and safety issues, with especial emphasis on the extrusion process because of its importance in the snacks market. Likewise, an overview of the trends driving snacks innovation is presented, particularly related to the search of alternative ingredients and new technologies applied to this food category that is very prone to adapt towards an ever-changing future.

5.1 Introduction

Snack and snacking are now very common terms adapted to consumers lifestyle. Nevertheless, meaning of "snack" and "snacking" is still very confusing. During the last years several studies have been carried out trying to clearly describe snack food and snacking behavior, but until now there is a lack of an unanimously recognized definition. According to some publications in scientific literature, "snack" meanings are principally focused on the daily moment of an eating event [1] [2], kind and quantity of eaten food, place of the eating occasion and on a conjunction of these factors [3]. Therefore, the term snack would be related to the rapid consumption of a small food between meals that does not require sitting (Figure 1). Frequently, the global snack food is segmented into salted snacks, bakery snacks, confectionary, specialty and frozen snacks. Nevertheless, the highest market segment is covered by bakery snacks.

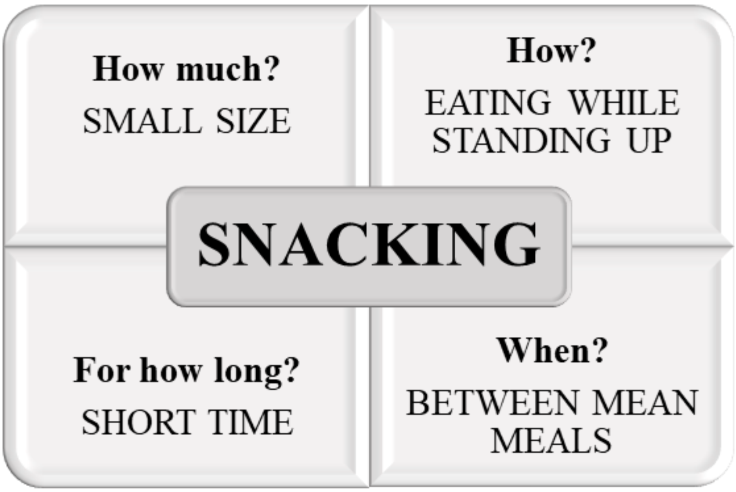

Figure 1: Snacking features. Adapted from Drapeau, Pomerleau, and Pomerleau (2016)

Snacks food market is rapidly growing as a consequence of changes in consumer lifestyle, rapid urbanization, and most of it owing to the increasing demand for on-the-go convenience foods. Even distribution channels have fueled this trend extending accessibility to these products through specialty stores, independent stores, on-line sales, convenience stores, supermarkets, malls, and also vending machines. This is a global trend with USD 605 billion in global sales in 2018 and with a projection of growing at a CAGR of 5.34% during 2020-2025 period [5]. Snacks food are not immune to the healthy and environmentally friendly drivers, and functional ingredients, organic foods or green packaging technology are fueling the innovation in this sector. Considering that urbanization and consumer lifestyle are the main reasons that have motivated the expansion of snacks, Europe is leading the market closely followed by North America. According to the data presented by Forecast [6] in August 2019, European snacks market reached the value of USD 107.07 billion in 2016 and it is expected to increase at a CAGR of 5.2 %, to achieve USD 220 billion by 2021. In Europe, the average per capita consumption amounted to nearly 4.6 kg in 2020 [7].

The Mediterranean basin is recognized for its rich gastronomic history and the beneficial effects associated to it [8] [9] [10]. Across the Mediterranean area, “snack” principally refers to a small food ration consumed in a short period time (eating while standing up) between main meals (breakfast, lunch and dinner), with a purpose of reducing the sense of hunger until the next regular meal. The rich gastronomy that characterizes the Mediterranean area is also extended to the snacks concept. Nevertheless, despite the variety of foods that can be consumed as snacks, this chapter will be focused on those snacks that are based on cereals. Specifically, in the Mediterranean area, a variety of grains including wheat, maize, oats and rice have typically been used as snacks ingredients, and in rather minor proportion others like rye, sorghum, millet and triticale. Moreover, in the last fifteen years, food industry has started adding pseudocereals and pulses to maize and rice to produce gluten free snacks, one of the growing food niches. The present chapter discusses the most popular cereal-based snacks from the Mediterranean area with an emphasis on ingredients, processing and safety. It also examines the nutritional quality, the improvements occurred during the last years and the new trends in terms of snacking. Given the wide variety of cereals-based snacks, a technological classification (Figure 2) based on their production process has been used to better categorize them. In the following sections, the production process and the characteristics of the snacks included within each category are described.

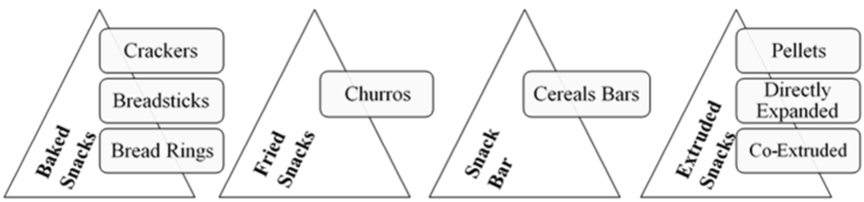

Figure 2: Technological classification of cereals-based snacks.

5.2 Baked Snacks

Many bakery cereal-based products fit in the category of snacks, like crackers, breadsticks and bread rings. Generally, they are produced from wheat following the main breadmaking stages: mixing, fermentation (yeast or chemical) and baking. This last step, baking, is the one that mostly characterizes this type of products. Through baking process, heating converts a raw dough into a crunchy crumb. Volume expansion, non-enzymatic browning reactions, yeast and enzymatic activities inactivation, protein denaturation, starch gelatinization and moisture loss represent the key transformations that occur during baking [11]. In the following sections, specific mention to the different baked snacks is included.

5.2.1 Crackers

Generally, crackers (Figure 3 a) are prepared from wheat flour but can be divided into three types (saltine, savory and chemically leavened) depending on the production process. Saltine requires a prolonged yeast fermentation (about 19 h) to get the typical dough sponge. After yeast fermentation additional ingredients might be added, like sodium bicarbonate to lower pH, and then dough is left to rest for additional 4-6 hours. Process continues with dough sheeting that is carried out using sheeting rolls with docking pins leading to a sheeted dough with 6-8 layers. The use of docking pins is needed to produce the dough cavities that facilitate the migration and evaporation of inner water during baking. Laminating process is essential for this type of crackers because it allows: improving the gluten network development, obtaining a characteristic layered structure and introducing other ingredients, like fat to generate the layered dough [12]. During baking, the thin dough sheets are subjected to high oven temperature (around 250 °C) for short-time (5-6 min) causing a fast-vertical expansion that results in the typical crumbly structure after baking.

Conversely, chemically leavened and savory crackers do not require fermentation time because chemical leavening agents (usually sodium bicarbonate, monocalcium phosphate) are used to get the desired expansion. In this case, the volume increase is primarily due to the chemical reaction of sodium bicarbonate with acid, followed by the thermal decomposition of soda alone and the water loss in form of steam during baking [13].

To imprint some specific sensory characteristics some oils and flavor (savory snacks) can be sprayed after baking. Final moisture content of most crackers is around 2%. From nutritional point of view, crackers are characterized for a very low amount of sugar, a moderate fat level (10% to 20%) and low dietary fiber content [13].

5.2.2 Breadsticks

Among the most appreciated baked snacks in the Mediterranean area, breadsticks take up an important position. These products are known as “grissini” (Figure 3 b) in Italy, “rosquilleta” (Figure 3 c) in Spain and “kaki” in Tunisia, but the production technology and the raw materials used are rather similar. The basic breadstick is a crispy elongated bread (40-80 cm) slightly salted. The most common varieties are made mixing wheat flour, water, salt, either oil or fat, and yeast or a chemical raising agent [14]. In the case of the Spanish snack, it may also be combined with peanuts, sunflower seeds, cheese or chocolate. Regarding the making process, after mixing the dough is fermented for 90-120 minutes and then cut into strips ready to be baked at 250 °C for 10 min in the case of grissini [15] and at around 160 °C for 30 min in the case of "Rosquilleta".

5.2.3 Bread Rings

Bread rings also known as "taralli" or "tarallini" (Figure 3 d) are frequently consumed as an appetizer during many occasions in Italy. These savory snacks are originally from the South of Italy (Apulia region) and usually they are flavored with fennel seeds, onion, pepper or simply salt, but also it is easy to find a sweet adaptation of them. These snacks have a rounded shape with a variable diameter from 2-3 cm to about 9-10 cm, and a thickness from 7 to 8 mm to 1.5 cm 1-1.5 cm [16]. As breadsticks texture, "taralli" is appreciated for its crunchy and friable sensation in the mouth. Core ingredients of "taralli" are wheat flour (sometimes re-milled semolina), olive oil, yeast, wine and salt. Ingredients mixing and kneading (20 min) are performed using an arms kneader followed by sheeting and shaping process, although in the past process were more artisanal. The raw "taralli" obtained from the previous process is immediately submerged in salty boiling water for 2 min and then rested for 30 min before baking at 230 °C for approximately 30 min. In past times, boiling process allowed stopping spontaneous fermentation before baking, because "taralli" were produced in public municipality bakeries [17]. Nowadays, the process has been automatized and dough hand-shaping is frequently substituted by a "tarallatrice", which is a specific machinery to shape rolled strips of dough enhancing the productivity [18]. To guarantee their characteristic crispy texture, "taralli" snacks contained around 20% fatty materials, and thus, prone to oxidations. In fact, Caponio et al. [16] studied the impact of processing and storage on the fatty fractions of "taralli" confirming that oxidation led to the formation of volatile compounds like aldehydes and polymerization complexes, both of them impairing negative characteristics to this snack. Specifically, aldehydes because they are related with off-flavors that reduce the product shelf life and consumers acceptance, while the polymerization compounds may have adverse effects on human's health. Because of the impact of fats on the quality on this snack, the effectiveness of different oils have been compared [19]. The effect of four different types of oil (extra virgin olive oil, olive oil, olive-pomace oil and refined palm oil) was evaluated on the final quality of this bread rings. Regarding visual appearance and odor, "taralli" containing extra virgin olive oil reached the highest score in the sensory test. Furthermore, samples with extra virgin olive oil had the lowest content of triacylglycerol oligopolymers and no presence of trans fatty acid isomers was found with respect to the other oils used in this study [19].

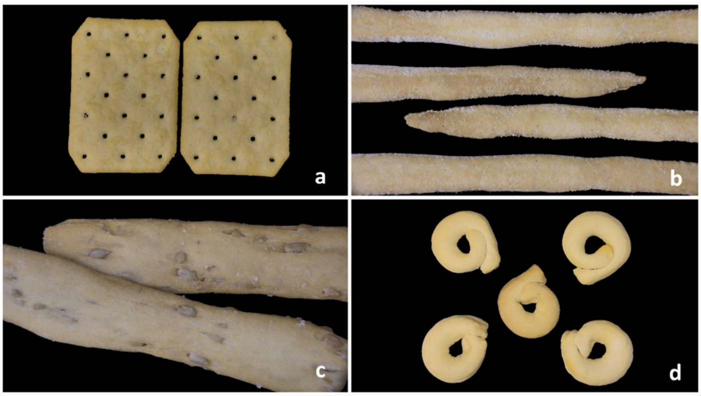

Figure 3: Baked snacks. a: crackers; b: bread sticks "grissini"; c: bread sticks "rosquilletas"; d: bread rings "tarallini"

5.3 Fried Snacks

Other type of snacks is obtained applying frying, in which fat or oil are the heat transfers to food. The direct contact between food and hot oil (160-200 °C) and fast heat transfer are responsible for the characteristic dual structure of these snacks, with a dry, crunchy and golden crust and a soft inner core. Principal physio-chemical modifications in fried snacks include starch gelatinization, protein coagulation, Maillard reaction, moisture loss with a concomitant oil uptake and texture changes [20]. Plenty of different snacks could be identified worldwide within this category, but following section is restricted to the typical fried snacks located in the Mediterranean area.

5.3.1 Churros

Sweet fritters are widespread everywhere in Spain. The Spanish word "churro" (Figure 4) refers to a deep-fried sweet dough, especially consumed during breakfast or as a snack. Basic ingredients are wheat flour, water and salt, which are mixed until obtaining a pretty sticky and fluid dough or batter. Then, a press called "churrera" allows extruding the dough that is cut it into small pieces directly submerged into boiling olive or sunflower oil. The raw strips are deep-fried, usually for 3-4 min at 185-200 °C, although those are quite variable, in fact, the golden color and crispy crust appearance are used as process end point marker [21]. Owing to their carbohydrate content and the high temperatures reached during frying, some undesirable and possibly dangerous compounds such as acrylamide or 5-(hydroxymethyl)- 2-furfural may be found in the final products [22]. Therefore, some studies have been carried out trying to identify and reduce these complexes molecules. Morales, Arribas-Lorenzo [21] studied the impact of frying temperatures and times on the levels of harmful compounds. For that, churros were deep fried in sunflower oil at 180, 190, 200 °C for 2, 3, 5 and 7 min and compared with commercial samples. Average values of acrylamide in commercial products were half of that found in the experimental products, while commercial samples contained higher amount of 5-(hydroxymethyl)- 2-furfural. The major increase in the acrylamide level was observed in the range 190-200 °C, suggesting the need for a more accurate control of the frying temperatures to limit the formation of this unfavorable compounds [21]. Mild vacuum frying (about 21 kPa) may be an attractive alternative to cut down the formation of these harmful molecules [23]. In fact, data coming from vacuum frying (21 kPa) at 100, 120 and 140 °C were compared with those acquired from the traditional frying (atmospheric pressure) at 140 and 180 °C, observing a significant reduction in the 5-(hydroxymethyl)- 2-furfural content [23]. Simultaneously, vacuum frying reduced the non-enzymatic browning with hardly impact on the snack color, compared with samples fried following the manufacturer recommended conditions (180 °C, 3 min). Besides, "churros" treated under vacuum conditions were crunchier than those fried at 140 °C under atmospheric pressure [23].

Figure 4: Spanish sweet fritters "churros"

5.4 Cereal Snack Bar Type

At the beginning of the '90s, a new product, which can be categorized within snack foods, was introduced into the world market. It was presented as a healthy choice for the consumers interested in a wholesome diet [24]. Nowadays, cereal bars (Figure 5) have reaped more relevance and acceptance worldwide. In fact, the global snack bars market has reached the value of USD 20.15 billion in 2018 and is supposed to increase at CAGR of 6.64% from 2019 to 2025 [25]. Generally, cereals bars refer to a rice- or oats-based snack combined with several ingredients such as chocolate, fruits, raisins, nuts or seeds. They have been consumed as a snack but for many occasions even as breakfast, energy food, meal replacement or sometimes for weight control [26]. The basic production process of cereal bars consists in mixing the dry and wet raw materials following the dough portioning and baking, varying time and temperature depending on the characteristics of the final snacks. In some type of cereal bar snacks, the baking phase is not needed, and the ingredients blend is just divided in the chosen shape. For both kinds of cereals bars, extra processes can be included, like filling, coating with assorted glazes, drying, etc. [27]. Usually, they are marketed as an individual portion of 25 to 30 g, thus the last step is packaging.

Figure 5: Cereal snack bar type

5.5 Extruded Snacks

Extrusion process was introduced for the first time in 1797 by Joseph Bramah, for making lead pipe and during the last 250 years this process has been employed to produce plastics and synthetic stuffs. At the beginning of the seventies, extrusion cooking was introduced in the food industry [28]. New opportunities of mixing different ingredients and the design of innovative layouts for extrusion equipment have allowed producing novel foods with new structures, increasing the range of their application in the food industry. Because of that, extrusion process has been extended to the production of pasta, breakfast cereals, puffed rice, texturized protein, instant drinks, meat analogs, pet foods and snacks [29]. In 2019, the global market for extruded snacks was around USD 48.3 billion and according to forecasts in 2026 will reach USD 65.2 with a CAGR of 4.4%. Mostly, the increase is associated to the growing available income and modifying lifestyle among the new generations [30].

In the extrusion-cooking ingredients are mixed and then subjected to heating, high pressure, shear and afterwards forced to flow through a die with a precise shape [31]. Final quality of the extrudate foods is influenced by: raw material type, feed moisture, barrels temperature, screw configuration and speed [32]. Considering the huge number of marketed extruded snacks, their classification is rather difficult, but it has been divided in three major groups: pellets, directly expanded snacks and co-extruded snacks.

5.5.1. Biochemical changes occurring during extrusion

Food products, which only undergo simple physical treatment have proven to be healthier [33][34]. Despite extrusion is a short time cooking technology, temperature and shear force reached in the barrel are able to cause important changes in macro and micronutrients of the raw materials.

In cereal based extruded snacks, starch is the main constituent and gelatinization during extrusion of cereal-based blends play a crucial role for defining snack features. Starch is a polymer constituted by a mostly linear polymer named amylose (20-30%) and a branched polymer termed amylopectin; and depending on the ratio between these two polysaccharides, starch can assume different properties in terms of viscosity and gel formation [35]. During extrusion amylose and amylopectin are subjected to shear force and thermal energy that may reduce their molecular weight also changing the crystallinity of the structure [36]. In fact, this phenomenon has been observed in maize starch [37], rice starch [38], banana starch [39] and sweet potato starch [40]. The consequence of the rupture of covalent hydrogen bonds within starch molecules is an increase of the starch digestibility [41]. Nevertheless, simultaneous extrusion-cooking may contribute to the formation of amylose-lipid complexes [42] and resistant starch [43], and both of those phenomena resulted in a decrease of the starch digestibility, which is considered nutritionally advantageous. Those phenomena could be promoted by selecting appropriate extrusion conditions, for instance, an increase in barrel temperature results in an increase of starch-lipid complexes, which in turn are strongly correlated to the moisture content of the blend [44].

Other nutrient that has technological impact in the extrusion process, apart from nutritional implications, is the dietary fiber. In general terms, dietary fiber is described as plant polysaccharides and lignin that are resistant to hydrolysis by digestive enzymes in human, and its intake promote beneficial health effects that have been largely demonstrated and worldwide accepted [45]. Based on its solubility in hot water, it can be classified in soluble and insoluble fiber [46]. Extrusion process can affect the dietary fiber functionality, particularly temperature and screw speed. Lately, several studies demonstrated that soluble fiber content increased with the extrusion of high insoluble fiber raw materials [47][48], [49]. For instance, when subjecting whole-grain wheat flour to extrusion, a significant increase in dietary fiber and resistant starch was observed in maize based extrudates, while total and digestible starch decreased [50]. Again, water content of the blends plays an important role during extrusion processing because insoluble and soluble fibers behave differently. In fact, insoluble fiber is responsible for the volume decrease in extruded products while soluble fiber improves the expansion with less effect on bulk density [51].

Regarding proteins, being thermolabile compounds, they are affected by extrusion-cooking. This technological process may facilitate the formation of non-covalent molecular interactions, covalent cross-linking and protein-lipid-starch interactions [52]. Moreover, shear force and high temperature cause a conformational alteration named denaturation, which makes protein sites more available for the proteases with a consequent increase of the *in vitro* protein digestibility [53]. The deactivation of antinutritional factors (protease inhibitors) might be also involved, favoring the accessibility of the proteases [35]. Actually, some studies have reported the destructive effect of extrusion towards antinutritional compounds like phytic acid, polyphenols, oxalates and trypsin inhibitors, highlighting temperature as a key factor in this reduction [54] [55]. This has a direct consequence on the bioavailability of minerals in extruded foods, which is enhanced by the disruption of some antinutritional compounds through extrusion-cooking [56].

The role of fat in extrusion have been extensively analyzed due to its great impact on processing performances. In food extrusion, fat mainly comes from raw materials and added ingredients. Cereals like wheat and rice have a low-fat content (< 2%), but content could be higher when including oats (10%) [35]. Raw materials with fat level higher than 6% are not recommended for the extrusion process, especially when the target product is an expanded snack. In fact, high fat content increases creep in the barrel, with consequent torque and pressure reduction, which lead to less expanded product [57]. However, when fat presence is < 6 %, it behaves as a plasticizer or emulsifier [58]. High temperature and force shear transform fat into liquid oil that is expelled from the die because of the high pressure [59]. Likewise, when high temperatures are reached a lipase and lipoxygenase activity decrease is observed with a reduction of fatty acid oxidation [60]. Total fat content in the extruded food may result lower than in the raw ingredients due to the complex lipid-amylose formation; this aggregation often occurs when raw materials with high free fatty acids and high amylose content are processed [61].

Other compounds that could be highly affected by the extrusion conditions are the bioactive constituents. Food antioxidant activity is related with phenolics compounds and their presence and functionality will depend on the extrusion process conditions. In general, high temperatures and high moisture content are responsible for the decarboxylation of phenolic compounds, thus their loss after extrusion [62]. Moreover, an antioxidant activity reduction of 60-68% and a decrease of 46-60% of total phenolics were found in barley extrudates [63]. Nevertheless, simultaneously extrusion can improve the level of these bioactive molecules. In fact, a progressive enhancement of the total phenolic compounds content was observed in extruded black rice when extrusion temperature increased and die pressure diminished [64]. In the case of vitamins, owing to their different structure and composition, their stability during extrusion may vary. Athar et al. [65] investigated the impact of extrusion on the vitamin retention in extruded foods. Authors reported that high barrel temperature and low feed moisture provoked ascorbic acid degradation in extruded snack. In whole oat grain and maize with pea grits, vitamin B retention was not related with its initial level, but it depended on the cereal type [65]. Among the fat-soluble vitamins, vitamin D and K are relatively stable compared with vitamin A and E [35]. Indeed, in oat, barley, wheat, rye and buckwheat a loss of 30% in tocopherol and tocotrienol was described [66]. Zieliński et al. [67] reported a reduction of about 63% in vitamin E when buckwheat groats were subjected to extrusion.

5.5.2 Safety issues related to extrusion process

Pertain to the cereal-based product, the issue of food safety is particularly crucial to preserve consumers health. Mycotoxins represent the main risk coming from plant-based commodities; particularly, those globally recognized as economically and toxicologically important: fumonisins, aflatoxins, deoxynivalenol and derivatives, zearalenone and derivatives and ochratoxins. Industrial food processes can somehow reduce the mycotoxins content in the final products. In the extrusion technology, this extent of the reduction may depend on the extruder and screw type, die configuration, initial mycotoxin concentration, barrel temperature, screw speed and raw material feed moisture. In cereals, fumonisins, aflatoxins and zearalenone levels declined by 100, 95 and 83%, respectively; while deoxynivalenol, ochratoxin A and moniliformin content dropped down by 55, 40 and 30%, respectively [68]. Another food safety problem related to extrusion is the acrylamide formation along the extruder barrels caused by high thermal and mechanical energy developed during the process. Acrylamide is one of a resulting products from the Maillard reaction, and it has been categorized as potential carcinogen to humans by the International Agency for Research on Cancer [69]. The amount of acrylamide formation is greatly dependent on the type of cereals used for the extrusion and particularly, their content of free asparagine. In fact, acrylamide formation requires the presence of this amino acid and reducing sugars (glucose) [70]. Because of the higher asparagine content, rye based extrudates contain higher levels of acrylamide compared to extrudates made by rice, maize and wheat [71]. A study carried out by Mulla et al. [72] investigated the effect of the extrusion parameters and some mitigating agents on the acrylamide formation in potato flakes and semolina blends. Acrylamide content was higher in those blends with more potato flour and it increased with low moisture and high die temperature. Calcium chloride at 50 mmol/g was able to reduce by 65% the level of acrylamide.

5.5.3 Types of extruded foods

5.5.3.1 Pellet Snacks

In general, pellet snacks (Figure 6 a) have similar characteristics to the starting dough. They are also known as unexpanded snacks. In fact, after extrusion they are considered like a half-product, which need an additional step to get the final expanded shape. For their preparation two kind of ingredients may be used: raw or precooked starch-based materials. During extrusion moisture level is kept around 25-35%, which does not allow the complete swelling of the starch granules and neither their gelatinization. At this stage, temperature ranges from 100 to 120 ºC, then the viscous material is cooled passing through the last extrusion module at around 80 ºC till reaching the die. In this type of snack, the absence of any air bubble in the final product is of key importance. For that purpose, after the extrusion the pellet is typically cut as a thin sheet (1 mm thick) and gently dried (50-60 ºC for 6-7 h) until reaching a hard-glassy consistency with moisture content of 10-12%. For pellet expansion, frying, baking or microwaving are employed to promote rapid heating that will convert water into vapor leading to puffing food [73]. At the end of the process, expanded pellets have a moisture content of 1-2% and they are generally subjected to a flavoring step to enhance the taste and the flavor [74].

5.5.3.2 Directly Expanded Snacks

This category represents the first form of industrially produced snacks. In the 1940s, Adams company started to produce this kind of snack using a single screw extruder and maize as ingredient. Usually, this extrusion requires low amount of moisture (16-18%). Nowadays, directly expanded snacks (Figure 6 b) are attracting more attention due to their sensorial quality like crunchiness. In fact, among the desirable qualities for a snack are high expansion rates, low densities, high specific volumes, light colors, and high freshness [75]. For the production of these snacks, the temperature in the first module of the extruder ranges from 80 to 150 ºC, hence starch granules from maize, wheat or rice start to lose their organized crystalline structure becoming amorphous, then they are squeezed in the screw reverse section and dispersed into the new viscous matrix. Pressure reached during the extrusion process is around 40-80 atmospheres [76]. When the hot-viscous material goes out through the die, pressure drop off causing the water vaporization. The high temperature and pressure reached during the process, contribute to the formation of air cells resulting in the expansion of the product. The expansion ratio depends on both the die shape and viscosity of the material in the barrels [28]. When the viscous starch-based matrix cools down, glass transition starts, which usually occurs at 40-60 ºC with 5-8% moisture content, resulting in the final brittle puffed structure, typical of the directly expanded snacks [73]. The shape of the snack depends on the die and the cutting action of the knives that makes possible the new tridimensional structures. Conventionally, flavorings of these snacks were coated at the end of the process, but new trends dictate the use of some flavor precursors (reducing sugar, amino acids and peptides) added directly into cereal blends (maize, wheat and rice), which with the high temperatures give rise to new colors and flavors [77] [78].

5.5.3.3 Co-extruded Snacks

Coextrusion technology involves the combination of two different materials in the extrusion die, coming from two extruders or from an extruder and a pump [79]. This technology extends the characteristics of the extruded foods varying textures, colors and flavors. Generally, the external part is composed by a dried cereal-based mixture, while the filler may be a material with solid, creamy (Figure 6 c) or gel-like consistency (fruit jam, ketchup, lemon cream or cheese cream) [80].

Figure 6: Extruded snacks. a: pellet snacks; b: directly expanded snacks; c: co-extruded snacks.

5.6 New Trends in Snacking

For decades, people have mostly considered as snacks, chips, biscuits and chocolate. They consumed this kind of "junk food" thinking of eating something with a reduced nutritional value. But the adoption of new lifestyles is pushing an increasing number of consumers, mainly new generations, to replace traditional meals with on-the-go snacks. Healthier and dietary patterns are driving the consumer selection for these products, making them very appropriate carriers for nutrients or bioactive compounds. For this purpose, food research has put the attention on nutritional enrichment of cereal-based products to improve consumers health. Although breads have received very much attention in the enriching strategies by adding pseudocereals, legumes and lately fruits and vegetables [81], snacks are following the same trend too [82]. With regards to snacks, the enrichment strategy has been quickly extended and it became even easier to find in the market snack foods made by pseudocereals, pulses and seaweed. Those trends have encouraged producers to look for alternative or new ingredients that imprint the innovative character. From a technological point of view, there is constant innovation in processing technologies for creating alternative foods within the cereals-based snack segment. The implementation of new technologies is allowing to increase efficiency, to reduce the environmental impact and to develop new functional and healthy foods. Moreover, with the recent increase trend of having and sharing new cooking and sensorial experiences, people are more and more attracted towards this short bites' foods for initial tastings of diverse foods. This trend is global, those products are not really from the Mediterranean area, but it is the general consumers' tendency. In fact, most of this products, present new characteristics in terms of texture, color and flavor that contributes to the attention-grabbing features.

5.6.1 Ingredients

Many of different ingredients have been tested in snack foods, and some recent examples have been selected to highlight their importance. In the search for alternative ingredients, it has been observed two motivation drivers; first, to explore for ingredients that might confer additional healthy benefits to the snack food [82], and secondly, immersed in the sustainability trends, to investigate the revalorization of by-products by using them as unconventional ingredients in the snack production. For instance, some researchers have evaluated the effect of the incorporation of brewer's spent grain in breadsticks [83]. Dietary fiber content increased in breadsticks containing brewer's spent grain, although they were significantly darker, less crispy and had lower volume [83].

Pseudocereals, pulses, algae and fruits and vegetables are being included as ingredients in the snacks production, either as powders, flours or some specific extracted fractions with nutritional interest. Commonly snack bars are composed by a nutrient-poor ingredient and due to this they cannot be classified as functional foods. In order to overcome this weakness, researchers and manufacturers began focusing on the snack bar enrichment with bioactive compounds so as to ensure more healthy choices [26]. For instance, adding bean flour to oats-based snack bars allowed increasing the dietary fiber and protein content and the antioxidant capacity [84]. Gluten-free has been a driving force in the innovation of the food market, reaching also to snacks foods. Many studies have been carried out with this focus, introducing pseudocereals and pulses. With that purpose, buckwheat flour (10, 20 and 30%) was added to maize for making an extruded snack, improving the diameter, redness, phenolic content and antioxidant capacity, while reducing bulk density and water absorption index [85]. Cueto et al. [86] reported the impact of quinoa and chia flour (20 and 5%) on the physical characteristics of extrudates made by maize. Chia flour increased density and crunchiness but lowered expansion index, whereas quinoa extrudates showed smoother structure than those containing chia. Regarding pulses, lentil based extruded snacks show high content of some prebiotics (raffinose and stachyose) [87]. Beans were also incorporated into extruded snacks made with blends of carob fruit and rice with a subsequent enhancement of the phenolic compounds [88]. Other non-conventional seeds with an attractive nutritional profile have also been used like flaxseed, which is rich in α-linolenic acid, lignans and dietary fiber [89]. Flaxseed flour added at different levels (6%, 12%, 18%) to oat-based bars affects their color, decreasing lightness and increasing redness, but provides a significant increase in polyunsaturated fat and dietary fiber with good sensory acceptance up to 12% addition [90].

Several studies have been carried out exploring the incorporation of algae in snacks foods. In the study carried out by Batista et al. [91] the influence of four microalgae (*Arthrospira platensis, Chlorella vulgaris Allma, Tetraselmis suecica* and *Phaeodactylum tricornutum*) addition (2 and 6%) was evaluated on crackers quality. Interesting results were obtained, since no differences in terms of structure were observed, thus gas retention was not significantly altered by microalgae adding. Samples with 6% of *Arthrospira platensis* and *Chlorella vulgaris Allma* presented the highest protein content. *Arthrospira platensis* samples reached the highest antioxidant activity and achieved better sensory analysis scores. Conversely, *Tetraselmis suecica* and *Phaeodactylum tricornutum* received the lowest liking score in the sensory test. The effect of algae was also tested in breadsticks by [92]. Authors evaluated the addition (1.5%) of two types of microalgae (*Chlorella vulgaris* and *Arthrospira platensiswere*) to wheat-based breadsticks. Microalgae addition decreased hardness, resilience, crispiness and brittleness of the breadsticks, but nutritionally snacks had high mineral content, specifically iron and selenium [92].

Among the most consumed cereals bars, those containing fruits have great popularity, especially those with certain berries, likely related with the perception of their health benefits besides their appearance and taste [93]. In a recent *in vivo* study, freeze-dried black raspberries (10% and 20%) and cranberry extract (0.5% and 1%) were incorporated into rice crisp cereal bars, comparing the results with those of a reference bar [94]. Raspberries bars dulled postprandial insulin peak and slightly enhanced the glycemic responses to a high carbohydrate food [94]. Likewise, fibers coming from grapes and orange seeds were used (2.9%) to prepare crackers, with the additional benefit of increasing antioxidant capacity and phenolics compounds [95]. Fibers from cooked pear apple co-product were incorporated in cereal bars [96], showing high antioxidant capacity and retaining a good amount of phenolics compounds [97].

More exotic ingredients added to crackers are the residues of *Hibiscus sabdariffa* [98]. When they were incorporated at different levels (1.25%, 2.5%, 3.75%, and 5%), crackers contained less protein and fat while more ash and total dietary fiber compared to the control (0%). Authors found that adding 5% of *Hibiscus sabdariffa* increased phenols and flavonoid content from 5.99 to 17.57 mg/g and from 49.36 to 104.63 mg/g, respectively; consequently, enhancing twice the 2,2-Diphenyl-1-picrylhydrazyl (DPPH) radical scavenging activity. Sensory test decreed that crackers with 3.75% of plant incorporation was the most acceptable sample [98].

One of the biggest challenges for the quality improvement of savory snacks that is still ongoing is salt reduction. In most of salty snacks, sodium chloride is employed as a flavor enhancer. The exceeding of sodium daily intake (10-20 mmol/day) may increase blood pressure and affect cardiovascular health [99]. In the last years, the presence of salt reduced products on the grocery stores shelves has raised, in which sodium has been replaced with potassium, calcium, magnesium and other flavor enhancers [100]. Fat reduction in the extruded snacks have been approached modifying processing technologies and with new ingredients combinations. For instance, a reduction of 15% of oil level was achieved by spraying flavors contained in an aqueous hydrocolloid solution rather than traditional oil-based solutions [101].

5.6.2 Technological innovations

5.6.2.1 Supercritical Carbon Dioxide Extrusion

As mentioned before, extrusion cooking is commonly applied in cereal-based snack production because of its flexibility. Nevertheless, high temperatures may originate some disadvantages like Maillard reaction products [102] and antioxidant activity reduction [103]. To avoid these phenomena and to produce high-quality snacks, in the last years a new extrusion system has been implemented in the food industry. Carbon dioxide supercritical fluid has been incorporated to the traditional extruder. This invention allows obtaining a large variety of puffed snacks at lower temperature (< 100 °C) [104]. Among the advantages of this technology, energy savings, preservation of highly nutritional compounds and blocking the formation of harmful molecules released at high temperatures are counting the most. Carbon dioxide is a super critical fluid, behaving in between gases and liquids at temperatures in the range 60-80 °C. Carbon dioxide rapidly solubilizes in the blend, generating the nuclei that will produce the air pockets when the pressure drops at the die exit [105]. Thus, gas expansion and diffusion are responsible for the snack expansion, which starts from the core and progressively migrates to the edge. Resulting snacks have smooth and uniform surface and regular shape with internal porous [106]. Many researches have applied this technology for producing new cereal-based snacks with some nutritional impact. In lentil and pre-gelatinized potato starch extrudates, total phenolics and DPPH radical scavenging activity increased by 30% and 18%, respectively [107]. Bilgi Boyaci et al. [108] demonstrated that CO_2 cold extrusion increased the retention of some thermolabile compounds such as thiamine and riboflavin in maize extrudates. The role of supercritical CO_2 as plasticizer and blowing instrument was also studied in puffed rice fortified with protein, dietary fiber and micronutrients, retaining all added minerals, 55-58% of vitamin A and 64-76% of vitamin C, besides complete bioavailability of lysine (98.6%) that was not blocked by Maillard reaction [109]. Nevertheless, there is much way ahead for this technology to become fully exploited.

5.6.2.2 3D Printing

Digitalization revolution has also reached snack production and 3D printing technology opens great opportunities to the food industry. This innovative process is digitally controlled by a robotic system that allows manufacturing three-dimensional objects built on a layer-by-layer deposition [110]. Until now, this futuristic approach has been applied in different field as medicine, pharmaceutical, biotechnology, engineering and more recently has been applied in the food industry [111]. Moreover, 3D printing could be the technological tool for making at home tailored made foods and more personalized nutrition [112]. Due to its low cost, ease to use, less waste, versatility and customizability, food 3D extrusion has become one of the most extensively explored technology [113]. Using this modern approach, edible ingredients with soft consistency are loaded into a cylinder and by a piston force, they are extruded through a nozzle in sequential layers that are then closely adhered [114]. The 3D shape is previously designed by a software that communicates with the 3D printer to produce the desired design. Essentially, 3D extrusion process is articulated in five main steps: powder preparation, binding method selection, binder selection, process specifications definition and post processing operations [115]. First applications of 3D printing were based on blends of starch, sugar, maize syrup and yeast mixture [116]. Lately, system optimization has allowed many researchers to carry out new 3D printing food applications. Different 3D products with various matrixes such as cereals dough, sugar powder, processed cheese, meat and fruit and vegetables have been extruded [117]. Krishnaraj et al. [118] applied this technology to produce snacks made by composite flour such as barnyard millet, green gram, fried gram, and ajwain seeds. Less explored ingredients like edible insect have been blended (at 20% addition level) with wheat flour for obtaining high protein snacks [114], while fruit and vegetables have provided nutritious tailored made fruit-based snacks [119]. Nevertheless, the manufacturing of complex food matrixes with this printing technology is still a challenge and a deep understanding of food rheology is needed to boost this food technology [120].

5.6.3 Other snacks globally consumed

5.6.3.1 Tortilla chips

In recent years, tortilla chips (Figure 7 a) have achieved a large acceptability among worldwide consumers. Originally produced in Mexico and Central America, they firstly spread to United States and then Europe [121]. In 2018, their global market represented USD 20.28 billion and from 2019 to 2025 it is supposed to reach a CAGR of 4.41% [122]. Mainly, tortilla chips are maize-based and are baked before frying. First step is the production of the "masa" (dough), for that maize kernels are mixed with three parts of water and 1% of lime (maize weight) and subjected to an alkaline (0.2-2% calcium hydroxide maize weight) cooking process (85-100 °C) for 15-45 min [123]. After cooking, the nixtamal is left resting for 8-16 hours and then washed with water to remove the excess of lime and most of the pericarp. Washed nixtamal is grounded in a stone mill until obtaining a consistent dough, named "masa". At this point masa can be kneaded using mixers or extruders, before going to a roll sheeter. A rotating cutter gives a typical triangular shape before baking (280 to 302 °C for 30-45 s) and frying (165 to 195 °C for 50-90 s). Immediately after, tortilla chips are regularly salted and flavored with cheese, hot/spicy, barbecue, lemon salt or jalapeño. To boost shelf life and texture, the traditional formulations may include also gums, emulsifiers, acidulants and preservatives (e.g., sorbates and/or propionates) [124].

5.6.3.2 Hard Pretzel Snacks

Pretzel (Figure 7 b) were introduced in the United States in the late 18th century by German-Swiss immigrants. Since then, pretzel consumption started to grow in the global food market and Pennsylvania occupies the first position in hard pretzel snacks production. Traditionally, these snacks are produced by wheat flour and have the distinctive knot shape. Conventional process consists in mixing, forming with a low-pressure extruder, cooking in a hot alkali solution and two stages baking [125]. Wheat flour, yeast or baking soda are mixed with hot water (38 °C) to obtain a soft dough. During this step, yeast activity must be controlled to avoid a massive gas production and the subsequent rupture of gluten network. After a short resting time, dough is shaped passing through some extruder dies under low pressure. The dough strands obtained are folded into knot shape and cooked in a boiling alkali solution (1-1.5% sodium hydroxide) for about 10-15 seconds. During this step, hot alkali solution gelatinizes the superficial starch and causes the protein hydrolysis. Following, coarse salt is sprinkled on the cooked pretzel and baked afterwards. The previously hydrolyzed carbohydrates and proteins participate in the Maillard reaction that takes places during baking, resulting in the exclusive hard texture and glossy brown color. The first phase of baking reduces the moisture up to 8-10% then is further reduced till 4% using a kiln. Pretzels are packed after cooling down [126].

5.6.3.3 Crispbread

Crispbread (Figure 7 c) has been baked for the first time in the Scandinavian countries, where was prepared from wholegrain rye. Currently, a renewed interest has permitted the development of new crispbreads from different cereals such as wheat, rice, maize and pseudocereals. Its worldwide success is principally attributable to its nutritional quality and its long shelf-life (few months). Traditionally, it is prepared by rolling and sheathing the dough before baking, but the advent of the extrusion technology changed the processing. Flours and usually milk powder, vegetables oil, sugar and salt are premixed and conveyed into a co-rotating twin screw extruder. Here, raw ingredients are cooked by high temperature steam under low moisture (10-15%). Products with an alternative fine cell structure are obtained using supercritical fluid extrusion [127]. The extruded product may undergo an additional toasting process, or it may be coated with chocolate. Packing in foil, trays or cardboard boxes represents the last production step [128].

5.6.3.4 Puffed Grain Cake

Largely consumed in Asia (Japan and Korea) mainly in the rice variant, this product has lately booming up in Europe, where is consumed principally as snack [129]. At the beginning, it was introduced as alternative bread for celiac people, but due to its low-calorie profile and being a good source of fiber, many consumers have come close to this snack [130]. Nowadays, there is a wide range of puffed grain cakes. Besides the classic rice (Figure 7 d), other cereals such as whole rice, maize and rye are used for their production. Some brief information is following to understand the process. The production of rice puffed cake starts with the soaking of raw rice in water until reaching the target moisture content (16-20%, w/w). After that, moist rice is ready to feed the cast-iron mold of the popping machine. The process core is the rice expansion induced by heat and pressure. Molds are heated (190-250 °C) and rapidly closed by a slide lid, which provides the internal vacuum. Meanwhile the steam pressure builds and after 8-10 seconds the rice amalgam expands filling all the available space. Once opening the mold, the fast pressure release causes a flash vaporization of the superheated water and steam. Cake is driven through a belt to the cooling zone where salt or flavor are sprayed before packaging [131].

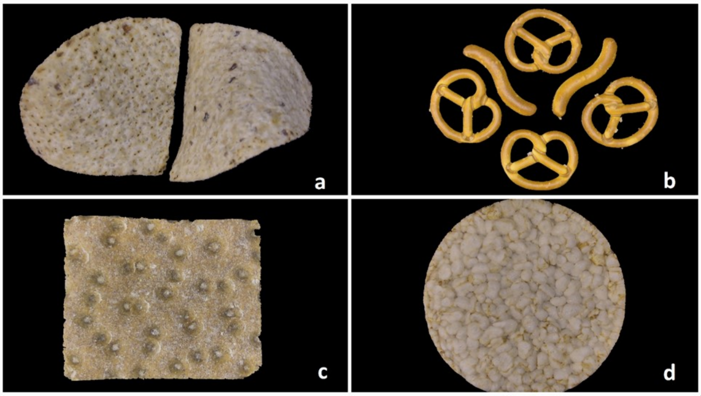

Figure 7: Other snacks globally consumed. a: tortilla chips; b: pretzel; c: crispbread; d: puffed rice cake

5.7 Concluding remarks

In Mediterranean area, cereal-based snacks represent a market segment that is undergoing a fast growth. Technological development and the study of the biochemical interaction among the ingredients have added substantial improvements to the process and product development. The outcomes have been reflected in the launching of snacks with enhanced nutritional quality and food safety. Along with the classic snacks, results of tradition, new ingredients and novel products have appeared in the Mediterranean food market. Concluding, snack products are a constantly evolving foods, and they are strongly related with the costumer's behaviors and on the introduction of new food technologies.

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6. Results III

Tiger Nut (*Cyperus esculentus*) as a Functional Ingredient in Gluten-Free Extruded Snacks

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Declaration: This chapter was written by author Nicola Gasparre that co-designed the study and performed the experimental work. J.P.: methodology, resources, supervision, writing—review and editing; P.L.d.S.A.: methodology, supervision, writing—review and editing; C.M.R.: conceptualization, funding acquisition, investigation, writing—review and editing; J.D.J.B.: conceptualization, funding acquisition, investigation, supervision, validation, writing—review and editing

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Article

Tiger Nut (*Cyperus esculentus*) as a Functional Ingredient in Gluten-Free Extruded Snacks

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Abstract

Tiger nut (TN) is a nutritious source of gluten-free flour, used generally in healthy beverages, but its incorporation in gluten-free extruded snacks has not been explored. TN flour was blended at different concentrations (up to 70%) with rice flour and soluble fiber, for the development of gluten-free snacks on a twin-screw extruder. The effect of TN inclusion in the formulations was evaluated on relevant physiochemical characteristics of the snacks. Viscoamylograph of the raw formulations showed that TN addition increased (p < 0.01) onset temperature and delayed peak viscosity. In the extruded flours, TN contributed to limit the starch degradation during extrusion. Diameter, expansion ratio, true density, and total pore volume of the extrudates were reduced (pf < 0.01) by the increased TN content in the formulations, while bulk density rose. The surfaces of the extruded snacks were modified by the increasing inclusion of TN in substitution of rice in the formulations. Extrudates containing 10% TN showed the best overall texture profile. Moreover, TN addition enhanced the ash and protein content of the snacks and increased their total antioxidant activity. This study demonstrated that incorporation of 10% TN flour into rice-based formulation was suitable for making gluten-free snacks with acceptable physical properties.

Keywords: tiger nut; rice flour; gluten-free; snacks; extrusion

6.1 Introduction

In the last twenty years, patients diagnosed with celiac disease have represented an important public health problem [1]. This event has prompted an increase in demand for gluten-free (GF) food products by consumers with and without celiac disease. In fact, many non-celiac individuals wrongly believe that a GF diet is an essentially healthier choice [2]. The market demand of GF foods has promoted the need for new research to develop products using GF agricultural commodities and novel technologies. Extrusion cooking is a versatile technology that has found dominant uses in the cereal and pet food industries as well as in dairy, bakery, and confections industries. In general, the final extrudate has low moisture content and considered a shelf-stable food product [3]. Extrusion process promotes starch gelatinization, protein denaturation, lipid oxidation and the formation of new complexes occur as result of macromolecules interaction which contribute to changes in microstructure [4] and color [5]. In the last decade, studies have been carried out to develop expanded GF snacks, made mainly from cereal-based mixes, to provide nutritious GF foods to consumers inflicted with celiac disease [6][7]. Most recently, other flour mixes containing unconventional crops and agriculture by-products, such as passion fruit shell, and rice flours [8], plantain and chickpea flours in a corn-based mix [6], blends of apple pomace, corn and sorghum [7] and amaranth, quinoa and kañiwa [9] were also evaluated for the development of GF foods. The interest in alternative agricultural commodities is still increasing.

Tiger nut (*Cyperus esculentus*) (TN) is an underutilized crop with great potential for the development of a variety of value-added, nutritious GF foods, including ready-to eat extruded snack-type products. TN is a tuber generally cultivated in the Eastern region of Spain and Western part of Africa [10]. Their rhizomes are used in some countries for human consumption in various forms. In Spain, TN is only grown and processed into a popular drink called "horchata de chufa" in the province of Valencia. "Horchata" is also gaining popularity in other countries due to numerous health related benefits. The composition of TN is characterized by high contents of insoluble fiber and unsaturated fat and with relatively low concentration of starch [11]. Literature on the use of TN in GF food formulation is limited. Aguilar et al. [12], used a combination of TN and chickpea flour to replace emulsifier and/or shortening partially or totally in GF batters or doughs and breads formulations, and reported acceptable specific volume and darker crust in the GF products. Demirkesen et al. [13] reported that TN flour added up to 25% to the rice flour increased the gelatinization temperatures in GF bread, while a significant reduction of the onset gelatinization temperature and peak temperature was observed when TN flour was added to corn-based biscuits [14]. Gasparre et al. [10], reported the effect of a selection of hydrocolloids on the dough rheology, texture properties and cooking performances of GF noodles made by TN. Only one study has reported using TN as food ingredient to produce extrudates [15]. The authors found the mixes of TN-cassava used in their study difficult to extrude as the TN concentration increased in the mixes, due to the high content of insoluble fiber and fat present in TN [16], that caused pressure drop in the extruder barrel and reduced expansion. Moreover, focusing on the sensory quality of GF cereal-based foodstuffs, TN incorporation (20%) produced the highest overall acceptability scores of corn-made biscuits [17]. It is known that insoluble fiber reduce expansion, while soluble fiber tends to increase the expansion of the extrudate [18], and that fat content can cause slippage of melt into the barrel with consequent low pressure at the die exit [19]. The aim of the present study was to develop GF extruded snacks from TN flour/rice flour mixes and the evaluation of nutritional, physical, and microstructural qualities of the extrudates.

6.2 Materials and Methods

6.2.1 Raw Materials

Tiger nut (TN) flour, short grain rice flour (Koshihikari, amylose: 17.6%) and Nutriose® were used for making the extruded snacks. TN flour had the following proximate composition: 26.3% fat 18.3% fiber and 8.5% of protein, as declared on the nutritional label, was provided by Mon Orxata (Valencia, Spain). Short grain rice was provided by a local rice grower (Richvale, CA, USA) and Nutriose® FM06, a plant derived soluble fiber (SF) with neutral taste, was supplied by Roquette Company (Geneva, IL, USA).

6.2.2 Tiger Nut Milling and Blends Preparation

Preliminary studies were carried out to mill the TN into flour by overcoming the problem related to its high fat and fiber content. Two different mills were tested to archive this goal. A laboratory Cyclone mill (Udy Corp., Fort Collins, CO, USA) fitted with a 0.5-mm screen and chilled grinding surface. Then, a comminuting mill fitted with a 3 mm screen (Model

D, the Fitzpatrick Company, Chicago, IL, USA), which gave the best result. To further reduce the particle size of the obtained coarse flour and avoid stickiness of the TN to the milling surface, due to its high fat content, the coarse flour mixed with dry ice (3:1) and milled on a Wiley mill (Arthur H. Thomas Co, Philadelphia, PA, USA) fitted with a 2 mm mesh. The resulting flour was a finer flour with a mean particle size below 200 μm. Short grain rice was milled in a pin mill (model 160Z, Micron Hosakawa, Köln, Germany) to a fine flour. TN flour was mixed in increasing proportions, 10%, 30%, 50% and 70% with rice flour and 10% SF, which represented formulations, referred from here on as, R1, R2, R3 and R4, respectively. A mix of 90% rice and 10% SF was used as a control, which allowed increasing the fiber content of the final products. The blends were stored at 4 °C until extrusion cooking was carried out.

6.2.3 Modified Feeding Device and Extrusion Processing Conditions

6.2.3.1 Modified Feeding Device

The high fat content in the TN flour made the formulated flours difficult to freely flow through the twin screw of the K-Tron feeder (Model KCL-24-KT-20, K, K-Tron Corp., Pitman, NJ, USA) used in this study, to deliver the correct amount of feed into the extruder. To enable their proper flow, a feeding modification system was built, which consisted of a 4 inches acrylic tube (Tap Plastic, San Leandro, CA, USA) cut lengthwise. The surface of the channel was coated twice with an anti-static solution (food-grade) containing fatty acid in its composition. The made flour conveyor was suspended underneath the feeder discharge chute and adjusted to provide an inclination of about 120 degrees. This modification allowed the proper flow of flour, to the set feed targets of flour, to be delivered into the extruder's feeding port (Figure 1).

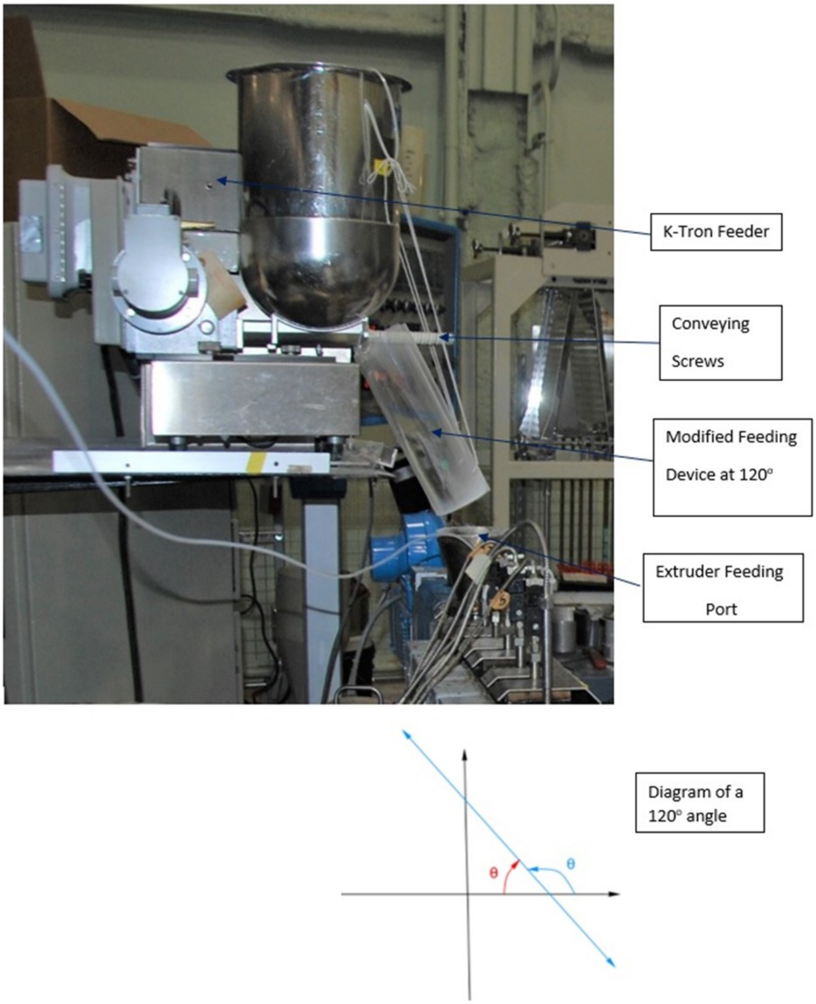

Figure 1: Modified feeding device for suitable conveying formulated flours, containing different levels of tiger nut, into the extruder.

6.2.3.2 Extrusion Processing Conditions

Extrusion of the formulated flours was performed using the Leistritz 18 mm co-rotating twin-screw extruder (MIC 18/GL 30D, Allendale, NJ, USA), equipped with six heating-cooling barrel zones. The heating profile of the barrel zones were set at the following temperatures: zone 1 cooled with tap water at approximately 25 °C, zone 2 at 60 °C, zone 3 at 80 °C, zone 4 at 100 °C, zone 5 at 100 °C and zone 6 at 120 °C. The first temperature was the temperature of the feeding zone and the last one corresponded to the temperature of the die zone (expansion zone). The die was a circular orifice 3 mm in diameter. During the extrusion process, the feed rate and the screw speed were kept constant at 3 kg/hour and 500 rpm, respectively, based on preliminary testing. During extrusion, the moisture content of the formulations was adjusted to 16% by injecting water through a preparatory HPLC pump (Model 305, Gilson, Middleton, WI, USA). The extrudates obtained were dried at 70 °C in a force air drying oven (Imperial IV, Labline, Melrose Park, IL, USA) to a final moisture content of 6%. Extrudates from formulations containing 10%, 30%, 50% and 70% TN flour were referred as E1, E2, E3 and E4, respectively. The dried extrudates were ground by a laboratory sample mill (Cyclone Mill, Udy Corporation, Fort Collins, CO, USA) fitted with a 0.5 mm mesh screen into fine flour for further evaluation. Figure 2 illustrates the detailed steps in obtaining TN extrudates from the raw ingredients.

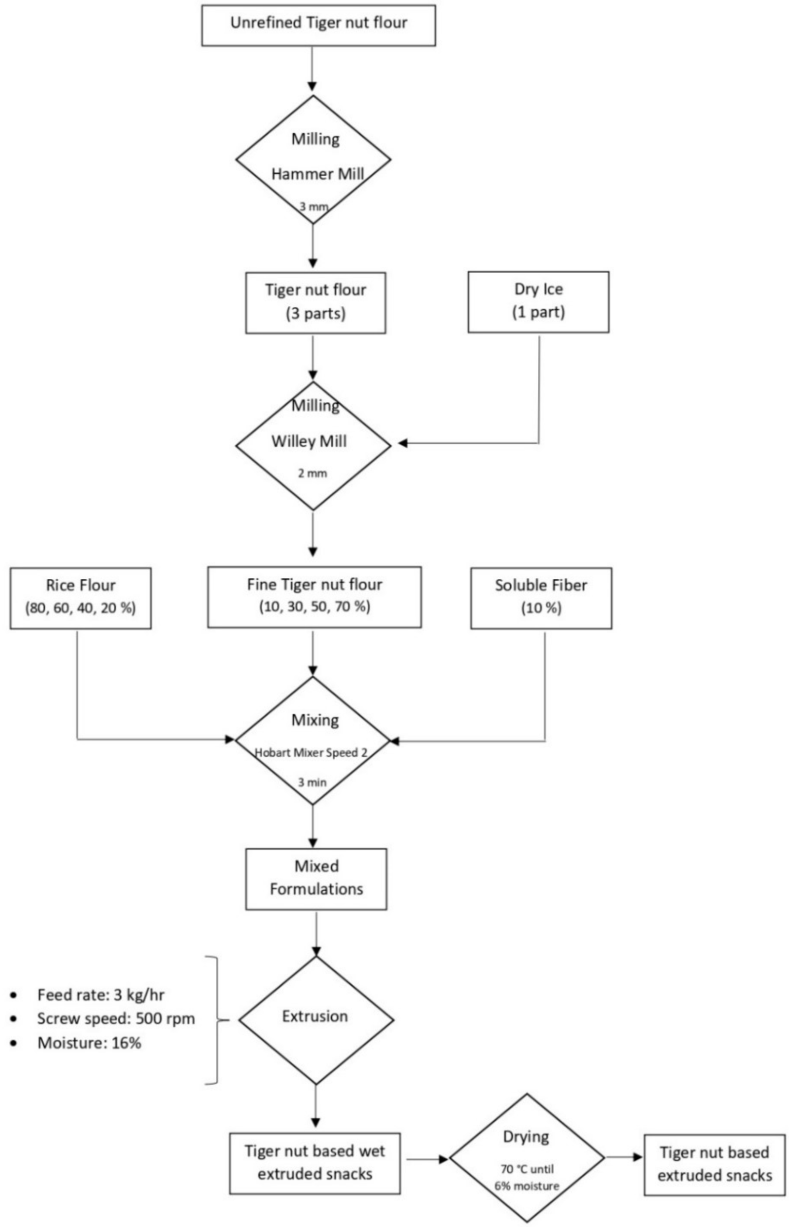

Figure 2: Flowchart of snacks production from tiger nut/rice flour mixture.

6.2.4 Proximate Analysis

Proximate composition of raw and extruded flours was determined in triplicate according to standard methods of the Association of Official Analytical Chemist AOAC [20]. Total nitrogen was determined by Leco FP628 (Leco Company, St. Joseph, MI, USA), and fat content determined on a Dionex ASE350 solvent extractor (Thermo Fisher Scientific, Inc., Waltham, MA, USA). Carbohydrate content was calculated by difference.

6.2.5 Apparent Viscosity

Apparent viscosity of the raw blends and extruded flours was determined using a Rapid Viscosity Analyzer (RVA 4500, Perten Instrument, Sydney, NSW, Australia). Samples of 3.50 g and 25.0 mL HPLC-grade water were mixed in an RVA canister and analyzed using the following temperature profiles: equilibrating at 25 °C for 1 min, then the temperature was ramp up to 95 °C over a period of 3 min, and holding at 95 °C for 4 min, followed by a cooling period of 3 min to 25 °C, and hold for 2 min. Thermocline software for Windows (TCW3) was used to calculate the pasting parameters: onset temperature where viscosity start to increase; peak viscosity (Pa·s): the maximum hot paste viscosity; trough viscosity (Pa·s): minimum hot paste viscosity; breakdown (Pa·s): the difference between peak viscosity and trough viscosity; final viscosity (Pa·s): the viscosity at the end of the run; and total setback (Pa·s): the difference between final viscosity and trough viscosity.

6.2.6 Microstructure Analysis

A scanning electron microscope (SEM) (Model TM300, Hitachi High-Technologies, Tokyo, Japan) at 2 kV accelerating voltage and at magnification of 100× was used for microstructural analysis. Extrudates were dried overnight in a desiccator with $CaSO_4$ prior to analysis. Colloidal graphite cement was applied around the bottom of the extrudates for a better conductivity and covered with approximately 30 nm of gold using a Technics Hummer V sputter coater. Stereoscopic pictures were taken using a stereoscopic microscope (Leica Microsystems Inc., Buffalo Grove, IL, USA.) at 1× magnification.

6.2.7 Final Products Quality Parameters

Expansion ratio (ER) of the extrudates was evaluated according to the method described by Berrios et al. [21] and calculated as followed:

$$ER_{(mm)} = \text{Area of extruded road}_{(mm^2)}/\text{Area of circular die hole}_{(mm^2)} \quad (1)$$

The bulk density (BD) of each of the extrudates was determined based on a volumetric displacement method, using glass beads with a diameter of 2 mm as a displacement medium, with some modifications as previously explained by Patil et al. [22], who standardized this measurement with optimum sample size. The values employed were obtained by averaging five measurements of the extrudates.

$$BD_{(g/cm^3)} = \text{Extrudate mass}_{(g)}/\text{Extrudate volume}_{(cm^3)} \quad (2)$$

True density of extrudates was measured with a pycnometer (AccuPyc II 1340, Micromeritics, Norcross, GA, USA) using a small sample holder with a volume of 350 cm^3. Helium was used as a volume displacement medium. To calculate sample volume and total pore volume, the pressure before and after expansion was measured. The analysis was carried out in triplicate for each sample.

A texture analysis of the extrudates was carried out using a texture analyzer (TA-XTPlus, Stable Micro System, Godalming, Surrey, UK) calibrated with a 2 kg mass, to determine the firmness and shear force of the samples by compression. A 3-point band test was used on individual extruded rods. A TA92A probe was set to press the extruded rod placed on a metal platform to a depth of 15%, with testing speed of 1 mm/s. The return distance and return speed were adjusted to 35 mm/s and 10 mm/s, respectively. A total of 20 measurements were performed for each sample. Firmness was defined as the peak force of the first compression required for the sample to rupture and the shear force was calculated as the area under the curve from the force/time graph.

6.2.8 Total Soluble Phenolics and Total Antioxidant Capacity of Final Products

Total soluble phenolics (TSP) were determined according to the Folin–Ciocalteu spectrophotometric method, with a slight modification to the method as described by Nayak et al. [23]. About 5 g of extruded flour samples were extracted with 20 mL of methanol at room temperature (25 °C) for 24 h. About 0.15 mL of Folin–Ciocalteu reagent was added to the extract (0.3 mL of aliquot). The mixture was set aside to equilibrate for 3 min and then mixed with 0.3 mL sodium carbonate. Subsequently, incubated at room temperature for 60 min, and absorbance of the mixture was read at 765 nm with a benchtop spectrophotometer (PharmaSpec UV-1700, Shimadzu Scientific Instruments, Inc., Kyoto, Japan). Methanol was used as a blank. TSP content was quantified from a gallic acid standard curve developed from 0–0.125 mg of gallic acid per mL and expressed as micrograms of gallic acid equivalent per milligrams of dry weight sample (μg GAE/mg DW). The analysis was carried out in triplicates.

Total antioxidant capacity (TAC) of extruded samples was measured using an adapted method of Patel et al. [24]. The methanol extracts from the TSP were used in this evaluation. A total of 0.5 mL of sample solution was reacted with 2.95 mL of 2,2-diphenyl-1-picrylhydrazyl (DPPH) (103.2 μM in methanol, absorbance of ~1.2 at 515 nm) on a covered shaker at room temperature for 20 h, and spectrophotometer was blanked with methanol. The absorbance was read at 515 nm. TAC was quantified from a Trolox standard curve developed from 0 to 750 μg of Trolox per mL and expressed as micrograms of Trolox equivalent per gram of dry weight sample (μg TE/g DW). The analysis was carried out in triplicates.

6.2.9 Statistical Analysis

Data were analyzed by multifactor analysis of variance (MANOVA) using Statgraphics Centurion XVII software (Statpoint Technologies, Warrenton, VA, USA). Fisher's least significant differences test was applied for the determination of significant differences among experimental mean values, with 95% confidence.

6.3 Results and Discussion

6.3.1 Raw Blends Characteristics

The moisture and carbohydrate content of raw blends decreased significantly ($p < 0.01$) whereas the protein, fat and ash contents increased (p < 0.01) as the TN content was increased (Table 1). Protein, fat and ash content significantly (p < 0.01) increased with the TN level substitution. In fact, this trend is attributed to a higher protein, fat and ash content of TN. The same trend was observed in the study by Kareem et al. [15], where the addition of TN (up to 100%) to a mixture of cassava and spices greatly enhanced the ash, protein, and fat content of the raw blends.

The Rapid Visco Analyzer (RVA) was used to visualize the physical transition points by recording the apparent viscosity of the formulated flours. Figure 3 depicts the viscoamylographs of the raw blends. Attributes determine from the recorded apparent viscosity are exhibited in Table 1, and the presence of TN was identified as main factor of variance. Statistical analysis indicated that TN incorporation in the formulations significantly (p < 0.01) increased the onset temperature for samples R3 and R4 (Table 1). The blend with the highest TN replacement level (R4) required more time to achieve the maximum peak viscosity (p < 0.01). This delay may be caused by the presence of more quantity of fiber and fat that could limit the water required for starch swelling. TN flour produced a significant (p < 0.01) reduction in the peak viscosity, trough viscosity, breakdown, final viscosity, and total setback. These outcomes were related with the lower starch content in the blends with TN flour that caused a viscosity drop in the mixture, which might constraint the further expansion of the extrudates. A similar trend was observed in a previous study by Adegunwa et al. [25] when TN was added to plantain flour.

Table 1: Proximate composition and apparent viscosity of raw blends containing increasing concentrations of tiger nut flour (R1: 10%; R2: 30%; R3: 50%; R4: 70%).

Parameters	Raw Blends				
	Control R	**R1**	**R2**	**R3**	**R4**
Proximate Composition					
Moisture (g/100 g)	11.76 ± 0.12 e	11.52 ± 0.06 d	10.66 ± 0.09 c	9.58 ± 0.08 b	8.72 ± 0.04 a
Protein (g/100 g)	5.02 ± 0.07 a	5.34 ± 0.08 b	5.71 ± 0.12 c	6.08 ± 0.10 d	6.30 ± 0.03 e
Fat (g/100 g)	0.28 ± 0.01 a	3.46 ± 0.08 b	9.06 ± 0.00 c	15.08 ± 0.06 d	20.45 ± 0.19 e
Ash (g/100 g)	0.36 ± 0.01 a	0.51 ± 0.00 b	0.80 ± 0.00 c	1.15 ± 0.03 d	1.46 ± 0.02 e
Carbohydrates (g/100 g)	82.58	79.17	73.77	68.11	63.07
Apparent Viscosity					
Onset temperature (°C)	70.3 ± 0.9 a	70.9 ± 0.8 a	71.9 ± 0.4 a	75.1 ± 1.3 b	78.2 ± 1.1 b
Peak Time (min)	5.7 ± 0.0 a	5.8 ± 0.1 a	5.8 ± 0.0 a	5.8 ± 0.1 a	7.1 ± 0.0 b
Peak viscosity (mPa·s)	3980 ± 70 e	3040 ± 60 d	1630 ± 20 c	710 ± 10 b	290 ± 0 a
Trough viscosity (mPa·s)	2130 ± 30 e	1750 ± 100 d	1080 ± 10 c	630 ± 0 b	280 ± 0 a
Breakdown (mPa·s)	1850 ± 40 e	1300 ± 40 d	550 ± 10 c	90 ± 10 b	10 ± 0 a
Final viscosity (mPa·s)	3140 ± 190 d	2900 ± 100 d	2100 ± 0 c	1400 ± 20 b	770 ± 10 a
Total Setback (mPa·s)	1010 ± 220 bc	1160 ± 0 bc	1030 ± 10 c	780 ± 10 b	480 ± 10 a

Means with different letters within the same parameter differ significantly ($p < 0.01$).

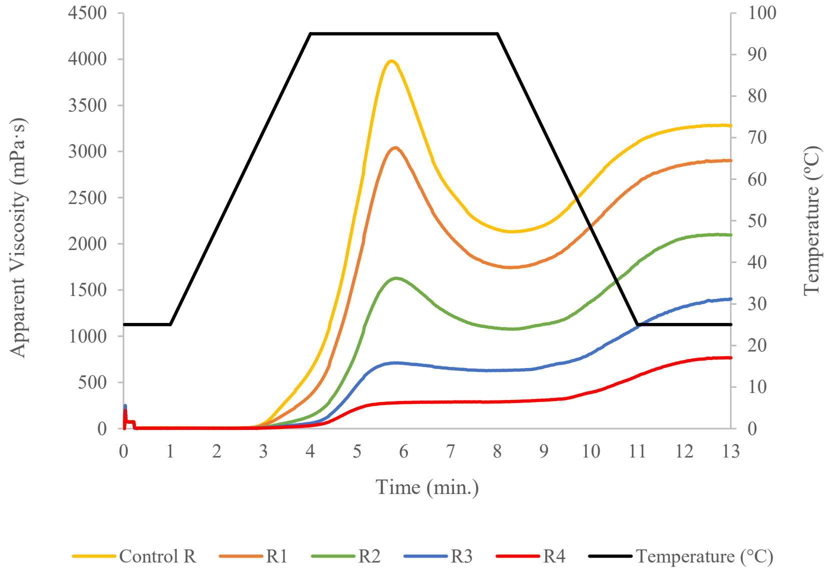

Figure 3: Rapid Visco Analyzer profiles of formulated unprocessed flours containing different concentration of tiger nut flour.

6.3.2 Extruded Products Characteristics

Expansion of the extrudates was significantly affected by the level of TN, as it can be observed in the surface and cross section images (Figure 4). The control rice extrudate showed a corrugated, cohesive overlapping surface that was more visible in the SEM micrograph, which is attributed to the gelatinization starch and expansion of the product during the extrusion process. The cross-section of the control sample (Figure 4, Control E), which had the greater expansion, depicted larger air pockets than samples containing TN. The extrudate containing 10% TN in the formulation (Figure 4, E1), presented a smoother surface with less obvious grooves, with a reduced diameter and expansion ratio; this is due to a content reduction of starchy rice as a result of high fat TN flour incorporation. With increasing TN concentration up to 30% in the formulations, the cohesiveness and continuity of the structural surface of the extrudates are disrupted and more uneven surfaces with porous structures were observed, particularly in SEM micrograph (Figure 4, E2). This disruption is more noticeable with the sample containing 50% TN (Figure 4, E3), which presents a coarser surface and particle disaggregation. A reduction in starch concentration as the TN was increased in the formulations mostly induced a breakage of the continuous matrix in the extrudates, rendering products with less expansion and surface uniformity. Rupture and non-uniform surface have also been reported as negative quality attributes determined on extruded cornstarch when adding wheat fiber [26]. The surface appearance of extrudates containing the highest content of TN of 70% (Figure 4, E4) displayed irregular areas with an alternating smooth, oily-looking appearance. The high fat content in this sample could have acted as a plasticizer, which was observed in the SEM image, resulting in a product with the indicated distinctive surface characteristics. This result corroborates with the compact, unexpanded cross-sectional view of this extrudate (Figure 4, E4), confirming the significant effect of the inclusion of the TN flour in the structural and surface characteristics of the developed snacks. Apart from that, the compactness above described might be also linked to the higher protein and fiber content. During extrusion, protein and fiber could behave as a dispersed phase within the continuous starch arrangement that causes an interruption of the cell wall formation [27].

Furthermore, the covalent and nonbonding interactions between protein leads to the formation of a network. This protein system may influence the water distribution in the matrix causing changes of the extensional properties of the melt with a consequent density increase of the final extrudates [28].

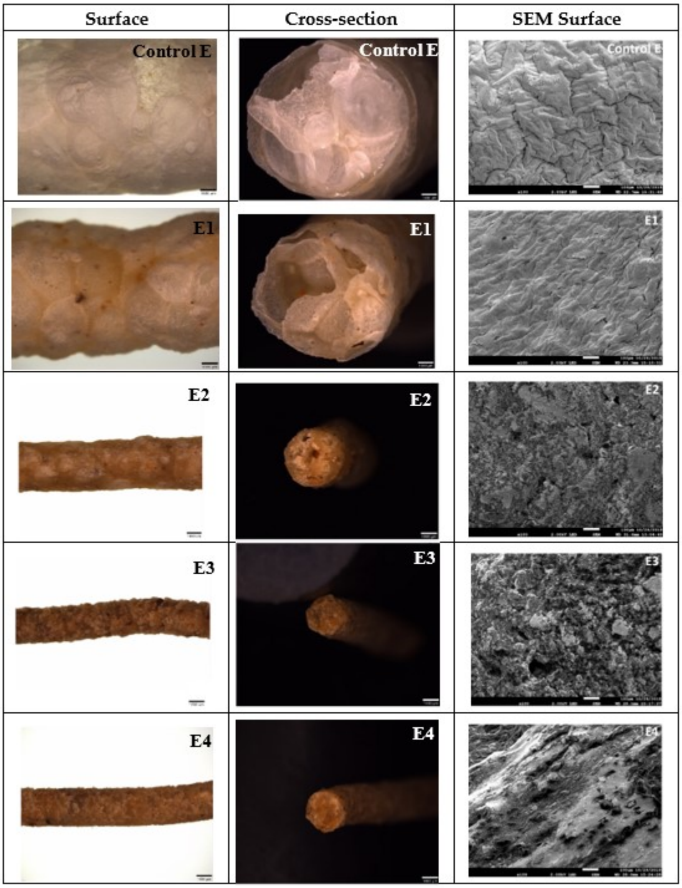

Figure 4: Stereoscopic images of surface and cross-sectional areas (1× magnification) and SEM micrographs (100× magnification) of the surface area of extruded samples containing different levels of tiger nut.

Figure 5 depicts profiles of extrudates characterized by lower viscosities than those obtained for the raw blends. Indeed, heating and mechanical shearing applied during the extrusion process caused starch gelatinization. It is known that heating and mechanical shearing cause the fragmentation of amylopectin and amylose structure, thereby reducing their absorbing and swelling capacity in starchy materials [29], which was reflected in a viscosity reduction found in this study. Moreover, it was determined that the apparent viscosity was extremely low in the control and E1 extrudate containing 10% TN, than extrudates E2 and E3 containing 30% and 50% TN, respectively (Figure 5). This could be due to the "protective" effect of fat on the starch granules [30], that kept some ungelatinized granules which contribute to increase the apparent viscosity. Another explanation may be related with the starch-protein interactions that limit the starch swelling and the changes associated to its gelatinization [31]. In this case, protein absorb more water, making it less available for the complete starch gelatinization. Viscosity of extrudate E4 with 70% TN did not follow the indicated pattern, because of the greater substitution, most of or all of the starch got gelatinized and dextrinized during the extrusion process. As shown in Table 2, TN flour addition significantly ($p < 0.01$) increased the onset temperature. In this regard, formulation E4 with the highest amount of TN presented the highest onset temperature. The delay in the peak viscosity caused by TN incorporation, observed in the raw blends, was also observed when analyzing the extruded samples, while peak viscosity, trough viscosity, final viscosity and total setback showed a completely different pattern. In general, except for extrudate sample E4, the content of TN in the extruded products corresponded with a significant ($p < 0.01$) increase in the apparent viscosity, despite the reduced starch content. This may be due to the protective effect exercised by the fat molecules that could arrange themselves around the starch granules mitigating the degradation effects of the heating and mechanical shearing.

Table 2: Main physical and nutritional characteristics of extruded samples made from formulations with increasing concentrations of tiger nut flour (E1: 10%; E2: 30%; E3: 50%; E4: 70%).

Parameters	Extruded Samples				
	Control E	E1	E2	E3	E4
Apparent Viscosity					
Onset temperature (°C)	50.5 ± 0.0 a	50.4 ± 0.1 a	70.4 ± 0.5 b	79.9 ± 2.5 c	87.2 ± 0.6 d
Peak Time (min)	2.2 ± 0.0 a	4.2 ± 0.2 b	6.5 ± 0.2 c	6.2 ± 0.0 c	7.15 ± 0.1 d
Peak viscosity (mPa·s)	100 ± 10 a	150 ± 0 b	300 ± 0 d	510 ± 10 e	210 ± 0 c
Trough viscosity (mPa·s)	50 ± 0 a	100 ± 0 b	290 ± 0 d	480 ± 10 e	210 ± 0 c
Breakdown (mPa·s)	50 ± 0 e	40 ± 0 d	10 ± 0 b	20 ± 0 c	0 ± 0 a
Final viscosity (mPa·s)	80 ± 0 a	170 ± 0 b	680 ± 0 d	1160 ± 10 e	580 ± 0 c
Total Setback (mPa·s)	30 ± 0 a	70 ± 0 b	390 ± 0 d	670 ± 0 e	370 ± 0 c
Quality Parameters					
Diameter (mm)	10.19 ± 0.28 d	9.27 ± 0.27 c	3.46 ± 0.12 b	2.53 ± 0.03 a	2.51 ± 0.03 a
Expansion ratio	16.63 ± 0.92 d	13.78 ± 0.81 c	1.92 ± 0.14 b	1.02 ± 0.02 a	1.01 ± 0.02 a
Bulk density (g/cm^3)	0.19 ± 0.02 a	0.21 ± 0.01 a	0.62 ± 0.01 b	0.69 ± 0.02 c	0.65 ± 0.02 b
True density (g/cm^3)	1.52 ± 0.01 d	1.54 ± 0.01 e	1.50 ± 0.01 c	1.44 ± 0.01 b	1.35 ± 0.00 a
Total Pore volume (cm^3/g)	0.34 ± 0.01 d	0.35 ± 0.00 e	0.33 ± 0.01 c	0.30 ± 0.01 b	0.26 ± 0.00 a
Firmness (g)	712 ± 146 c	922 ± 261 d	242 ± 52 a	367 ± 39 b	313 ± 73 ab
Work of Shear (g·s)	11 ± 3 b	19 ± 6 c	2 ± 1 a	4 ± 1 a	4 ± 1 a
Proximate Composition					
Moisture (g/100 g)	6.67 ± 0.07 b	6.12 ± 0.04 a	6.14 ± 0.06 a	6.20 ± 0.05 a	6.24 ± 0.02 a
Protein (g/100 g)	4.65 ± 0.13 a	4.87 ± 0.07 b	5.25 ± 0.09 c	5.57 ± 0.06 d	5.83 ± 0.04 e
Fat (g/100 g)	0.05 ± 0.01 a	1.47 ± 0.01 b	6.62 ± 0.02 c	14.11 ± 0.04 d	19.32 ± 0.14 e
Ash (g/100 g)	0.36 ± 0.00 a	0.67 ± 0.02 b	0.93 ± 0.01 c	1.22 ± 0.02 d	1.52 ± 0.01 e
Carbohydrates (g/100 g)	88.26	86.87	81.07	72.90	67.09
Total Soluble Phenolics Gallic acid Equivalent (μg/mg)	10 ± 0.00 a	110 ± 0.00 b	240 ± 0.01 c	550 ± 0.02 d	720 ± 0.03 e
Antioxidant Capacity Trolox Equivalent (μg/g)	533 ± 17 a	596 ± 15 b	730 ± 10 c	852 ± 10 d	937 ± 9 e

Means with different letters within the same parameter differ significantly ($p < 0.01$).

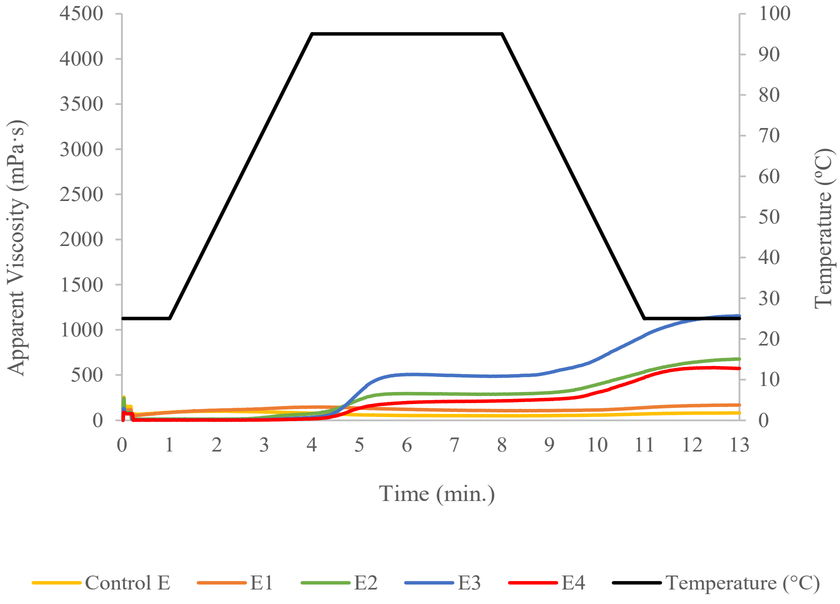

Figure 5: Rapid Visco Analyzer profiles of formulated extruded samples containing different levels of tiger nut flour.

The data in Table 2 indicate that the addition of TN in the formulations produced significant ($p < 0.01$) changes in the developed extrudates. The diameters, expansion ratios, true densities, and total pore volumes progressively decreased ($p < 0.01$) as a consequence of the increased content of TN in the samples. Formulations E3 and E4 recorded the lowest values in terms of diameter and expansion ratio, as shown in Figure 4 and Table 2. The reduction in expansion with the progressively higher inclusion of TN in the samples might be attributed to a reduction in the starch content and an increase in the insoluble fiber [18], fat and protein, which are known as factors that negatively correlate with expansion [32]. In literature, studies agree with the results presented in this work, the expansion ratio decreased when TN flour was blended with rice flour [15] and also when lentil [33], mango peel [34], and partially defatted hazelnut flour, high in fiber and protein [35] were used in combination with corn flour and rice grits, respectively. Berrios et al. [21] have previously indicated that bulk density was inversely related to expansion ratio, which agrees with the results from this study. Overall, with the increase of the TN flour in the formulation a rise of the bulk density of the extrudates was observed. In particular, between the control and sample E1 there was a slight increase that was even more pronounced between the samples E2 and E4, most likely due to the excessive fat content in those samples containing more TN in their formulation. A similar trend in BD was observed by previous researchers in extrudates made with corn and soybean hull [36]. The determination of true density and porosity of the extrudates provided additional quality parameters on the properties of the developed products, as these parameters provide insight into the structural properties of the dried materials. Addition of TN significantly reduced ($p < 0.01$) the true density of the extrudates. A similar trend was observed for the total pore volume of extrudates with higher inclusion of TN in their formulation (Table 2). During extrusion, as the melt exit the die, numerous small air cells are generated by the rapid release of the high pressure [26]. The pressure difference out of the die causes the water flash off with the formation of internal pores of varying sizes that are responsible for the product expansion. The high fat content in samples with the greatest inclusion of TN in their formulation, decreased the melt viscosity of the extruded material, which caused a reduction in die pressure, resulting in less expanded products with higher bulk densities (Table 2) [37]. This phenomenon is clearly shown in Figure 4. Extrudate control E had larger diameter and more air pockets, while sample E4, with the highest amount of TN, showed a cross-section devoid of air pockets or porous structure. Internal microstructure of the extrudates are of important consideration in the production of snack-type products as this structural pore formation is an important quality parameter associated with the crispiness and crunchiness of expanded products [37].

Texture was evaluated by measuring the firmness (force required to break the extruded rod) and work of shear (area under the curve). The results in Table 2 show significant effect ($p < 0.01$) of TN on the texture of the developed extrudates. The sample with 10% of TN presented the highest firmness and shear force values. This textural effect may be due to a higher integration of the matrix components and the amylose-lipid complex formation. In fact, the interaction of fatty acids or long-chain alcohols with amylose double helices forms the amylose-lipid complexes that cause functional modifications in the physical and chemical behavior of the starch [38]. Given that, the interactions between amylose and fatty acids increase the elasticity of the starch matrix, the structure resulted to be more resistant to breakdown [39]. Conversely, both texture parameters were reduced with an increase of TN in the formulations. This could be explained by the reduction of starch content as the addition of TN increased in the formulations, while fat content greatly increased resulting in extrudates with softer and more brittle texture. This same trend was also reported in a previous study on extrudates containing different levels of cassava-TN mixtures [15]. Moreover, the outcomes about the maximum force value from this study (from 242 g to 922 g) are in the same range of those reported by Kareem et al. [15] (from 251 g to 1272 g).

The proximate analysis of the raw blends determined that the TN addition significantly ($p < 0.01$) increased the protein, fat and ash content compared to the control sample with no TN addition. It was also observed that extrusion process reduced the protein and fat amount in the extruded snacks. Protein losses ranged from 11% to 16% but those were much greater for the fat content. The control suffered the greatest fat loss followed by sample E1 (150%). This reduction of fat most likely occurred at the die opening as free oils [40]. As melt exits the die, a rapid temperature and pressure drop occur, resulting in rapid expansion of the water molecules into steam, and in this research, a small quantity of liquified fat was observed at the die, especially for the samples E4 and E5. Another possible explanation of the fat reduction could be the formation of new complexes with amylose or protein that trap the lipid, making their extraction more difficult with conventional method [40]. After extrusion, all the samples, (except the control) presented a slightly higher ash content with respect to their raw counterparts. From a nutritional standpoint, it is well known that GF foodstuffs are poorer in minerals and protein content, while their saturated fat amount result to be higher compared to their gluten-containing counterparts [41]. TN included in GF free snacks production may represent a valuable alternative when it comes to cover these nutritional deficiencies. Moreover, the lipidic profile mostly characterized by polyunsaturated fatty acids [42], may help to reduce the quantity of the saturated ones. Although, its high fat content imposes a correct evaluation when deciding to employ TN in GF food product development.

3.3 Total Soluble Phenolics and Total Antioxidant Capacity of Extruded Snacks

The total soluble phenolic content and the antioxidant capacity were evaluated to assess the nutritional improvement provided by TN (Table 2). The control sample showed low amount of total soluble phenolics, but it had antioxidant activity, which might be related to the reaction of some peptides or amino acids with the DPPH. There was a significant increase in total soluble phenolic and antioxidant capacity when rice flour was replaced with TN in extruded samples. The TSP in the extrudates varied between 10 to 720 µg GAE/mg DW. Adebowale et al. [43] using a cassava-based formulation containing increasing TN, reported a slightly higher TSP (370 to 890 µg GAE/mg DW values), which might be due to the presence of different spices (onion, ginger, chili pepper) in the formulations. TAC results (Table 2) reflected similar trend as that of TSP. The amount of TAC significantly ($p < 0.01$) increased with addition of TN in the extruded products. Those increases may be related with the presence, in the tiger nut cell wall, of some antioxidant monomeric phenols, such as p-hydroxybenzoic acid, vanillic acid, p-hydroxybenzaldehyde, vanillin, p-trans-coumaric acid, trans-ferulic acid, p-cis-coumaric acid, cis-ferulic acid that may contribute to improve the antioxidant capacity [42]. Another possible explanation may come from the presence of tocopherols in TN that have been described as the most important natural group of antioxidants found in vegetable oils [44].

6.4 Conclusions

This study presents the potential use of TN to produce novel gluten-free extruded snacks, providing an attractive alternative to consumers with celiac disease. Pasting profile analysis showed that TN inclusion, into the rice-based formulated flours, increased the onset temperature and delayed the peak viscosity while in the extruded flours. Progressive addition of TN in the formulations promoted a reduction in diameter, expansion ratio, true density, and total pore volume in the extrudates, while their bulk densities increased. Furthermore, TN incorporation was responsible for an increase in ash, protein, and total phenol content, which is an added value to the developed snack. This study demonstrated that extrudates with 10% TN in the formulation showed the best overall texture profile. A future study is proposed using specialty starches to further promote expansion of the TN-based snacks.

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7. General Discussion

To create products of various shape, volumes and consistency, cereal food industry has universally resorted to extrusion technology. The principal benefits of this technology are represented by its adaptability, energy efficiency and low-cost production [1]. Through a series of steps (mixing, kneading, cooking, forming) extrusion leads a controlled combination of materials and energy into a food giving rise to a reorganizing and plasticizing operations [2]. Throughout this thermomechanical process, the main physicochemical modifications are to be borne by starch and protein. Indeed, extrusion promote starch gelatinization and protein denaturation [3]. Due to this, cereal and others vegetable flours are transformed in added-value products such as pasta, noodles and snacks. Contrary to the conventional pasta making process, where gluten role is of prime importance, pasta GF production is completely based on the gelatinization and retrogradation starch capability [4].

Despite the many studies carried out with GF pasta and the use of different raw material for nutritionally enriching of those products, there are very few information about the quality of the marketed GF pasta foods. Because of that, a selection of GF spaghetti from eight different European brands was analyzed and compared with an analog wheat one in terms of cooking performances and nutritional value (before and after cooking). During GF pasta making, specifically during drying at high temperatures, undesirable compounds like furosine could be produced, indicative of very rapid drying process and in turn low quality products [5]. Maize and rice flours were predominant in all the formulations as confirmed in the study conducted by Morreale et al. [6], while mono and diglycerides of fatty acids were used in 3 samples. Other minor ingredients were quinoa, brown rice, and millet flour. Comparison with their wheat-based homologues showed a deficiency in protein and ash content in most of dry and cooked GF samples. This type of formulation led to greater nutrient losses during cooking compared to wheat pasta. This lack of components retention was caused by the gluten absence which is responsible for the solids entrapping [7]. Therefore, GF pasta had a no cohesive structure, that resulted improved in samples containing emulsifiers; this evidence was clear also in the study published by Lai [8] where the use of the emulsifiers provided a better cooking behavior and less surface stickiness in rice pasta. The texture was greatly influenced by the flour source, and only maize gave higher resilience, elasticity, and firmness. Technological strategies to simulate the gluten network and strong drying process may cause a heat damage with the development of some harmful compounds like furosine [9]. Until now, no information has been reported about the heat damage in GF pasta before and after cooking. GF pasta had lower furosine content than the wheat-based control and the cooking process seemed to increase their levels in all the tested samples.

Among the products manufactured by the extrusion technology and considering their increasing consumption, noodles were selected as a potential food matrix to be made with TN. Indeed, the flexibility of their process production, beside rice and corn, has allowed the incorporation of other ingredients as pseudocereals and legumes to improve the nutritional quality [10] [11]. Additionally, alternative flours with functional qualities were employed but their role in the process is still in needed of more clarifications [12]. TN has moderate protein content, high fiber content and an interesting lipid profile, that explained the interest in this sweet tuber for human consumption. Studies about its use in food industry have been carried out allowing the development of fresh pasta [13] [14] and biscuits [15], while the *horchata* by product has been utilized for bread fiber enrichment [16] and for meat products enhancing [17] [18]. Very few studies had as object the use of TN in extrusion-cooking technology. Given the absence of gluten in TN, it was necessary to identify a structuring agent that helped in reaching the right noodles consistency. Hydrocolloids in GF design products are principally utilized because of their behaviors as thickeners, emulsifiers, stabilizers, foaming agents, improvers of water retention and texture [19]. Authors reported rice noodles with improved cooking performances and texture following the introduction in their formulations of some hydrocolloids such as carboxymethyl cellulose, inulin, xanthan, and guar gum [20] [21]. Anyway, in most cases these products are usually empirically made by mixing gluten free ingredients with no knowledge about their role in the food matrix. For this purpose and to provide a certain extensibility, a selection of different hydrocolloids (guar gum, xanthan gum, inulin and carboxymethyl cellulose) was used for making GF noodles based on TN. Due to their different water binding capacity, the effects of hydrocolloids on the hydration in GF noodles making, certainly deserves to be deeply investigated since that water has a crucial role in defining noodles dough consistency. For this reason, the evaluation of two distinct hydration levels, constant usually employed in literature and adapted to the food matrix requests, allowed to understand the technological differences found among the different sample sets. Due to the diverse capability of the hydrocolloids to bind water, doughs showed distinct thermomechanical behaviors. When the level of the water was kept constant, samples with hydrocolloids had higher consistency. This is explained by their great skill in water binding. In fact, after the water adjustment the differences were abated. Xanthan gum and guar gum were the hydrocolloids that required higher water amount to correct the dough consistency. As a result, doughs with different rheology generated fresh and cooked noodles that differed in terms of textural properties. Hydrocolloids were responsible for the diameter increase, hardness and firmness improving, while acting as a structure improver, they allowed the reduction of solids release into cooking water. Therefore, all told, TN noodles made with 0.5% of xanthan gum and optimized hydration level presented better cooking performances and final texture attributes.

To expand the application of TN to other GF products, the focus was shifted to the snacks. Currently, they represent a food-stuffs segment which is constantly increasing because is deeply associated with the costumers' comportments. Due to this, a thorough examination of the cereals-based snacks originally from the Mediterranean area was carried out. Through this part of the study a better understanding of the snacks processing, their ingredients interactions, their quality attributes, and safety problems was given with a particular attention to the extrusion-cooking process.

Lately, new alternative flours are processed through the extrusion technology for the development of new GF expanded snacks. In fact, when kale leaves powder (up to 20%) was incorporated into corn-based snacks, an expansion ratio reduction and a deep color change were observed [22]. Black elderberry, chokeberry and strawberry powder were utilized from 5 to 20% in directly expanded corn snacks [23]. High amount of fruit powder substitution, leaded an expansion ratio reduction, a bulk density increasing and a higher firmness value. Extrusion process caused a drop of phenolic compounds content in GF extruded snacks composed by rice (50-80%), beans (20-40%) and carob fruit (5-10%) [24]. In the case of TN, designed formulations included different levels of TN as a non-conventional ingredient, and rice and soluble fiber to improve the expansion ratio. Decrease of peak viscosity, trough viscosity and total setback occurred as a result of the extrusion-cooking that provided a starch gelatinization. Extruded samples with TN had smaller diameter, expansion ratio, true density, and total pore volume. The analysis of the snack surface structure showed that in general, TN contributed to make the surface smoother and less uniform. TN impact was observed on the textural characteristics of the extruded samples; indeed, as TN increased, starch content diminished, and a reduction of the firmness and shear force was found. This section of the study confirms the suitability of TN (10%) for GF snacks development as a new alternative for the celiacs.

Under a nutritional standpoint, TN incorporation was responsible for the enhancement of ash, protein, and fat content with respect to the rice control. Total soluble phenolics content and total antioxidant capacity increased with the TN introduction while the high pressure, force and temperature reached during extrusion, caused their drop.

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8. Conclusions

Research conducted through the different chapters allows concluding that TN flour could be used as an alternative ingredient in the design of GF noodles and extruded products, after defining appropriate structuring agents and extrusion settings, respectively.

Particularly, the following concluding remarks can be highlighted:

- There were significant differences in the nutritional composition between the gluten free spaghetti and the durum wheat pasta. In general, gluten free samples in both stage (dried and cooked) were poorer in protein and ash with respect to the wheat pasta. Regarding quality indicators, GF samples showed significantly different behavior, and they could not be related to specific ingredients. Nevertheless, pasta made with corn flour required longer cooking and had high cooking loss values but resulted in more resilient and elastic pasta. The furosine content in dried GF pasta was majorly lower than that in wheat pasta, and those differences were greatly magnified after cooking.

- Gluten free noodles could be made from TN flour, giving special attention to the amount of water used in the production process and the type of hydrocolloid added, because they play a crucial role on the dough rheology and the quality of fresh and cooked noodles. Overall, gluten free TN noodles made with xanthan gum and adapted amount of water showed the best quality considering the lowest cooking losses obtained and its higher firmness.

- Snacks market segment represented a good opportunity to launch innovative products with enhanced nutritional quality and food safety. Costumer's behaviors and the introduction of new food technologies represent a very good asset for succeeding in the snacks segment. Particularly, the inclusion of new ingredients leading to novel products with appealing textures and sensory perceptions.

- TN flour could be used to produce novel gluten-free extruded snacks, providing an attractive alternative to consumers with celiac disease. Pasting profile analysis showed that TN inclusion, into the rice-based formulated flours, increased the onset temperature and delayed the peak viscosity while in the extruded flours. Progressive addition of TN in the formulations promoted a reduction in diameter, expansion ratio, true density, and total pore volume in the extrudates, while their bulk densities increased. Furthermore, TN incorporation was responsible for an increase in ash, protein, and total phenol content, which is an added value to the developed snack. The extrudates with 10% TN in the formulation showed the best overall texture profile.

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ORIGINAL PAPER

Quality Indicators and Heat Damage of Dried and Cooked Gluten Free Spaghetti

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Abstract
The quality and safety indicators of commercial dried gluten free (GF) pasta were analyzed to investigate, for the first time, the real nutritional intake through the chemical composition and the heat damage during processing by quantification of furosine. Eight samples of GF spaghetti were compared with wheat spaghetti. Dried and cooked GF pasta had lower protein and ash content than wheat spaghetti. GF samples composed solely by corn flour had higher optimal cooking time. Samples with emulsifier showed lower losses during cooking. Considering their composition, no trend could be established to explain textural behavior. Samples constituted merely by corn showed the highest resilience and elasticity. Spaghetti constituted only from corn and rice showed the highest firmness. The furosine content in dried samples ranged between 19 and 134 mg FUR/100 g proteins and in cooked samples ranged between 48 to 360 mg FUR/100 g proteins. Furosine content of GF pasta was in general lower than in wheat pasta, and those differences were even enlarged when comparing them after cooking. The results of PCA indicated it was possible to discriminate GF pasta regarding their technological and nutritional behavior.

Keywords Gluten free pasta · Cooking quality · Heat damage · Furosine

Introduction

Consumption of gluten free (GF) products has become trendy because of growing number of diagnosed celiac patients and the adoption of gluten free diets not related to pathologies. Among the non-gluten foods, pasta is one of the most consumed due to its convenience, palatability and long shelf-life [1]. Although gluten pasta could be considered a simple processed food, it gets more complex when going to GF pasta. A good quality pasta is characterized by adequate performance during cooking, which depends on the raw materials, particularly the quantity and quality of proteins and starch that give rise to the formation of a gluten network that traps the starch granules [2]. In the case of GF pasta, to develop a pseudo gluten network, the industry uses different ingredients (flour/starch from corn, rice, tapioca, pseudo cereals and legumes) and may add emulsifiers, hydrocolloids or adopt nonconventional pasta-making processes [1]. Consequently, market offers a great range of GF pasta, which in opposition to what happens with gluten pasta, shows great variability. Nevertheless, the integration of the different ingredients might lead to complete different pseudo gluten network with rather diverse performance during cooking, and in turn, pasta quality characteristics. Similarly, scarce attention has been paid to their nutritional quality. Previous studies have pointed out the deficient nutritional pattern of GF breads [3, 4], but information regarding the GF pasta is almost non-existent.

The diverse composition of GF pasta might respond different to process, particularly to pasta drying, which is a decisive stage for ensuring its microbiological stability by keeping final water content lower than 12.5%. The high temperature-short time system (> 75 to 100 °C for 2 to 3 h) usually applied for pasta drying allows producing gluten pasta with excellent cooking properties [5], but with a concomitant increasing risk of occurring Maillard reaction (MR) and the release of melanoidins as final products [6]. In early stages of MR, Amadori compounds (fructosyl–lysine, lactulosyl–lysine, and maltulosyl–lysine) are formed, which generate furosine (FUR) (ε-N-furoylmethyl-L-lysine) that has been established as a good marker of heat damage during pasta processing, as well as an indicator for the nutritional value and safety of the pasta products. Moreover, in the last years, some studies have

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stressed the toxic consequences of FUR on the biological system, specifically its adverse effects on mice liver and kidney [7].

The aim of this study was to analyze the quality indicators of commercial gluten free pasta (cooking behavior, color, texture properties), to investigate for the first time the real nutritional intake through the chemical composition of commercial cooked GF pasta and to evaluate the heat damage by quantification of FUR in dried and cooked GF pasta.

Materials and Methods

A total of eight commercial GF spaghetti samples, manufactured by different European companies, were purchased in the market. Samples were labeled with different codes for identification, GF1, GF2, GF3, GF4, GF5, GF6, GF7, and GF8, respectively. A wheat pasta was also included as a reference and labeled as W. Information contained in the package was recorded (Table 1). Chemical reagents and solvents of analytical grade were purchased to Sigma–Aldrich (Madrid, Spain). Standard of furosine was from Extrasynthese (Genay, France). Hydrochloric acid (37%) and methanol were purchased from Merck (Darmastadt, Germany). Ultra-pure water was obtained using a Mili-Q water purification system (Millipore, USA).

Cooking Quality Indicators

Optimal cooking time (OCT) was evaluated according to AACC official method [8] with some modifications. Spaghetti (5 g) were cooked in 60 mL of boiling water and every 30 s spaghettis were squeezed between two Petri dishes to determine the OCT that corresponded to the disappearance of the white core (ungelatinized starch). For each sample, two measurements were performed.

Cooking loss (CL), the organic material lost during cooking, was analyzed. For that, the cooking water was dried to constant weight in an air oven at 65 °C. The remnant (solid loss) was weighted and expressed as percentage following Eq. 1. The mean value was a result of three replicates.

$$\mathrm{CL}\ (\%) = \frac{\text{Solid loss (g)}}{\text{Raw Pasta (g)}} \times 100 \qquad (1)$$

Water absorption (WA) was determined weighing five pieces of cooked spaghetti (5 cm long) and following the Eq. 2 as reported by Tudorică et al. [9]:

$$\mathrm{WA}\ (\%) = \frac{\text{Cooked (g)} - \text{Raw Pasta (g)}}{\text{Raw Pasta (g)}} \times 100 \qquad (2)$$

Three measurements were performed for each analysis, and the mean values were calculated.

Swelling index (SI) was determined following the same steps of WA, five pieces of cooked spaghettis (5 cm long) were weighted and dried during 24 h to constant weight in air oven at 65 °C. SI was determined following the Eq. 3:

$$\mathrm{SI}\ (\text{g water/g dry pasta}) = \frac{\text{Cooked Pasta (g)} - \text{Cooked Pasta after drying (g)}}{\text{Cooked Pasta after drying (g)}} \qquad (3)$$

Each sample was analyzed in triplicate and mean value was noted.

Color of dried and cooked spaghetti was measured by a Minolta Chromameter (Model CR-400, Minolta Co., Osaka, Japan) using CIELAB coordinates. L^* for the lightness (0–100), a^* for the green – red (negative values relate to green while the positive ones refer to red) and b^* for blue – yellow b^* (negative values relate to blue while the positive ones refer

Table 1 Ingredients in commercial gluten free pasta as indicated in the label

Code	Ingredients
GF1	White Corn Flour, Yellow Corn Flour, Rice Flour, Water, Emulsifier: E-471 Mono and Diglycerides of fatty acids
GF2	Corn Flour, Millet Flour, Rice Flour, Cane sugar syrup
GF3	Corn Flour, Rice Flour, Corn Starch, Quinoa Flour, Emulsifier: E-471 Mono and Diglycerides of fatty acids
GF4	Corn Flour
GF5	Corn Flour
GF6	Corn Flour, Rice Flour, Brown Rice Flour, Quinoa Flour, Emulsifier: E-471 Mono and Diglycerides of fatty acids
GF7	Corn Flour, Rice Flour
GF8	Corn Flour, Rice Flour, Emulsifier: E-471 Mono and Diglycerides of fatty acids
W	Durum Wheat Semolina

to yellow). From these parameters, the cylindrical coordinates hue or hue angle (h_{ab}) and Chroma (C^*_{ab}) were obtained following the Eqs. 4 and 5 and the mean value came from five measurements.

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

$$h_{ab} = \tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (5)$$

Pasta Characterization

Dried and cooked spaghetti samples were analyzed for moisture content, crude protein by Dumas combustion principle using 6.25 as conversion factor, total fat and ash following the ICC standard methods [10]. Total carbohydrates data were obtained by subtraction from 100 the moisture, protein, fat and ash values. Results are the average of two determinations and are expressed as g/100 g.

Using the texture analyzer (TAHDi/500, TAHD Co., Stable Micro System, Surrey, UK) the texture profile analysis (TPA) was studied. The instrument was equipped with a load cell of 30 kg and P/36 cylinder probe (36 mm Ø) and the following settings were used: pre-test, test and post-test speed was 2 mm/s, and trigger force was 10 g. The distance was 10 mm with 75% strain and holding time between compressions was 2 s. Five pieces (5 cm long) of cooked spaghetti were submitted to a double compression and hardness, adhesiveness, chewiness and resilience were measured using five replicates; data were calculated using the software of the instrument. Following the official method of the AACC International [8], firmness (g) (the maximum force necessary to cut spaghetti) and work of shear (g·s) (the area under the curve of force/time graphic) were investigated using a texture analyzer (TAHDi/500, TAHD Co., Stable Micro System, Surrey, UK) fitted with a plastic cutting tool. Each sample was analyzed in triplicate and data were calculated by software instrument. Elastic limit (g) and elasticity (mm) of spaghetti were determined by a texture analyzer equipped with a load cell of 30 kg and with tensile grips (ref. A/SPR, Stable Micro Systems), following certain conditions: test speed was 3 mm/s and initial distance was 100 mm. Five measurements were performed for each sample and data calculated by the instrument software.

Furosine Determination

Furosine was determined in dried and cooked spaghettis. Previously to furosine determination, cooked samples were lyophilized for 24 h at −70 °C and 933.26 Pa with a Virtis SP Scientific equipment (Pennsylvania, USA). Samples were hydrolyzed with 8 N HCl at 110 °C for 24 h. One milliliter of the hydrolysate was filtered in a 0.22 μm cellulose filter. Chromatographic determination of FUR in pasta samples was performed on a HPLC instrument (HP Series 1050, USA) with a DAD detector (1040A) following the method of Li et al. [11]. The separation of FUR was accomplished on a C18 column (4.6 × 150 mm, 5 μm, Hewlett-Packard) and two mobile phases consisting of 0.1% trifluoroacetic acid (TFA) in water (A) and 0.1% TFA in acetonitrile (B). A gradient elution, 1–21% B at 0–25 min and 21–1% B at 25–30 min, was applied. A cleaning process with 1% tetrahydrofuran (THF) during 5 min was performed after each sample analysis. The flow rate was maintained at 1 mL/min. The identification of FUR was carried out by DAD spectra and the quantification was performed by an external standard method. The content of FUR was expressed as mg of compound in 100 mg of proteins. The analyses of each sample were carried out in duplicates.

Statistical Analysis

Data were analyzed by one-way analysis of variance (ANOVA) and multiple sample comparison, which was performed by using Statgraphics Centurion XVII (Statpoint Technologies, Warrenton, USA). Fisher's least significant differences (LSD) test was used to describe means with 95% confidence. Principal component analysis (PCA) was also performed to significantly differentiate ($P < 0.05$) among samples.

Results and Discussion

Chemical Composition

Most of the commercial samples of GF spaghetti analyzed in this study contained a mixture of corn and rice flours in their formulations; only two GF samples were composed only by corn, as shown in Table 1. Quinoa flour was present in two of the samples and other ingredients included brown rice and millet flour. Despite their coincidence in ingredients, there was significant difference in their nutritional composition (Table 2). Dried and cooked GF pasta had lower protein and ash content than wheat pasta, except for GF2, likely due to the presence of millet in the formulation as it has higher mineral content than corn or rice [12]. Conversely, dried GF pasta contained higher amount of fats than wheat pasta, excepting GF3, but those differences were greatly reduced in the cooked pasta. Differences among dried and cooked pasta confirms that cooking promoted significant loses of nutrients. When

Table 2 Proximate composition (d.b.), furosine content (d.b.) and color of the pasta samples before and after cooking

Sample	Moisture (%)	Ash (%)	Protein (%)	Fat (%)	Carbohydrates (%)	Furosine (mg/100 g of protein)	Hue angle	Chroma
Dried								
GF1	10.02 ± 0.13 b	0.31 ± 0.01 a	8.37 ± 0.04 e	1.17 ± 0.19 cd	80.14	24 ± 2 ab	−1.52 ± 0.00 c	57.25 ± 4.92 cd
GF2	9.89 ± 0.18 b	0.89 ± 0.00 f	9.90 ± 0.02 g	1.08 ± 0.22 c	78.23	19 ± 1 a	1.53 ± 0.00 e	55.61 ± 0.79 bc
GF3	11.52 ± 0.08 de	0.42 ± 0.00 c	7.71 ± 0.02 d	0.68 ± 0.00 a	79.67	134 ± 2 g	−1.56 ± 0.00 a	51.85 ± 0.43 a
GF4	11.26 ± 0.17 cde	0.37 ± 0.00 b	5.42 ± 0.03 b	1.42 ± 0.12 d	81.52	66 ± 3 d	1.57 ± 0.00 f	64.81 ± 1.52 g
GF5	11.88 ± 0.53 e	0.43 ± 0.00 c	5.29 ± 0.01 a	1.05 ± 0.02 bc	81.35	95 ± 6 e	1.51 ± 0.00 d	62.66 ± 2.28 fg
GF6	11.22 ± 0.81 cde	0.49 ± 0.00 d	8.86 ± 0.07 f	1.19 ± 0.01 cd	78.24	49 ± 5 c	−1.55 ± 0.00 b	60.44 ± 1.46 ef
GF7	10.48 ± 0.16 bc	0.63 ± 0.02 e	7.12 ± 0.19 c	0.95 ± 0.15 abc	80.82	30 ± 2 b	1.57 ± 0.00 f	64.80 ± 1.54 g
GF8	10.85 ± 0.14 cd	0.64 ± 0.01 e	7.00 ± 0.07 c	0.95 ± 0.04 abc	80.56	50 ± 2 c	−1.53 ± 0.00 c	59.85 ± 0.74 de
W	8.78 ± 0.20 a	0.90 ± 0.01 f	13.78 ± 0.03 h	0.76 ± 0.19 ab	75.78	121 ± 1 f	1.53 ± 0.01 e	53.62 ± 1.51 ab
Cooked								
GF1	65.62 ± 0.18 b	0.18 ± 0.03 a	8.92 ± 0.01 e	0.87 ± 0.02 ab	24.41	84 ± 0 bc	−1.43 ± 0.01 d	34.48 ± 3.60 c
GF2	66.32 ± 0.07 bc	0.61 ± 0.05 d	10.81 ± 0.06 g	1.12 ± 0.21 bcd	21.14	71 ± 7 b	−1.49 ± 0.00 b	30.32 ± 0.80 b
GF3	64.57 ± 0.18 a	0.31 ± 0.01 bc	8.24 ± 0.04 d	0.85 ± 0.19 ab	26.03	198 ± 7 e	−1.47 ± 0.02 bc	16.53 ± 2.18 a
GF4	66.81 ± 0.04 cd	0.17 ± 0.05 a	5.97 ± 0.01 b	1.17 ± 0.09 cd	25.88	131 ± 6 d	−1.48 ± 0.00 b	42.49 ± 1.21 d
GF5	69.78 ± 0.13 f	0.24 ± 0.10 ab	5.63 ± 0.03 a	0.82 ± 0.05 a	23.53	48 ± 1 a	−1.49 ± 0.01 b	40.52 ± 3.05 d
GF6	67.32 ± 0.78 d	0.31 ± 0.01 bc	9.32 ± 0.10 f	0.70 ± 0.03 a	22.36	79 ± 5 bc	−1.45 ± 0.03 cd	17.34 ± 2.36 a
GF7	67.38 ± 1.08 d	0.38 ± 0.02 c	7.44 ± 0.05 c	1.32 ± 0.12 d	23.48	77 ± 6 b	−1.43 ± 0.05 d	16.53 ± 1.28 a
GF8	68.17 ± 0.03 e	0.39 ± 0.01 c	7.56 ± 0.10 c	0.90 ± 0.14 ab	22.99	91 ± 10 c	−1.43 ± 0.03 d	29.85 ± 2.95 b
W	67.15 ± 0.06 d	0.60 ± 0.02 d	14.46 ± 0.01 h	0.92 ± 0.04 abc	16.87	360 ± 4 f	−1.52 ± 0.00 a	29.77 ± 0.48 b

Mean ± standard deviation. Different letters within the same parameter for dried or cooked pasta differ significantly ($P < 0.05$)

proximate composition (Table 2) of dried and cooked pasta was compared, it was observed that ash and carbohydrates were released to the cooking water, resulting in higher protein content in the cooked pasta. Cooking affected the fat content of the GF pasta in different way, increasing it in GF7 but decreasing in GF1, GF5 and GF6.

Different cooking indicators (OCT, CL, WA, SI) of the cooking quality were evaluated (Table 3). GF samples had higher OCT than the wheat control, indicating that GF pasta required longer cooking to gelatinize all the starch granules, particularly those composed solely by corn flour (GF4 and GF5). Significant variability was observed among the amount of solid losses during cooking (CL). Considering that lower values are associated to better consistency and low stickiness [13], which in wheat pasta are related to the development of a stable and strong network of coagulated gluten proteins that entraps the starch granules [14], GF matrixes gave diverse network. In general, samples with emulsifier (GF1, GF3, GF6, GF8) showed lower CL, and samples only composed by corn (GF4 and GF5) had higher CL. Indeed, emulsifiers can interact with starch and/or protein [15] to form a good structure reducing the cooking losses. As described by Schoenlechner et al. [15], the use of emulsifiers (1.2%) reduced cooking loss of gluten-free pasta made with quinoa, amaranth and buckwheat flour blends. Great variability was also observed in the water absorption (WA) that ranged from 49 (GF2) to 107% (GF7). In fact, there was a strong negative correlation between WA and CL ($r = -0.73$, $P < 0.001$). Regarding SI, or rather the capacity of protein-starch-fiber network to retain the water, the highest value was obtained with GF5 that only contains corn flour. Results agree with those reported by Foschia et al. [16]. Results related to the pasta color (Table 2) show that in the dried group, GF4 and GF7 had the highest values of Hue angle and with GF5 they had the highest chroma values. Among the cooked samples GF1, GF7 and GF8 were found to be the samples with hue angle highest values. Nevertheless, cooking affected the chroma parameter, in fact all the cooked samples had lower values that the ones observed for the dried group. GF4 and GF5 had the highest chroma parameter, related to yellowness, which could be expected owing to the solely presence of corn flour in their composition.

Texture Properties

Texture properties of cooked pasta was evaluated (Table 3). Hardness, a required force to get a compression of spaghetti strands, is a good index of the pasta consistency. GF7 showed the highest consistency followed by GF6 and GF8. Hardness values were within the range obtained by Motta Romero et al. [17], when making pasta with proso millet flour and hydrocolloids (4,480–8,454 g). A negative weak correlation ($r = -0.5125$; $P < 0.05$) was found between hardness and the protein content in cooked sample, thus when the proteins amount increased, hardness decreased. Adhesiveness, related to the stickiness of pasta, also significantly varied within samples, showing GF8 the lowest value and GF4 was the stickiest owing to the highest adhesiveness. No trend could be established to explain that behavior considering the composition. In general, chewiness of GF pasta was higher than that of wheat pasta, thus high energy was required for swallowing GF spaghetti. Resilience of GF pasta was higher than that of wheat pasta, except for GF7 and GF8. GF4 and GF5, made with corn flour, showed the highest resilience or capacity to recuperate the initial state after compression force. GF5 also showed the highest firmness, which is related to good quality pasta. Firmness values were close to those reported for wheat-based spaghetti (174–183 g) [18] and much lower than those reported by Bouasla et al. [19] when gluten free pasta was enriched with different amount and kind of legumes flours (199.50–326.50 N). The work of shear showed the same tendency of firmness. Elastic limit, measured as the resistance (g) to extension was higher in GF1, GF4, GF5 and GF8 had the lowest value. A strong positive correlation ($r = 0.8366$; $P < 0.05$) was observed between elastic limit and resilience, namely samples with high capacity to recuperate the state before compression had more resistance to tensile force. Elasticity (mm) was higher in pasta made simply by corn flour (GF4 and GF5).

Furosine Content

Furosine were measured to assess possible heat damage during pasta drying. The statistical analysis showed significant differences ($P < 0.05$) in FUR content in both pasta samples: dried and cooked (Table 2), indicating high variability in drying conditions and raw materials in all 9 pasta samples. No information has been previously reported regarding the content of FUR in GF pasta. Values of FUR in dried pasta ranged between 19 and 134 mg FUR/100 g proteins, with the highest values of FUR obtained for samples GF3 and GF5 (134 and 95 mg FUR/100 g proteins, respectively). Much research has been dedicated to demonstrate the influence of drying cycles on FUR content in wheat pasta, ranging from 45 up to 209 mg/100 g of protein for pasta from artisanal processes (short temperatures long times), while in industrial ones (high temperatures short times) FUR reached values from 390 up to 562 mg/100 g of proteins [5, 20]. In addition, recipes can also impact the FUR content of wheat spaghetti. In fact, in those enriched with common bean, values of FUR ranged between 25 and 77 mg /100 g of proteins with higher values obtained in products with higher bean content (30%) and higher drying temperatures (80 °C) [17]. In lupine enriched spaghetti (0–20%) FUR values ranged between 200 and 300 mg /100 g of protein after drying at 60 °C for 18–20 h [21]. Cooking process significantly increased the FUR content ($P < 0.05$) except in

Table 3 Cooking quality indicators and texture parameters

Sample	OCT (min)	CL (%)	WA (%)	SI (g water/g dry pasta)
GF1	14.00 ± 0.71 d	3.22 ± 0.66 a	106 ± 3 f	1.68 ± 0.04 ab
GF2	12.75 ± 0.35 bc	20.56 ± 1.22 f	49 ± 2 a	1.74 ± 0.05 bc
GF3	13.25 ± 0.35 cd	8.21 ± 0.37 d	84 ± 8 cd	1.61 ± 0.04 a
GF4	15.50 ± 0.71 e	29.82 ± 0.94 g	68 ± 6 b	1.81 ± 0.08 cd
GF5	16.00 ± 0.71 e	19.95 ± 0.10 f	75 ± 4 bc	2.06 ± 0.08 e
GF6	14.25 ± 0.35 d	4.83 ± 0.46 b	93 ± 6 de	1.83 ± 0.03 cd
GF7	13.75 ± 0.35 cd	12.06 ± 0.20 e	107 ± 5 f	1.75 ± 0.04 bc
GF8	11.75 ± 0.35 ab	6.98 ± 0.72 c	93 ± 7 de	1.88 ± 0.02 d
W	11.25 ± 0.35 a	5.67 ± 0.20 b	94 ± 6 e	1.83 ± 0.10 cd

Sample	Hardness (g)	Adhesiveness (g·s)	Chewiness (g)	Resilience	Firmness (g)	Work of Shear (g·s)	Elastic Limit (g)	Elasticity (mm)
GF1	5408 ± 348 a	7.8 ± 0.7 c	3747 ± 553 cd	0.42 ± 0.01 cd	231 ± 4 e	28 ± 1 cd	42 ± 2 f	36 ± 5 ab
GF2	6560 ± 735 bc	7.9 ± 0.6 c	4317 ± 786 de	0.39 ± 0.02 c	166 ± 9 b	24 ± 1 bc	38 ± 2 e	36 ± 4 ab
GF3	6254 ± 655 abc	7.6 ± 0.8 c	3295 ± 512 bc	0.35 ± 0.02 b	155 ± 8 b	26 ± 5 bcd	31 ± 2 c	56 ± 6 c
GF4	6927 ± 844 cd	9.5 ± 0.7 d	5007 ± 434 e	0.44 ± 0.01 d	201 ± 10 cd	21 ± 3 ab	42 ± 1 f	73 ± 8 e
GF5	6331 ± 487 abc	7.6 ± 0.3 c	3843 ± 543 cd	0.43 ± 0.02 d	268 ± 6 f	28 ± 1 d	43 ± 2 f	64 ± 3 d
GF6	7795 ± 873 de	6.2 ± 0.4 b	3735 ± 558 cd	0.34 ± 0.02 b	161 ± 6 b	26 ± 1 cd	35 ± 2 d	34 ± 4 a
GF7	8366 ± 875 e	6.1 ± 1.0 ab	4936 ± 443 e	0.30 ± 0.03 a	110 ± 8 a	18 ± 3 a	25 ± 1 b	34 ± 3 a
GF8	7990 ± 533 de	5.1 ± 0.7 a	3224 ± 401 ab	0.30 ± 0.05 a	190 ± 9 c	22 ± 3 ab	20 ± 1 a	38 ± 3 ab
W	5615 ± 598 ab	7.8 ± 0.5 c	2121 ± 239 a	0.29 ± 0.02 a	205 ± 4 d	28 ± 0 cd	31 ± 3 c	41 ± 4 b

Mean ± standard deviation. Different letters within the same parameter differ significantly ($P < 0.05$)

OCT Optimal cooking time, *CL* Cooking loss, *WA* Water absorption, *SI* Swelling index

GF5, in which unexpectedly the FUR content significantly decreased. However, some authors have pointed out that Amadori compounds, which are formed in the early stages of the MR, are not stable and they are easily converted to intermediate and final products, depending on the progress of the MR [22]. In fact, GF5 had the highest cooking time. According to Giannetti et al. [20] high intensity heat treatments can promote a dramatic progression of MR leading to FUR underestimation. The values obtained in cooked samples ranged between 48 and 360 mg FUR/100 g proteins with an increase in the content of FUR of around 38%. The highest FUR content was obtained for wheat pasta. Despite the toxicity of FUR, no data on the content of FUR in pasta after cooking are available in the literature. Only FUR content of fresh wheat egg pasta after double pasteurization (1: 95 °C for 1.5 min; 2: 70–93 °C for 60–90 min) were reported in the range 11.12–87.61 mg/ 100 g of proteins [23].

Principal Component Analysis

To understand the possible role of specific GF ingredients in the quality of the resulting pasta, all the parameters were subjected to a principal component analysis. The results of PCA indicated that 34 and 23% of the variation was explained by principal components 1 (PC1) and 2 (PC2), respectively (Fig. 1). GF2 and GF3 were the samples closer to wheat durum pasta for protein and ash content, furosine and work of shear. GF1, GF4 and GF5, behaved similar regarding cooking performance (OCT, CL, SI), texture (adhesiveness, resilience, firmness, elastic limit, elasticity) and color (hue angle of dried samples and chroma in cooked stage). GF6, GF7 and GF8 had in common water absorption. Therefore, it was possible to discriminate GF pasta regarding their technological and nutritional behavior.

Conclusion

There were significant differences in the nutritional composition between the gluten free spaghetti and the durum wheat pasta. In general, gluten free samples in both stage (dried and cooked) were poorer in protein and ash with respect to the wheat pasta. Regarding quality indicators, GF samples showed significantly different behavior, and they could not be related to specific ingredients. Nevertheless, pasta made with corn flour required longer cooking and had high cooking loss values but resulted in more resilient and elastic pasta. The furosine content in dried GF pasta was majorly lower than that in wheat pasta, and those differences were greatly magnified after cooking. In the recent years, food technology applied to gluten free pasta production has taken great steps forward. Nevertheless, looking at the results, today nutritional

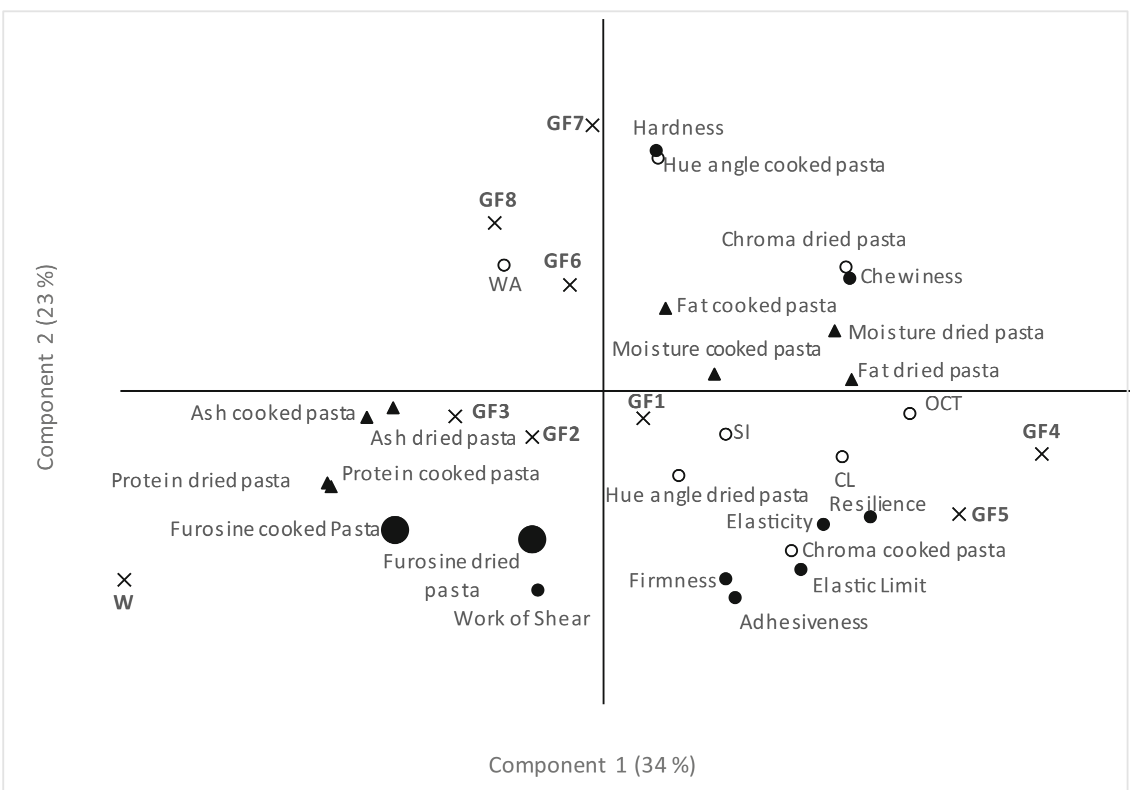

Fig. 1 Score plot from a principal component analysis of the combination of components weight (chemical composition ▲, cooking quality indicators ○, texture parameters • and furosine content ●)

improvements and new technologies approaches are needed to assure a nutritional balanced food.

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Compliance with Ethical Standards

Conflicts of Interest The authors declare that they do not have any conflict of interest.

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Role of hydrocolloids in gluten free noodles made with tiger nut flour as non-conventional powder

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ABSTRACT

Gluten free noodles have been made from a variety of ingredients, but very often without applying ingredients and process knowledge. Aim of this study was to build up gluten free noodles using tiger nut flour, a selection of hydrocolloids (guar gum, xanthan gum, inulin and carboxymethyl cellulose) and considering the impact of dough hydration in noodles making. Dough rheology, fresh noodles characteristics, cooking quality indicators and noodles quality after cooking were evaluated. Results showed that hydration level significantly affected dough rheology during mixing, heating and cooling, with a significant ($P < 0.05$) impact on hardness and firmness of fresh noodles and adhesiveness and firmness of cooked noodles. Hydrocolloids type significantly affected the characteristics of fresh and cooked noodles, but the extent of their effect was dependent on the hydration level applied during noodles making. In general, hydrocolloids increased dough consistency, resulting in fresh noodles with higher diameter, hardness and firmness, trend that was maintained after cooking, reducing cooking losses. In particular, gluten free tiger nut noodles made in the presence of 0.5% xanthan gum and adjusting the amount of water showed the best performance, with low cooking losses and high firmness.

1. Introduction

The design and development of gluten free (GF) foods is attracting very much attention, as a consequence of their increased consumption linked to whatever motivation (intolerances, lifestyle and so on). GF bread is the food that has prompted more extensive research trying to understand the matrixes, interactions among polymers and the impact of process conditions on the quality of the fresh bread (Matos & Rosell, 2015; Renzetti & Rosell, 2016). However, there are other widely consumed gluten free foods that are usually empirically designed by mixing gluten free ingredients without understanding their inner functionality.

Among the worldwide most consumed GF foodstuffs are noodles. They are a staple food owing to its variety, versatility, taste and price (Heo, Lee, Shim, Yoo, & Lee, 2013), representing a viable alternative for gluten intolerant patients. Noodles can be produced from various grain flours such as wheat, rice, buckwheat and corn, following three steps: raw material mixing (flour, water, salt), dough sheeting and cutting (Heo et al., 2013). Nevertheless, in the GF field, noodles are generally produced through extrusion or sheeting of a rice batter (Fu, 2008). The versatility of those technologies has stimulated the development of multitude of noodles, incorporating ingredients for nutritional enrichments, like pseudocereals and legumes (Bilgici, 2013; Levent, 2017), or looking for healthy patterns (Levent, 2017). Lately, extensive research has been reported adding alternative raw materials that provide functional properties (Heo, Jeon, & Lee, 2014), but their role within the process requires additional understanding.

Tiger nut (*Cyperus esculentus* L.) (TN) is a specie of herbaceous plant that produces an edible sweet tuber and it is widely cultivated in Spain, Burkina Faso, Mali, Niger and Nigeria (Pascual, Maroto, López-Galarza, Sanbautista, & Alagarda, 2000). It has been used to produce a milky beverage (*orchata*) and for feeding fish and farm animals (Pascual et al., 2000). From a nutritional point of view, TN is characterized by a good amount of fiber and omega-6 fatty acids (Codina-Torrella, Guamis, & Trujillo, 2015; Sánchez-Zapata, Fernández-López, & Angel Pérez-Alvarez, 2012). These components could be play a key role in the prevention of some diseases such as coronary heart disease, colon cancer, diabetes and obesity (Martín-Esparza, Raigón, Raga, & Albors, 2018a), diabetes and obesity. For this reason, TN is attracting great interest in human nutrition. In fact, many researches tried to exploit those properties developing food applications like TN oil (Ezeh, Gordon, & Niranjan, 2014), flours and biscuits (Chinma, Abu, & Abubakar, 2010; Pascual et al., 2000) and fresh egg pasta (Albors, Raigon, García-Martinez, & Martín-Esparza, 2016). In addition, the natural absence of gluten makes TN a potentially useful raw material for the manufacture of foods intended to consumers with specific nutritional needs. Despite its great potential, as mentioned above, the

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number of researches aimed to the development of TN based gluten free foods is very limited in literature, which include the use of up to 25% TN flour to improve the nutritional and functional quality of GF breads (Demirkesen, Sumnu, & Sahin, 2011) or extruded snacks mixed with cassava flour (Adebowale et al., 2017). Therefore, no previous study has been focused on developing GF noodles using TN as major ingredient.

To make GF noodles, the lack of gluten must be counteracted with ingredients that overcome the loss of extensibility. Hydrocolloids represent a group of water-soluble polysaccharides with different chemical structure widely used in the food industry as gelling agents and thickeners, emulsifiers, stabilizers, foaming agents, improvers of water retention and texture (Dickinson, 2003). Some studies have already emphasized the hydrocolloids role on GF noodles, mainly proposing the use of xanthan gum (XG), guar gum (GG) and carboxymethyl cellulose (CMC) to improve their texture and cooking quality (Srikaeo, Laothongsan, & Lerdluksamee, 2018).

The objective of this study was to build gluten free noodles applying knowledge on ingredients and processing. With that purpose, a gluten free flour or powder with described healthy properties (TN flour) was chosen, and a selection of hydrocolloids with varied chemical structure (GG, XG, inulin and two types of CMC with diverse viscosity) were evaluated. Moreover, the role of hydration level in noodles making was assessed by using two distinct hydrations: constant hydration usually applied in literature and adapted hydration depending on the food matrix requirements.

2. Materials and methods

2.1. Materials

Milled TN was kindly granted by Món Orxata (Alboraia, Valencia, Spain). TN powder had 8.62% moisture, 48.29% carbohydrates, 26.27% total fat, 7.67% protein, 7.31% total fiber, 1.84% ash content. GG from Cargill (Spain), XG food grade from Jungbunzlauer (Ladenburg, Germany), inulin (Fibruline® XL) from Cosucra Groupe (Warcoing, Belgium), while CMC (Methocel A15 and Methocel A4M) were generously provided by Dow Pharma & Food Solutions (La Plaine Saint Denis, France).

2.2. Noodles preparation

Commercial TN powder was ground using a laboratory mill (IKA Eurostar M 20, Staufen, Germany) equipped with a water-cooling jacket, applying 3 cycles for 10 s, with a pause of 10 s between cycles. Particle size of the resulting powder was measured by laser diffraction (Mastersizer Scirocco 2000; Malvern Instruments Ltd., Worcestershire, UK), displaying d(0.50) and d(0.90) of 553,06 μm, and 1337.87 μm, respectively.

For noodles making, preliminary studies were run to determine the amount of water that allowed preparing a dough with TN powder with appropriate consistency to be extruded. In a control sample TN powder and distilled water (8:2, w:w) were mixed using a laboratory mixer (IKA Eurostar 40, Staufen, Germany) for 2 min at 100 rpm. Resulting dough was then extruded using a syringe (ø 3 mm) and noodles were poured directly into one liter of boiling water. The water was standardized following the official method (AACC official method 66–50.01). After ± 30 s, noodles were drained and cooled at ± 25 °C for 5 min before drying at 40 °C for 2 h in a vacuum oven (NÜVE EV 018, Ankara, Turkey). Noodles containing hydrocolloids were prepared adding 0.5% (w/w in solid basis) of GG, XG, I, CMC A15 or CMC A4M, following the same protocol described above to obtain those samples referred as constant hydration level. Other set of samples, named adapted water hydration, was prepared having same consistency (similar Mixolab torque during the mixing-heating-cooling cycle) than the control. The amount of water needed to reach same torque during mixing was determined in preliminary assays carried out with the Mixolab®. Three batches were prepared for each type of noodles.

2.3. Rheological behavior

Dough (75 g) prepared as previously described was placed into the Mixolab® bowl (CHOPIN Technologies, France). Rheological behavior was recorded using the protocol Chopin +, with the following settings: mixing for 8 min at 30 °C, heating from 30 °C to 90 °C at the rate of 4 °C/min, hold at 90 °C for 7 min, cooling to 50 °C at 4 °C/min and finally held at 50 °C for 5 min (Matos & Rosell, 2013). The dough consistency obtained with the control sample was used as a target consistency and the amount of water required to reach that consistency with each recipe was determined. That water absorption was applied to obtain the set of noodles referred as adapted water hydration. Parameters recorded from the plots included: consistency during mixing (Nm), minimum torque during heating stage (Nm) and maximum torque during cooling stage (Nm). Two assays were performed for each sample, and mean values calculated.

2.4. Extrusion force

The force needed to extrude the dough (8 g) through the 12 mL syringe (ø 3 mm) was quantified using a Texture Analyzer (TAHDi/500, TAHD Co., Stable Micro System, Surrey, UK) equipped with a load cell of 30 kg and P/36 cylinder probe (36 mm Ø) and the following settings: pre-test speed and test speed was 1 mm/s, post-test speed was 10 mm/s, and trigger force was 10 g. The maximum peak of a compression test was identified as extrusion force. Three measurement were carried out for a single dough and mean value was recorded.

2.5. Cooking quality indicators

Optimal Cooking Time (OCT) was evaluated according to AACC official method (AACC International, 2009) with some modifications. Noodles (5 g) were immersed in boiling water (60 mL), one piece of noodles (5 cm) was taken out every 30 s and squeezed between two Petri dishes to visually observe the time of disappearance of the white core (ungelatinized starch). The OCT was achieved when center core just disappears, thus complete starch gelatinization. Two measurements were performed for each sample and mean values recorded.

Cooking loss (CL) was evaluated according to AACC official method (AACC International, 2009) with some modifications. After reaching OCT, noodles were drained in a Büchner funnel and rinsed with 50 mL of distilled water for 30 s. Cooking and rinse waters were combined and the total volume annotated. An aliquot of this water (1 mL) was dried during 24 h up to constant weight in air oven (J. P. Selecta 2000210, Barcelona, Spain) at 65 °C. After 1 h in a desiccator, the residue (Solid loss) was weighted and calculated CL as percentage using Equation (1). Three measurements were performed for each sample, and mean value was calculated.

$$CL_{(g/100\,g)} = (\text{Solid loss}_{(g)} \times \text{Final cooking water}_{(mL)}) / (\text{Raw Noodles}_{(g)}) \times 100 \quad \text{[Eqn 1]}$$

Drained noodles were allowed to cool down to room temperature for 10 min in a sealed Petri dish. Then, five pieces of noodles (5 cm long) were weighed and water absorption (WA) expressed in percentage was determined following equation (2):

$$WA_{(g/100\,g)} = (\text{Cooked Noodles}_{(g)} - \text{Raw Noodles}_{(g)}) / (\text{Raw Noodles}_{(g)}) \times 100 \quad \text{[Eqn 2]}$$

Three measurements were performed for each analysis, and mean values calculated.

Five pieces of drained noodles (5 cm long) were weighed and dried for 24 h to constant weight in an air oven (J. P. Selecta 2000210, Barcelona, Spain) at 65 °C. Swelling index (SI) expressed as grams of water per gram of dry noodles was determined following the procedure described by Cleary and Brennan (2006) and calculated as indicated in equation (3):

$$SI_{\text{(g water/g dry noodles)}} = (\text{Cooked Noodles}_{(g)} - \text{Cooked Noodles after drying}_{(g)}) / (\text{Cooked Noodles after drying}_{(g)}) \quad \text{[Eqn 3]}$$

Three measurements were performed for each sample, and the mean values were calculated.

Diameter of uncooked and cooked GF noodles was measured with a digital caliper. Five measurements were performed for each sample and mean value reported.

Color of uncooked and cooked GF noodles was measured using a Minolta Chromameter (Model CR-400, Minolta Co., Osaka, Japan) following CIELAB scale: L^* (lightness 0–100), a^* (positive values measure redness, negative values measure greenness) and b^* (positive values measure yellowness, negative values measure blueness). Each data represents the mean of five measurements.

Texture profile analysis (TPA) was carried out in a Texture Analyzer (TAHDi/500, TAHD Co., Stable Micro System, Surrey, UK) equipped with a load cell of 30 kg and P/36 cylinder probe (36 mm Ø) and with the following settings: pre-test, test and post-test speed was 2 mm/s and the trigger force was 10 g. Distance was set up at 30 mm with 75% strain and 2 s the holding time between compressions. Five strands (5 cm long) of fresh and cooked noodles were submitted to a double compression test and Hardness (g), Adhesiveness (g·s), Chewiness and Resilience were measured with the software instrument. Five replicates were evaluated for each experimental value reported.

Firmness, expressed as the maximum force (g) necessary to shear a strand of noodles, and work of shear (g·s) corresponding to the area under the curve of force/time graphic, were investigated using a Texture Analyzer (TAHDi/500, TAHD Co., Stable Micro System, Surrey, UK) as described by the official method 66–50.01 (AACCInternational, 2001). Samples were analyzed in triple and Data were calculated with the software instrument and the average of three replicates used.

2.6. Statistical analysis

Experimental data were expressed as a mean ± standard deviation and statistically analyzed by multifactor analysis of variance (MANOVA) using Statgraphics V.7.1 (Bitstream, Cambridge, MN). Fisher's least significant differences test was used for assessment of significant differences among experimental mean values with 95% confidence.

3. Results and discussion

3.1. Rheological behavior

Mixolab® has been traditionally applied to record bread doughs performance during mixing when subjected to a heating-cooling cycle. In the present study, Mixolab® was used to record the rheology of a GF noddle dough made from TN powder. Therefore, plots (Fig. 1) displayed completely different patterns than the ones observed either with gluten matrixes or non-gluten flours. Fresh doughs were directly placed into the bowl, recording initial consistency that showed a slight decay as mixing proceeds, indicating some loss of stability. A pronounced dough softening was observed during heating stage and no increase in consistency, usually attributed to starch gelatinization, was observed owing to the low amount of starch or limited amount of water. Dough consistency only exhibited a steady increase during the cooling stage.

In GF noodles, hydrocolloids are needed to provide extensibility. But, considering their diverse ability to bind water, it was assumed that water would have a crucial role in defining noodles dough consistency and the quality of fresh noodles. Nevertheless, usually constant amount of water is applied when producing rice noodles, and slightly varied when changing the flour. Considering that a non-cereal flour was used and the diverse characteristics of the hydrocolloids, dough rheology was tested applying different hydrations till reaching the same consistency in all the blends for obtaining noodles (Fig. 1). Two types of CMC were selected due to their great variation on viscosity (CMC A15 15 mPa s and CMC A4M 4000 mPa s). Firstly, doughs were prepared using the same amount of water, which were referred as constant hydration (Fig. 1 A), and secondly, doughs were prepared adjusting the level of water to hydrate all ingredients, leading to doughs with the same consistency (same torque during mixing with a Mixolab) than the control sample, those were referred as constant consistency (Fig. 1 B). The incorporation of hydrocolloids to doughs resulted in doughs with higher consistency when the level of water was kept constant (Fig. 1 A), due to their water retention ability. Those differences were minimized when the water level was adapted, allowing hydrocolloids hydration, being GG followed by XG the hydrocolloids that required higher amount of water to adjust consistency (Fig. 1 B). Parameters used to quantify rheological behavior are showed in Table 1, where type of dough hydration and presence of hydrocolloids were identified as main factors of variance. Statistical analysis confirmed the significant ($P < 0.05$) effect of hydration on the dough rheology during mixing, heating and cooling stages, but no significant effect was observed due to the type of hydrocolloid because differences were eliminated when constant consistency was applied. Nevertheless, the analysis of variance within the samples group with constant hydration showed a significant ($P < 0.05$) effect of hydrocolloid on the consistency (Nm) during the Mixolab® stages. When constant hydration (20 g water/100 g powder) was used for making doughs, the addition of hydrocolloids, except for inulin, significantly increased dough consistency during mixing and cooling stages with respect to control (in the absence of hydrocolloid). XG and GG were the hydrocolloids that mostly increased consistency during mixing and cooling stages, indicating their higher absorption of water. The enhancement of consistency during cooling promoted by all hydrocolloids, with exception of inulin, suggested higher interaction between hydrocolloids and leached amylose, which might indicate that gums were preferentially placed on the surrounding solution instead of being located at the external side of the granules (Rosell, Yokoyama, & Shoemaker, 2011).

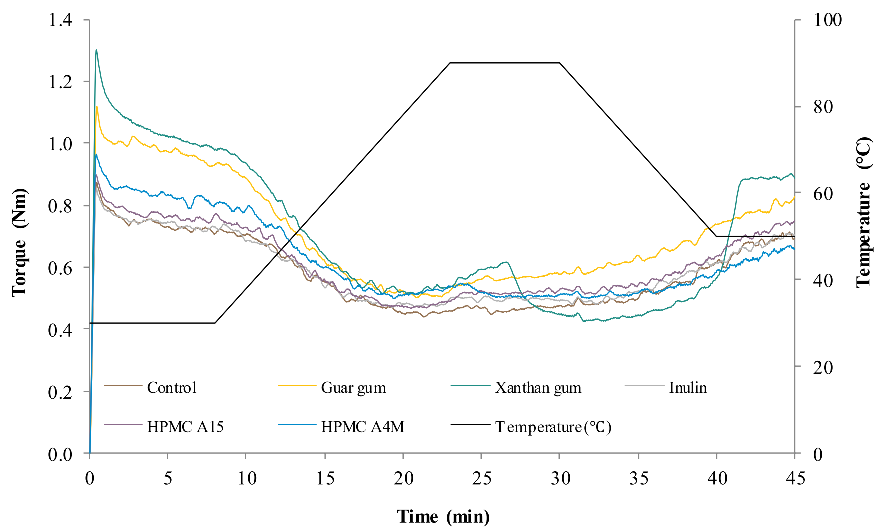
To obtain a constant consistency, doughs required higher amount of water with the subsequent reduction in the torque (Nm), apart from inulin that did not affect consistency (Table 1). After adjusting water levels, no significant differences were observed due to hydrocolloids along mixing, heating and cooling stages.

Extrusion force required to obtain noodles were also evaluated, and significant differences were observed due to the hydration level and the type of hydrocolloids (Table 1). Differences within hydrocolloids type were only significant when constant hydration was applied to obtain doughs, confirming that doughs containing XG or GG required higher extrusion force. Same findings were observed when GG was added (0–10%) to various flours (corn, potato, rice, and wheat) to produce extruded snacks, observing an increase of extrusion torque (Nm) with all the flours (Parada, Aguilera, & Brennan, 2011).

3.2. Quality indicators of fresh noodles

It was possible to make noodles with TN powder, despite the absence of gluten and the particular characteristics of this small tuber. Characterization of GF TN noodles was carried out at the different stages of production, including fresh noodles and their quality after cooking. Regarding fresh noodles, the statistical analysis revealed that hydration level significantly affected the luminosity of the noodles (L^*), hardness, firmness and the work of shear; conversely hydrocolloids type significantly affected all the parameters evaluated with the exception of work of shear (Table 2). Therefore, on fresh noodles, the type of hydrocolloid was crucial for producing GF noodles. Going into detailed

A.



B.

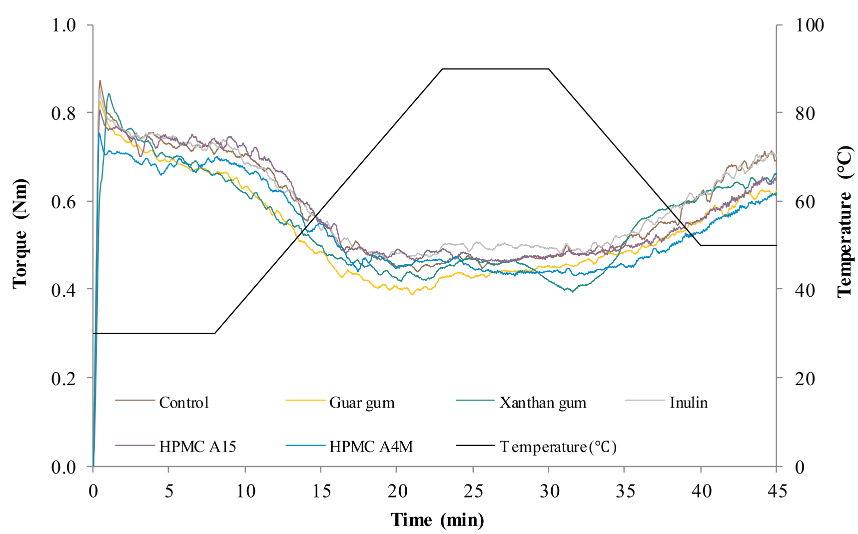


Fig. 1. Mixolab® curves representing the rheological behavior of the noodle's doughs. A. Doughs obtained with the same hydration; B. Doughs obtained with adapted hydration level to keep constant consistency. Solid black line displays the temperature gradient applied during the rheology assessment.

effects, it was observed that GG, XG and CMC A4M increased the noodles diameter, independently of the amount of water used for producing them. That increase confirmed the high ability of those hydrocolloids to retain water. Color was differently affected by hydrocolloids, observing a decrease of luminosity, although only significant when hydration was adapted and in the presence of XG, GG or CMC A15. Additionally, a^* and b^* were significantly ($P < 0.05$) influenced by hydrocolloids, particularly, XG had the higher values in terms of redness, whereas GG led to lower redness and yellowness. The parameters a^* and b^* were very close to those obtained with fresh durum pasta containing TN (Martín-Esparza, Raigón, Raga, & Albors, 2018b).

Hardness and firmness were significantly affected by hydration level and hydrocolloids ($P < 0.05$) (Table 2). In general, hardness, the force necessary to attain a given deformation (Li, Dhital, & Wei, 2017), was higher in the noodles with constant hydration, after adjusting the water softer noodles were attained. When hydrocolloids were added, hardness increased especially when CMC A15 was used, independently of the hydration, and GG, but only when constant hydration was used. Thus, hydrocolloids improved the stiffness of GF noodles structure. Similar result was obtained by Padalino, Mastromatteo, De Vita, Maria Ficco, and Del Nobile (2013) that reported an increase of hardness in GF spaghetti made by corn and oat when they were separately added (2% of GG and CMC). Firmness, the force required to cut the noodle, was higher in noodles with adapted hydration, except for those containing inulin. XG was the hydrocolloid that conferred higher firmness. Adhesiveness, the work necessary to overcome the attractive forces between the surface of the noodle and the surface of teeth (Li et al., 2017), was significantly reduced in the presence of hydrocolloids, excepting XG when it was added without adjusting the hydration level, which led to more adhesive noodles. Likewise, Chauhan, Saxena, and Singh (2017) described the decrease of adhesiveness when 0.5% or 1% of hydrocolloids (GG, acacia gum and tragacanth gum) were added to amaranth GF pasta. Chewiness or the energy needed to make noodles in a state suitable for swallowing (Li et al., 2017) were significantly increased by all the hydrocolloids tested, but the extent of the impact was dependent on the hydration level applied to noodles. In fact, CMC A15 resulted in

Table 1
Thermomechanical behavior of Tiger nut doughs with constant hydration and constant consistency.

Hydration	Hydration level (g/100 g)	Hydrocolloid	Mixing Consistency (Nm)				Heating Consistency (Nm)				Cooling Consistency (Nm)				Extrusion Force (g)			
Constant	20.00	None	0.71	±	0.01	**ab**	0.44	±	0.03	**abc**	0.70	±	0.02	**de**	5371	±	175	**a**
	20.00	Guar gum	0.96	±	0.01	**e**	0.50	±	0.01	**c**	0.83	±	0.00	**g**	6453	±	135	**c**
	20.00	Xanthan gum	1.01	±	0.03	**f**	0.43	±	0.01	**ab**	0.89	±	0.02	**h**	6516	±	23	**c**
	20.00	Inulin	0.73	±	0.01	**b**	0.47	±	0.01	**bc**	0.71	±	0.01	**e**	5246	±	53	**a**
	20.00	CMC A15	0.77	±	0.01	**c**	0.46	±	0.01	**abc**	0.76	±	0.01	**f**	5967	±	236	**b**
	20.00	CMC A4M	0.82	±	0.03	**d**	0.50	±	0.01	**c**	0.66	±	0.01	**c**	6085	±	178	**b**
Adapted	20.00	None	0.71	±	0.01	**ab**	0.44	±	0.03	**abc**	0.70	±	0.02	**de**	5371	±	175	**a**
	26.00	Guar gum	0.70	±	0.01	**ab**	0.39	±	0.01	**a**	0.63	±	0.01	**ab**	5264	±	161	**a**
	24.00	Xanthan gum	0.70	±	0.01	**ab**	0.39	±	0.05	**a**	0.67	±	0.02	**cd**	5281	±	70	**a**
	20.00	Inulin	0.73	±	0.01	**b**	0.47	±	0.01	**bc**	0.71	±	0.01	**e**	5246	±	53	**a**
	21.06	CMC A15	0.73	±	0.01	**abc**	0.44	±	0.01	**abc**	0.66	±	0.00	**bcd**	5385	±	247	**a**
	21.30	CMC A4M	0.69	±	0.01	**a**	0.42	±	0.05	**ab**	0.62	±	0.01	**a**	5125	±	58	**a**
***P*-value**		Hydration	0.00				0.00				0.00				0.00			
		Hydrocolloid	0.10				0.16				0.08				0.01			

*means with different letters within the same parameter differ significantly ($P < 0.05$).

the highest chewiness when using constant hydration, despite its lower initial viscosity, but GG led to the highest value when adapted hydration was applied. Hydrocolloids also significantly increased the noodles resilience, except for XG when constant hydration was used. Analyzing the work required to shear, it significantly ($P < 0.05$) depended on the hydration level, samples group with constant hydration was characterized by lower results. GG, CMC A15 and CMC A4M after adjusting hydration led to noodles with the highest value in work of shear. Therefore, level of hydration significantly affected fresh noodles characteristics.

3.3. Quality indicators of cooked noodles

Generally, good quality noodles shall be characterized by low cooking loss, high firmness and absence of stickiness (Marti et al., 2013). In this study other quality indicators related to cooking have been evaluated (Table 3). The level of hydration significantly ($P < 0.05$) affected the color of the cooked noodles, and hydrocolloids affected the OCT, CL and water absorption, as well. Hydrocolloids incorporation increased significantly ($P < 0.05$) the OCT, which might be expected considering water limitation, and in consequence the delay in the starch gelatinization. Same trend was described by Kaur, Shevkani, Singh, Sharma, and Kaur (2015) when GG and XG were added (0.25% or 0.35%) to produce potato, corn and mung bean starch noodles, which has been recently explained by the protective behavior of the hydrocolloids towards starch granules that cause the increase in the gelatinization temperature onset (Pongpichaiudom & Songsermpong, 2018). On the other hand, hydrocolloids addition decreased ($P < 0.05$) the cooking loss, and those losses tended to be lower when adapted hydration was applied during the making process. The hydrocolloids improve the noodles' structure, preventing the release of solid matters into cooking water. The addition of XG allowed obtaining GF TN noodles that kept better their structure, and that effect was even greater when prepared adapting the hydration. Same action of gums on CL has been reported for GG, CMC and XG when making organic red jasmine rice noodles (Kraithong, Lee, & Rawdkuen, 2019) and dried-naturally fermented rice noodles (Srikaeo et al., 2018). Therefore, present research shows that hydrocolloids are also able to held a tuber powder for producing noodles.

The great ability of hydrocolloids to bind water was also sustained by the WA results, which were significantly ($P < 0.05$) higher, whatever hydration was applied (Table 3), except for the GG when the adapted hydration was applied. Even after adjusting the hydration, the presence of hydrocolloids promoted higher water absorption. Other authors described same results obtained with rice noodles (Srikaeo et al., 2018). The effect of hydrocolloids was evident also in the SI, indicating that hydrocolloids significantly increased the amount of bonded water to starch and proteins, leading to lower released of soluble compounds, except in the case of inulin. As mentioned above, hydration and hydrocolloids had a significant ($P < 0.05$) effect on the color parameter L^*, a^* and b^* of cooked noodles. TN tubers have a brownish color, that is maintained in TN noodles. In general, noodles made with constant hydration had higher L^*, a^* and b^*. Noodles containing inulin were brighter, with reddish and brownish tone.

Hydration type significantly ($P < 0.05$) affected the adhesiveness, resilience and firmness, and hydrocolloids type significantly affected all the texture parameters evaluated (hardness, adhesiveness, chewiness, resilience, firmness, work of shear) (Table 3). Regarding hardness, the most pronounced effect was the softening effect of GG and CMC A15, but only when constant hydration was applied; and the same trend was observed in the noodles' resilience. Hardness of cooked samples (Table 3) are in line with the values obtained for other noodles prepared with different flours, like tartary buckwheat noodles that showed hardness from 5960 g to 7780 g (Wu et al., 2017). In contrast with what was observed in fresh samples, in cooked noodles the presence of hydrocolloids increased the adhesiveness, although it was not significant in the case of inulin and CMC A15, and the former had significant effect when adjusting hydration. Consequently, gelling and starch gelatinization modified the impact of hydrocolloids on adhesiveness. Although hydrocolloids significantly affected chewiness, the effect was dependent on the type of hydrocolloids, being the impact greater when constant hydration was applied. It must be stressed the effect induced by GG, that reduced chewiness when noodles were prepared with constant hydration but when adjusting it, a significant increase in chewiness was obtained. Similarly, opposite effects were observed when adding XG depending on the hydration type applied during noodle making. XG induced a significant increase in chewiness when constant hydration, but no significant difference with the control was observed when using adapted hydration. As it was previously described with fresh noodles, firmness during shearing was significantly affected by hydration and hydrocolloid type, but the work required to cut noodles was significantly dependent on the type of hydrocolloids. Xanthan gum was the hydrocolloid that mostly affected the performance of noodles during shearing, especially when adapted hydration was used.

4. Conclusion

Gluten free noodles could be made from TN flour, giving special attention to the amount of water used in the production process and the

Table 2
Diameter, color and textural properties of fresh Tiger nut gluten free noodles.

Hydration	Hydrocolloid	Diameter (mm)	Color			Firmness	
			L*	a*	b*	Firmness (g)	Work of Shear (g·cm)
Constant	None	3.43 ± 0.06 a	44.40 ± 1.24 cd	5.70 ± 0.34 b	21.24 ± 0.31 de	32 ± 2 a	5.5 ± 0.4 ab
	Guar gum	3.57 ± 0.06 c	43.55 ± 1.70 bcd	4.09 ± 0.57 a	18.45 ± 0.16 a	35 ± 1 abc	5.0 ± 0.9 a
	Xanthan gum	3.57 ± 0.06 c	43.02 ± 1.27 bc	6.36 ± 0.51 c	21.65 ± 0.68 e	38 ± 1 abcd	5.6 ± 0.5 abc
	Inulin	3.47 ± 0.06 ab	43.20 ± 1.79 bc	5.59 ± 0.74 b	19.33 ± 0.49 b	33 ± 7 ab	5.8 ± 0.5 abc
	CMC A15	3.47 ± 0.06 ab	42.92 ± 1.03 abc	5.87 ± 0.41 bc	20.18 ± 0.22 c	36 ± 5 abcd	5.1 ± 1.3 a
	CMC A4M	3.53 ± 0.06 bc	45.19 ± 0.80 d	5.46 ± 0.40 b	20.79 ± 0.46 d	38 ± 2 abcd	6.8 ± 0.4 cd
Adapted	None	3.43 ± 0.06 a	44.40 ± 1.24 cd	5.70 ± 0.34 b	21.24 ± 0.31 de	32 ± 2 a	5.5 ± 0.4 ab
	Guar gum	3.53 ± 0.06 bc	41.20 ± 0.40 a	5.72 ± 0.18 b	20.87 ± 0.31 d	40 ± 2 cde	6.9 ± 0.5 d
	Xanthan gum	3.57 ± 0.06 c	42.44 ± 1.52 ab	5.70 ± 0.44 b	21.50 ± 0.30 e	44 ± 1 e	6.3 ± 1.0 bcd
	Inulin	3.47 ± 0.06 ab	43.20 ± 1.79 bc	5.59 ± 0.74 b	19.33 ± 0.49 b	33 ± 7 ab	5.8 ± 0.5 abc
	CMC A15	3.47 ± 0.06 ab	41.93 ± 1.41 ab	5.52 ± 0.37 b	20.11 ± 0.58 c	41 ± 3 de	7.5 ± 0.6 d
	CMC A4M	3.53 ± 0.06 bc	43.52 ± 1.92 bcd	5.52 ± 0.37 b	19.87 ± 0.29 c	38 ± 4 bcde	6.9 ± 1.0 d
P-value	Hydration	0.73	0.02	0.48	0.23	0.01	0.00
	Hydrocolloid	0.00	0.00	0.01	0.00	0.00	0.09

Hydration	Hydrocolloid	Texture profile analysis (TPA)			
		Hardness (g)	Adhesiveness (g·s)	Chewiness (g)	Resilience
Constant	None	8758 ± 534 a	93 ± 20 c	798 ± 144 a	0.19 ± 0.01 a
	Guar gum	12765 ± 658 de	39 ± 4 a	2086 ± 397 cde	0.25 ± 0.01 e
	Xanthan gum	10711 ± 559 b	114 ± 28 d	1433 ± 271 abc	0.19 ± 0.01 a
	Inulin	11920 ± 493 c	35 ± 5 a	2020 ± 563 cde	0.23 ± 0.01 cd
	CMC A15	13377 ± 652 e	22 ± 2 a	2495 ± 531 e	0.27 ± 0.02 f
	CMC A4M	10447 ± 678 b	83 ± 8 bc	1918 ± 666 cde	0.22 ± 0.01 bc
Adapted	None	8758 ± 534 a	93 ± 20 c	798 ± 144 a	0.19 ± 0.01 a
	Guar gum	10986 ± 767 b	79 ± 12 bc	2397 ± 663 cde	0.22 ± 0.02 bcd
	Xanthan gum	10349 ± 795 b	70 ± 7 b	1285 ± 244 ab	0.21 ± 0.01 b
	Inulin	11920 ± 493 c	35 ± 5 a	2020 ± 563 cde	0.23 ± 0.01 cd
	CMC A15	12190 ± 621 cd	26 ± 4 a	1837 ± 208 bcd	0.24 ± 0.02 de
	CMC A4M	10617 ± 662 b	38 ± 4 a	2027 ± 366 cde	0.24 ± 0.01 de
P-value	Hydration	0.01	0.19	0.61	0.41
	Hydrocolloid	0.00	0.00	0.00	0.00

*means with different letters within the same parameter differ significantly ($P < 0.05$).

Table 3
Quality indicators, color and textural properties of cooked Tiger nut gluten free noodles.

Hydration	Hydrocolloid	Cooking quality indicators				Color		
		OCT (min)	CL (g/100 g)	WA (g/100 g)	SI (g/g)	L^*	a^*	b^*
Constant	None	4.75 ± 0.35 a	18.38 ± 0.32 f	82.12 ± 2.82 a	1.43 ± 0.08 bc	47.70 ± 1.71 cde	3.56 ± 0.44 bc	14.37 ± 0.42 a
	Guar gum	5.75 ± 0.35 b	17.96 ± 0.82 ef	110.73 ± 3.43 f	1.28 ± 0.15 a	46.31 ± 1.47 abc	4.08 ± 0.45 d	15.40 ± 0.35 b
	Xanthan gum	6.00 ± 0.00 b	13.68 ± 0.43 b	94.47 ± 2.77 cd	1.22 ± 0.06 a	48.58 ± 1.43 de	4.03 ± 0.42 d	15.58 ± 0.28 b
	Inulin	5.75 ± 0.35 b	16.52 ± 0.29 d	105.50 ± 7.15 ef	1.50 ± 0.06 c	50.25 ± 1.09 f	4.99 ± 0.10 e	17.59 ± 0.36 d
	CMC A15	5.50 ± 0.00 b	15.38 ± 0.69 c	103.45 ± 1.72 ef	1.22 ± 0.09 a	49.24 ± 1.25 ef	3.95 ± 0.44 cd	16.73 ± 0.36 c
	CMC A4M	5.50 ± 0.00 b	14.65 ± 0.95 c	93.68 ± 5.39 bc	1.22 ± 0.09 a	47.60 ± 0.52 bcd	3.91 ± 0.42 cd	17.07 ± 0.62 c
Adapted	None	4.75 ± 0.35 a	18.38 ± 0.32 f	82.12 ± 2.82 a	1.43 ± 0.08 bc	47.70 ± 1.71 cde	3.56 ± 0.44 bc	14.37 ± 0.42 a
	Guar gum	5.75 ± 0.35 b	13.06 ± 0.40 b	87.56 ± 6.75 ab	1.24 ± 0.06 a	46.05 ± 0.88 ab	3.57 ± 0.27 bc	15.36 ± 0.39 b
	Xanthan gum	6.00 ± 0.00 b	12.04 ± 0.70 a	101.37 ± 2.90 de	1.34 ± 0.08 ab	46.44 ± 0.92 abc	3.25 ± 0.30 ab	14.58 ± 0.18 a
	Inulin	5.75 ± 0.35 b	16.52 ± 0.29 d	105.50 ± 7.15 ef	1.50 ± 0.06 c	50.25 ± 1.09 f	4.99 ± 0.10 e	17.59 ± 0.36 d
	CMC A15	5.50 ± 0.00 b	17.43 ± 0.41 de	92.04 ± 2.21 bc	1.29 ± 0.02 a	45.32 ± 1.29 a	3.04 ± 0.23 a	14.40 ± 0.40 a
	CMC A4M	5.50 ± 0.00 b	14.82 ± 0.57 c	95.81 ± 0.93 cd	1.33 ± 0.04 ab	46.80 ± 0.96 abc	3.43 ± 0.38 ab	15.11 ± 0.15 b
P-value	Hydration	1.00	0.12	0.07	0.08	0.00	0.00	0.00
	Hydrocolloid	0.00	0.00	0.00	0.04	0.00	0.00	0.00

Hydration	Hydrocolloid	Texture profile analysis (TPA)				Firmness	
		Hardness (g)	Adhesiveness (g·s)	Chewiness (g)	Resilience	Firmness (g)	Work of Shear (g·cm)
Constant	None	6556 ± 543 cde	96 ± 6 a	905 ± 80 c	0.15 ± 0.01 b	17.7 ± 1.4 a	2.1 ± 0.4 a
	Guar gum	5375 ± 907 ab	128 ± 2 d	600 ± 187 ab	0.12 ± 0.01 a	24.0 ± 0.5 bc	3.1 ± 0.2 ab
	Xanthan gum	7205 ± 1129 e	120 ± 9 cd	2132 ± 526 e	0.14 ± 0.01 b	30.3 ± 0.6 e	4.6 ± 0.3 c
	Inulin	6744 ± 537 cde	103 ± 12 abc	832 ± 24 bc	0.14 ± 0.01 b	22.1 ± 2.5 b	2.4 ± 0.3 a
	CMC A15	5289 ± 936 a	99 ± 13 ab	507 ± 88 ab	0.12 ± 0.02 a	25.5 ± 1.3 c	2.5 ± 0.4 a
	CMC A4M	7243 ± 638 e	122 ± 9 d	981 ± 134 cd	0.15 ± 0.01 b	26.1 ± 0.5 cd	2.3 ± 0.3 a
Adapted	None	6556 ± 543 cde	96 ± 6 a	905 ± 80 c	0.15 ± 0.01 b	17.7 ± 1.4 a	2.1 ± 0.4 a
	Guar gum	7439 ± 739 e	157 ± 8 e	1261 ± 362 d	0.14 ± 0.01 b	26.4 ± 1.8 cd	2.8 ± 0.5 a
	Xanthan gum	7084 ± 227 de	148 ± 14 e	959 ± 196 c	0.14 ± 0.01 b	33.9 ± 0.6 f	3.6 ± 0.3 b
	Inulin	6744 ± 537 cde	103 ± 12 abc	832 ± 24 bc	0.14 ± 0.01 b	22.1 ± 2.5 b	2.4 ± 0.3 a
	CMC A15	5965 ± 481 abc	116 ± 10 bcd	865 ± 47 bc	0.14 ± 0.01 b	29.5 ± 3.0 e	2.5 ± 0.2 a
	CMC A4M	6199 ± 789 bcd	148 ± 17 e	999 ± 298 cd	0.14 ± 0.01 b	28.2 ± 0.4 de	3.0 ± 0.2 ab
P-value	Hydration	0.23	0.00	0.78	0.03	0.00	0.76
	Hydrocolloid	0.01	0.00	0.00	0.01	0.00	0.00

*means with different letters within the same parameter differ significantly ($P < 0.05$).

type of hydrocolloid added, because they play a crucial role on the dough rheology and the quality of fresh and cooked noodles. Hydration applied during noodle processing significantly affected the dough rheology during mixing, heating and cooling, but also the luminosity, hardness and firmness of fresh noodles and the color and firmness of cooked noodles. Instead, hydrocolloids type affected properties of both fresh and cooked noodles. Overall, gluten free TN noodles made with xanthan gum and adapted amount of water showed the best quality considering the lowest cooking losses obtained and its higher firmness.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodhyd.2019.105194.

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foods

Article

Tiger Nut (*Cyperus esculentus*) as a Functional Ingredient in Gluten-Free Extruded Snacks

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Abstract: Tiger nut (TN) is a nutritious source of gluten-free flour, used generally in healthy beverages, but its incorporation in gluten-free extruded snacks has not been explored. TN flour was blended at different concentrations (up to 70%) with rice flour and soluble fiber, for the development of gluten-free snacks on a twin-screw extruder. The effect of TN inclusion in the formulations was evaluated on relevant physiochemical characteristics of the snacks. Viscoamylograph of the raw formulations showed that TN addition increased ($p < 0.01$) onset temperature and delayed peak viscosity. In the extruded flours, TN contributed to limit the starch degradation during extrusion. Diameter, expansion ratio, true density, and total pore volume of the extrudates were reduced ($pf < 0.01$) by the increased TN content in the formulations, while bulk density rose. The surfaces of the extruded snacks were modified by the increasing inclusion of TN in substitution of rice in the formulations. Extrudates containing 10% TN showed the best overall texture profile. Moreover, TN addition enhanced the ash and protein content of the snacks and increased their total antioxidant activity. This study demonstrated that incorporation of 10% TN flour into rice-based formulation was suitable for making gluten-free snacks with acceptable physical properties.

Keywords: tiger nut; rice flour; gluten-free; snacks; extrusion

1. Introduction

In the last twenty years, patients diagnosed with celiac disease have represented an important public health problem [1]. This event has prompted an increase in demand for gluten-free (GF) food products by consumers with and without celiac disease. In fact, many non-celiac individuals wrongly believe that a GF diet is an essentially healthier choice [2]. The market demand of GF foods has promoted the need for new research to develop products using GF agricultural commodities and novel technologies. Extrusion cooking is a versatile technology that has found dominant uses in the cereal and pet food industries as well as in dairy, bakery, and confections industries. In general, the final extrudate has low moisture content and considered a shelf-stable food product [3]. Extrusion process promotes starch gelatinization, protein denaturation, lipid oxidation and the formation of new complexes occur as result of macromolecules interaction which contribute to changes in microstructure [4] and color [5]. In the last decade, studies have been carried out to develop expanded GF snacks, made mainly from cereal-based mixes, to provide nutritious GF foods to consumers inflicted with celiac disease [6,7]. Most recently, other flour mixes containing unconventional crops and agriculture by-products, such as passion fruit shell, and rice flours [8], plantain and chickpea flours in a corn-based mix [6], blends of apple pomace, corn and sorghum [7] and amaranth, quinoa and kañiwa [9] were also evaluated for the development of GF foods. The interest in alternative agricultural commodities is still increasing.

Tiger nut (*Cyperus esculentus*) (TN) is an underutilized crop with great potential for the development of a variety of value-added, nutritious GF foods, including ready-to eat extruded snack-type products. TN is a tuber generally cultivated in the Eastern region of Spain and Western part of Africa [10]. Their rhizomes are used in some countries for human consumption in various forms. In Spain, TN is only grown and processed into a popular drink called "horchata de chufa" in the province of Valencia. "Horchata" is also gaining popularity in other countries due to numerous health related benefits. The composition of TN is characterized by high contents of insoluble fiber and unsaturated fat and with relatively low concentration of starch [11]. Literature on the use of TN in GF food formulation is limited. Aguilar et al. [12], used a combination of TN and chickpea flour to replace emulsifier and/or shortening partially or totally in GF batters or doughs and breads formulations, and reported acceptable specific volume and darker crust in the GF products. Demirkesen et al. [13] reported that TN flour added up to 25% to the rice flour increased the gelatinization temperatures in GF bread, while a significant reduction of the onset gelatinization temperature and peak temperature was observed when TN flour was added to corn-based biscuits [14]. Gasparre et al. [10], reported the effect of a selection of hydrocolloids on the dough rheology, texture properties and cooking performances of GF noodles made by TN. Only one study has reported using TN as food ingredient to produce extrudates [15]. The authors found the mixes of TN-cassava used in their study difficult to extrude as the TN concentration increased in the mixes, due to the high content of insoluble fiber and fat present in TN [16], that caused pressure drop in the extruder barrel and reduced expansion. Moreover, focusing on the sensory quality of GF cereal-based foodstuffs, TN incorporation (20%) produced the highest overall acceptability scores of corn-made biscuits [17]. It is known that insoluble fiber reduce expansion, while soluble fiber tends to increase the expansion of the extrudate [18], and that fat content can cause slippage of melt into the barrel with consequent low pressure at the die exit [19]. The aim of the present study was to develop GF extruded snacks from TN flour/rice flour mixes and the evaluation of nutritional, physical, and microstructural qualities of the extrudates.

2. Materials and Methods

2.1. Raw Materials

Tiger nut (TN) flour, short grain rice flour (Koshihikari, amylose: 17.6%) and Nutriose® were used for making the extruded snacks. TN flour had the following proximate composition: 26.3% fat 18.3% fiber and 8.5% of protein, as declared on the nutritional label, was provided by Mon Orxata (Valencia, Spain). Short grain rice was provided by a local rice grower (Richvale, CA, USA) and Nutriose® FM06, a plant derived soluble fiber (SF) with neutral taste, was supplied by Roquette Company (Geneva, IL, USA).

2.2. Tiger Nut Milling and Blends Preparation

Preliminary studies were carried out to mill the TN into flour by overcoming the problem related to its high fat and fiber content. Two different mills were tested to archive this goal. A laboratory Cyclone mill (Udy Corp., Fort Collins, CO, USA) fitted with a 0.5-mm screen and chilled grinding surface. Then, a comminuting mill fitted with a 3 mm screen (Model D, the Fitzpatrick Company, Chicago, IL, USA), which gave the best result. To further reduce the particle size of the obtained coarse flour and avoid stickiness of the TN to the milling surface, due to its high fat content, the coarse flour mixed with dry ice (3:1) and milled on a Wiley mill (Arthur H. Thomas Co, Philadelphia, PA, USA) fitted with a 2 mm mesh. The resulting flour was a finer flour with a mean particle size below 200 µm. Short grain rice was milled in a pin mill (model 160Z, Micron Hosakawa, Köln, Germany) to a fine flour. TN flour was mixed in increasing proportions, 10%, 30%, 50% and 70% with rice flour and 10% SF, which represented formulations, referred from here on as, R1, R2, R3 and R4, respectively. A mix of 90% rice and 10% SF was used as a control, which allowed increasing the fiber content of the final products. The blends were stored at 4 °C until extrusion cooking was carried out.

2.3. Modified Feeding Device and Extrusion Processing Conditions

2.3.1. Modified Feeding Device

The high fat content in the TN flour made the formulated flours difficult to freely flow through the twin screw of the K-Tron feeder (Model KCL-24-KT-20, K, K-Tron Corp., Pitman, NJ, USA) used in this study, to deliver the correct amount of feed into the extruder. To enable their proper flow, a feeding modification system was built, which consisted of a 4 inches acrylic tube (Tap Plastic, San Leandro, CA, USA) cut lengthwise. The surface of the channel was coated twice with an anti-static solution (food-grade) containing fatty acid in its composition. The made flour conveyor was suspended underneath the feeder discharge chute and adjusted to provide an inclination of about 120 degrees. This modification allowed the proper flow of flour, to the set feed targets of flour, to be delivered into the extruder's feeding port (Figure 1).

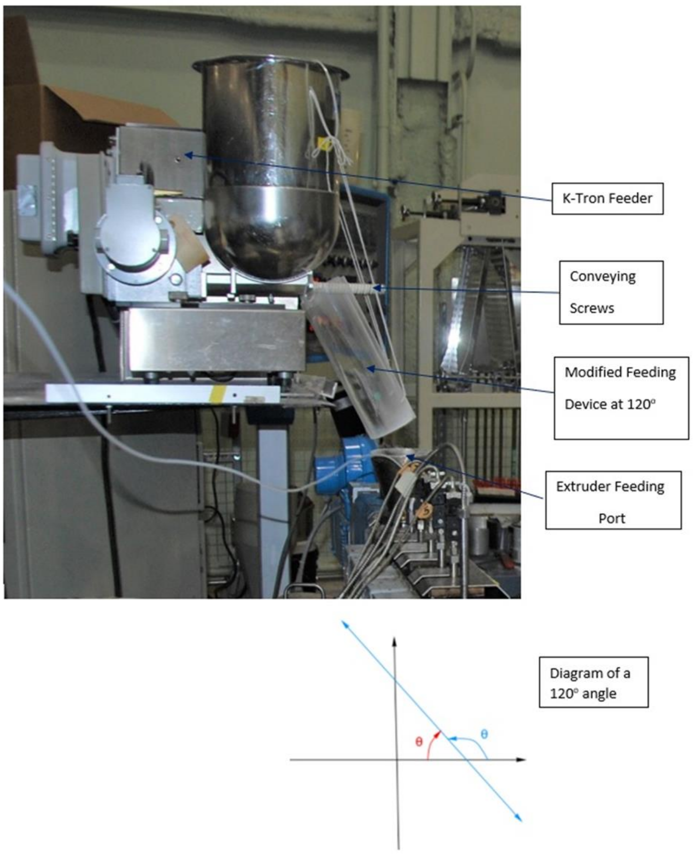

Figure 1. Modified feeding device for suitable conveying formulated flours, containing different levels of tiger nut, into the extruder.

2.3.2. Extrusion Processing Conditions

Extrusion of the formulated flours was performed using the Leistritz 18 mm co-rotating twin-screw extruder (MIC 18/GL 30D, Allendale, NJ, USA), equipped with six heating-cooling barrel zones. The heating profile of the barrel zones were set at the following temperatures: zone 1 cooled with tap water at approximately 25 °C, zone 2 at 60 °C, zone 3 at 80 °C, zone 4 at 100 °C, zone 5 at 100 °C and zone 6 at 120 °C. The first temperature was the temperature of the feeding zone and the last one corresponded to the temperature of the die zone (expansion zone). The die was a circular orifice 3 mm in diameter. During the extrusion process, the feed rate and the screw speed were kept constant at 3 kg/hour and 500 rpm, respectively, based on preliminary testing. During extrusion, the moisture content of the formulations was adjusted to 16% by injecting water through a preparatory HPLC pump (Model 305, Gilson, Middleton, WI, USA). The extrudates obtained were dried at 70 °C in a force air drying oven (Imperial IV, Labline, Melrose Park, IL, USA) to a final moisture content of 6%. Extrudates from formulations containing 10%, 30%, 50% and 70% TN flour were referred as E1, E2, E3 and E4, respectively. The dried extrudates were ground by a laboratory sample mill (Cyclone Mill, Udy Corporation, Fort Collins, CO, USA) fitted with a 0.5 mm mesh screen into fine flour for further evaluation. Figure 2 illustrates the detailed steps in obtaining TN extrudates from the raw ingredients.

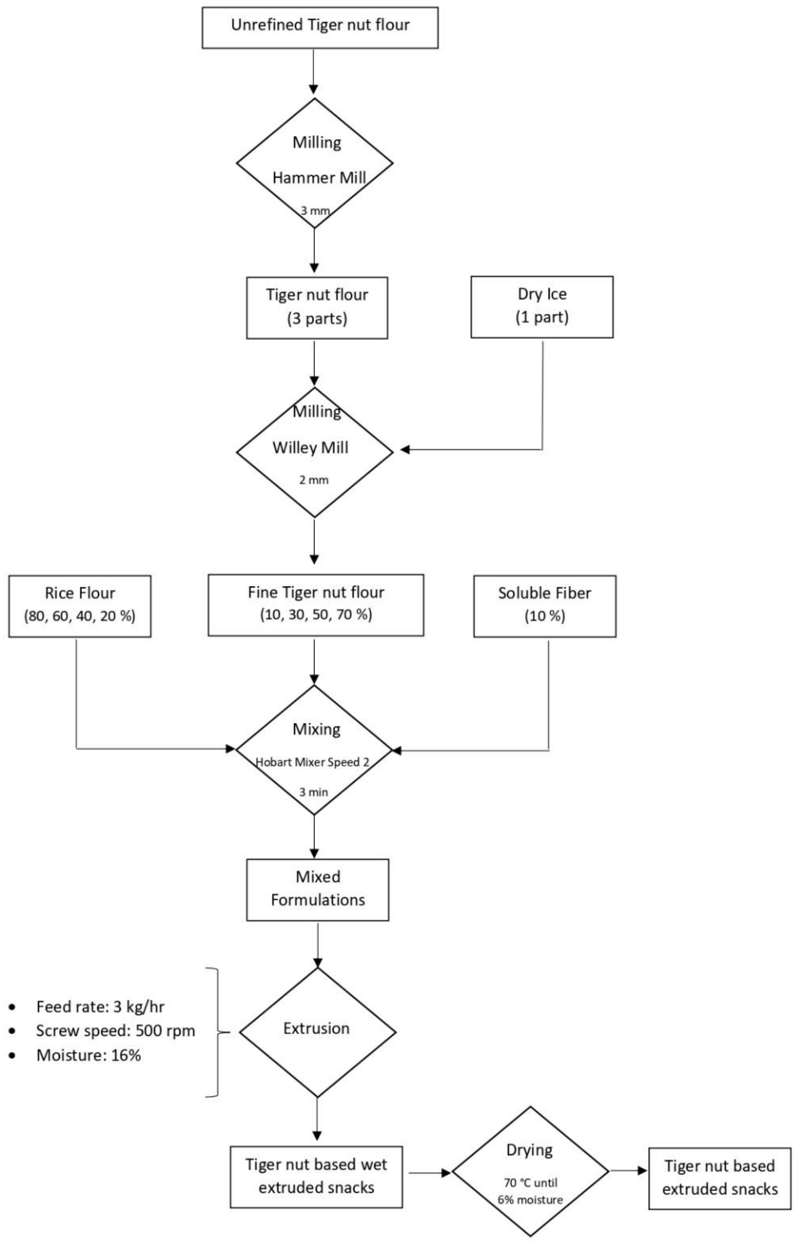

Figure 2. Flowchart of snacks production from tiger nut/rice flour mixture.

2.4. Proximate Analysis

Proximate composition of raw and extruded flours was determined in triplicate according to standard methods of the Association of Official Analytical Chemist AOAC [20]. Total nitrogen was determined by Leco FP628 (Leco Company, St. Joseph, MI, USA), and fat content determined on a Dionex ASE350 solvent extractor (Thermo Fisher Scientific, Inc., Waltham, MA, USA). Carbohydrate content was calculated by difference.

2.5. Apparent Viscosity

Apparent viscosity of the raw blends and extruded flours was determined using a Rapid Viscosity Analyzer (RVA 4500, Perten Instrument, Sydney, NSW, Australia). Samples of 3.50 g and 25.0 mL HPLC-grade water were mixed in an RVA canister and analyzed using the following temperature profiles: equilibrating at 25 °C for 1 min, then the temperature was ramp up to 95 °C over a period of 3 min, and holding at 95 °C for 4 min, followed by a cooling period of 3 min to 25 °C, and hold for 2 min. Thermocline software for Windows (TCW3) was used to calculate the pasting parameters: onset temperature where viscosity start to increase; peak viscosity (Pa·s): the maximum hot paste viscosity; trough viscosity (Pa·s): minimum hot paste viscosity; breakdown (Pa·s): the difference between peak viscosity and trough viscosity; final viscosity (Pa·s): the viscosity at the end of the run; and total setback (Pa·s): the difference between final viscosity and trough viscosity.

2.6. Microstructure Analysis

A scanning electron microscope (SEM) (Model TM300, Hitachi High-Technologies, Tokyo, Japan) at 2 kV accelerating voltage and at magnification of 100× was used for microstructural analysis. Extrudates were dried overnight in a desiccator with $CaSO_4$ prior to analysis. Colloidal graphite cement was applied around the bottom of the extrudates for a better conductivity and covered with approximately 30 nm of gold using a Technics Hummer V sputter coater. Stereoscopic pictures were taken using a stereoscopic microscope (Leica Microsystems Inc., Buffalo Grove, IL, USA.) at 1× magnification.

2.7. Final Products Quality Parameters

Expansion ratio (ER) of the extrudates was evaluated according to the method described by Berrios et al. [21] and calculated as followed:

$$\text{ER (mm)} = \text{Area of extruded road (mm}^2\text{)} / \text{Area of circular die hole (mm}^2\text{)} \quad (1)$$

The bulk density (BD) of each of the extrudates was determined based on a volumetric displacement method, using glass beads with a diameter of 2 mm as a displacement medium, with some modifications as previously explained by Patil et al. [22], who standardized this measurement with optimum sample size. The values employed were obtained by averaging five measurements of the extrudates.

$$\text{BD (g/cm}^3\text{)} = \text{Extrudate mass (g)} / \text{Extrudate volume (cm}^3\text{)} \quad (2)$$

True density of extrudates was measured with a pycnometer (AccuPyc II 1340, Micromeritics, Norcross, GA, USA) using a small sample holder with a volume of 350 cm^3. Helium was used as a volume displacement medium. To calculate sample volume and total pore volume, the pressure before and after expansion was measured. The analysis was carried out in triplicate for each sample.

A texture analysis of the extrudates was carried out using a texture analyzer (TA-XTPlus, Stable Micro System, Godalming, Surrey, UK) calibrated with a 2 kg mass, to determine the firmness and shear force of the samples by compression. A 3-point band test was used on individual extruded rods. A TA92A probe was set to press the extruded rod placed on a metal platform to a depth of 15%, with testing speed of 1 mm/s. The return distance and return speed were adjusted to 35 mm/s and 10 mm/s, respectively. A total of 20 measurements were performed for each sample. Firmness was defined as the peak force of the first compression required for the sample to rupture and the shear force was calculated as the area under the curve from the force/time graph.

2.8. Total Soluble Phenolics and Total Antioxidant Capacity of Final Products

Total soluble phenolics (TSP) were determined according to the Folin–Ciocalteu spectrophotometric method, with a slight modification to the method as described by Nayak et al. [23]. About 5 g of extruded flour samples were extracted with 20 mL of methanol at room temperature (25 °C) for 24 h. About 0.15 mL of Folin–Ciocalteu reagent was added to the extract (0.3 mL of aliquot). The mixture was set aside to equilibrate for 3 min and then mixed with 0.3 mL sodium carbonate. Subsequently, incubated at room temperature for 60 min, and absorbance of the mixture was read at 765 nm with a benchtop spectrophotometer (PharmaSpec UV-1700, Shimadzu Scientific Instruments, Inc., Kyoto, Japan). Methanol was used as a blank. TSP content was quantified from a gallic acid standard curve developed from 0–0.125 mg of gallic acid per mL and expressed as micrograms of gallic acid equivalent per milligrams of dry weight sample (μg GAE/mg DW). The analysis was carried out in triplicates.

Total antioxidant capacity (TAC) of extruded samples was measured using an adapted method of Patel et al. [24]. The methanol extracts from the TSP were used in this evaluation. A total of 0.5 mL of sample solution was reacted with 2.95 mL of 2,2-diphenyl-1-picrylhydrazyl (DPPH) (103.2 μM in methanol, absorbance of ~1.2 at 515 nm) on a covered shaker at room temperature for 20 h, and spectrophotometer was blanked with methanol. The absorbance was read at 515 nm. TAC was quantified from a Trolox standard curve developed from 0 to 750 μg of Trolox per mL and expressed as micrograms of Trolox equivalent per gram of dry weight sample (μg TE/g DW). The analysis was carried out in triplicates.

2.9. Statistical Analysis

Data were analyzed by multifactor analysis of variance (MANOVA) using Statgraphics Centurion XVII software (Statpoint Technologies, Warrenton, VA, USA). Fisher's least significant differences test was applied for the determination of significant differences among experimental mean values, with 95% confidence.

3. Results and Discussion

3.1. Raw Blends Characteristics

The moisture and carbohydrate content of raw blends decreased significantly ($p < 0.01$) whereas the protein, fat and ash contents increased ($p < 0.01$) as the TN content was increased (Table 1). Protein, fat and ash content significantly ($p < 0.01$) increased with the TN level substitution. In fact, this trend is attributed to a higher protein, fat and ash content of TN. The same trend was observed in the study by Kareem et al. [15], where the addition of TN (up to 100%) to a mixture of cassava and spices greatly enhanced the ash, protein, and fat content of the raw blends.

The Rapid Visco Analyzer (RVA) was used to visualize the physical transition points by recording the apparent viscosity of the formulated flours. Figure 3 depicts the viscoamylographs of the raw blends. Attributes determine from the recorded apparent viscosity are exhibited in Table 1, and the presence of TN was identified as main factor of variance. Statistical analysis indicated that TN incorporation in the formulations significantly ($p < 0.01$) increased the onset temperature for samples R3 and R4 (Table 1). The blend with the highest TN replacement level (R4) required more time to achieve the maximum peak viscosity ($p < 0.01$). This delay may be caused by the presence of more quantity of fiber and fat that could limit the water required for starch swelling. TN flour produced a significant ($p < 0.01$) reduction in the peak viscosity, trough viscosity, breakdown, final viscosity, and total setback. These outcomes were related with the lower starch content in the blends with TN flour that caused a viscosity drop in the mixture, which might constraint the further expansion of the extrudates. A similar trend was observed in a previous study by Adegunwa et al. [25] when TN was added to plantain flour.

Table 1. Proximate composition and apparent viscosity of raw blends containing increasing concentrations of tiger nut flour (R1: 10%; R2: 30%; R3: 50%; R4: 70%).

Parameters	Raw Blends				
	Control R	R1	R2	R3	R4
Proximate Composition					
Moisture (g/100 g)	11.76 ± 0.12 [e]	11.52 ± 0.06 [d]	10.66 ± 0.09 [c]	9.58 ± 0.08 [b]	8.72 ± 0.04 [a]
Protein (g/100 g)	5.02 ± 0.07 [a]	5.34 ± 0.08 [b]	5.71 ± 0.12 [c]	6.08 ± 0.10 [d]	6.30 ± 0.03 [e]
Fat (g/100 g)	0.28 ± 0.01 [a]	3.46 ± 0.08 [b]	9.06 ± 0.00 [c]	15.08 ± 0.06 [d]	20.45 ± 0.19 [e]
Ash (g/100 g)	0.36 ± 0.01 [a]	0.51 ± 0.00 [b]	0.80 ± 0.00 [c]	1.15 ± 0.03 [d]	1.46 ± 0.02 [e]
Carbohydrates (g/100 g)	82.58	79.17	73.77	68.11	63.07
Apparent Viscosity					
Onset temperature (°C)	70.3 ± 0.9 [a]	70.9 ± 0.8 [a]	71.9 ± 0.4 [a]	75.1 ± 1.3 [b]	78.2 ± 1.1 [b]
Peak Time (min)	5.7 ± 0.0 [a]	5.8 ± 0.1 [a]	5.8 ± 0.0 [a]	5.8 ± 0.1 [a]	7.1 ± 0.0 [b]
Peak viscosity (mPa·s)	3980 ± 70 [e]	3040 ± 60 [d]	1630 ± 20 [c]	710 ± 10 [b]	290 ± 0 [a]
Trough viscosity (mPa·s)	2130 ± 30 [e]	1750 ± 100 [d]	1080 ± 10 [c]	630 ± 0 [b]	280 ± 0 [a]
Breakdown (mPa·s)	1850 ± 40 [e]	1300 ± 40 [d]	550 ± 10 [c]	90 ± 10 [b]	10 ± 0 [a]
Final viscosity (mPa·s)	3140 ± 190 [d]	2900 ± 100 [d]	2100 ± 0 [c]	1400 ± 20 [b]	770 ± 10 [a]
Total Setback (mPa·s)	1010 ± 220 [bc]	1160 ± 0 [bc]	1030 ± 10 [c]	780 ± 10 [b]	480 ± 10 [a]

Means with different letters within the same parameter differ significantly ($p < 0.01$).

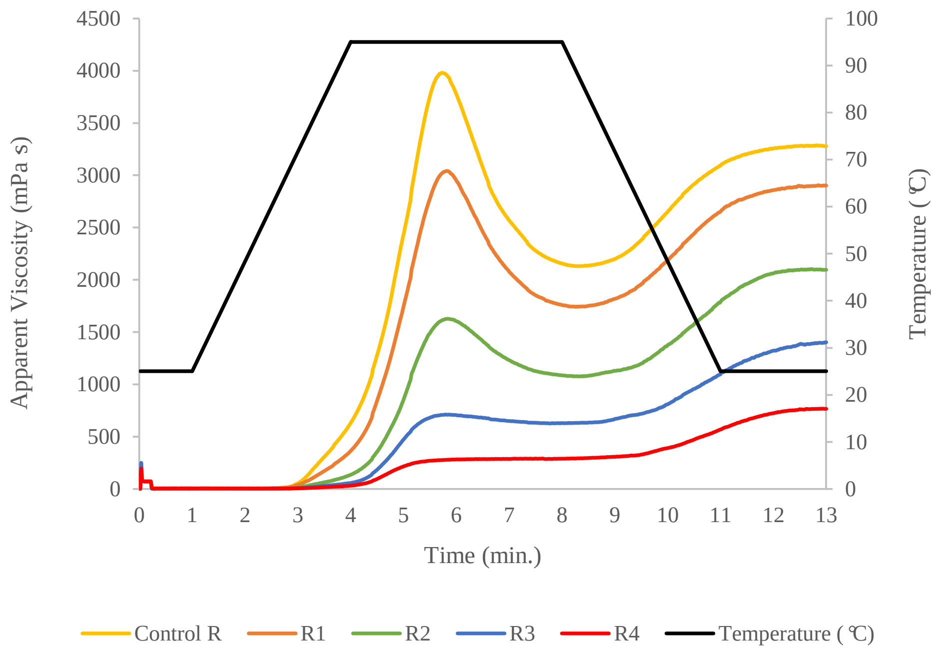

Figure 3. Rapid Visco Analyzer profiles of formulated unprocessed flours containing different concentration of tiger nut flour.

3.2. Extruded Products Characteristics

Expansion of the extrudates was significantly affected by the level of TN, as it can be observed in the surface and cross section images (Figure 4). The control rice extrudate showed a corrugated, cohesive overlapping surface that was more visible in the SEM micrograph, which is attributed to the gelatinization starch and expansion of the product during the extrusion process. The cross-section of the control sample (Figure 4, Control E), which had the greater expansion, depicted larger air pockets than samples containing TN. The extrudate containing 10% TN in the formulation (Figure 4, E1), presented a smoother surface with less obvious grooves, with a reduced diameter and expansion ratio; this is due to a content reduction of starchy rice as a result of high fat TN flour incorporation. With increasing TN concentration up to 30% in the formulations, the cohesiveness and continuity of the structural surface of the extrudates are disrupted and more uneven surfaces with porous structures were observed, particularly in SEM micrograph (Figure 4, E2). This disruption is more noticeable with the sample containing 50% TN (Figure 4, E3), which presents a coarser surface and particle disaggregation. A reduction in starch concentration as the TN was increased in the formulations mostly induced a breakage of the continuous matrix in the extrudates, rendering products with less expansion and surface uniformity. Rupture and non-uniform surface have also been reported as negative quality attributes determined on extruded cornstarch when adding wheat fiber [26]. The surface appearance of extrudates containing the highest content of TN of 70% (Figure 4, E4) displayed irregular areas with an alternating smooth, oily-looking appearance. The high fat content in this sample could have acted as a plasticizer, which was observed in the SEM image, resulting in a product with the indicated distinctive surface characteristics. This result corroborates with the compact, unexpanded cross-sectional view of this extrudate (Figure 4, E4), confirming the significant effect of the inclusion of the TN flour in the structural and surface characteristics of the developed snacks. Apart from that, the compactness above described might be also linked to the higher protein and fiber content. During extrusion, protein and fiber could behave as a dispersed phase within the continuous starch arrangement that causes an interruption of the cell wall formation [27]. Furthermore, the covalent and nonbonding interactions between protein leads to the formation of a network. This protein system may influence the water distribution in the matrix causing changes of the extensional properties of the melt with a consequent density increase of the final extrudates [28].

Figure 5 depicts profiles of extrudates characterized by lower viscosities than those obtained for the raw blends. Indeed, heating and mechanical shearing applied during the extrusion process caused starch gelatinization. It is known that heating and mechanical shearing cause the fragmentation of amylopectin and amylose structure, thereby reducing their absorbing and swelling capacity in starchy materials [29], which was reflected in a viscosity reduction found in this study. Moreover, it was determined that the apparent viscosity was extremely low in the control and E1 extrudate containing 10% TN, than extrudates E2 and E3 containing 30% and 50% TN, respectively (Figure 5). This could be due to the "protective" effect of fat on the starch granules [30], that kept some ungelatinized granules which contribute to increase the apparent viscosity. Another explanation may be related with the starch-protein interactions that limit the starch swelling and the changes associated to its gelatinization [31]. In this case, protein absorb more water, making it less available for the complete starch gelatinization. Viscosity of extrudate E4 with 70% TN did not follow the indicated pattern, because of the greater substitution, most of or all of the starch got gelatinized and dextrinized during the extrusion process. As shown in Table 2, TN flour addition significantly ($p < 0.01$) increased the onset temperature. In this regard, formulation E4 with the highest amount of TN presented the highest onset temperature. The delay in the peak viscosity caused by TN incorporation, observed in the raw blends, was also observed when analyzing the extruded samples, while peak viscosity, trough viscosity, final viscosity and total setback showed a completely different pattern. In general, except for extrudate sample E4, the content of TN in the extruded products corresponded with a significant ($p < 0.01$) increase in the apparent viscosity, despite the reduced starch content. This may be due to the protective effect exercised by the fat molecules that could arrange themselves around the starch granules mitigating the degradation effects of the heating and mechanical shearing.

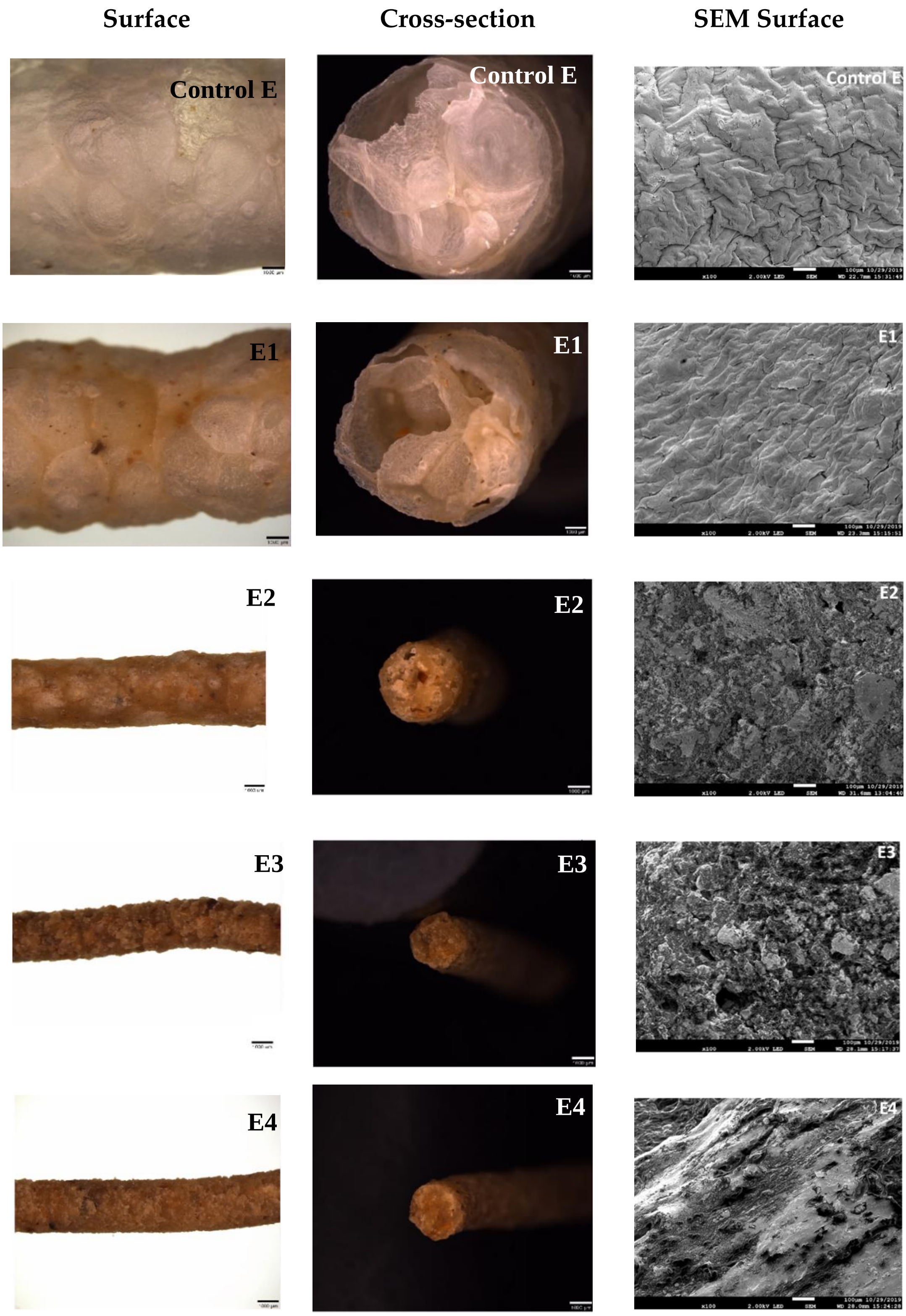

Figure 4. Stereoscopic images of surface and cross-sectional areas (1× magnification) and SEM micrographs (100× magnification) of the surface area of extruded samples containing different levels of tiger nut. Bottom bars in the pictures are referred to magnification, which is described above for better understanding.

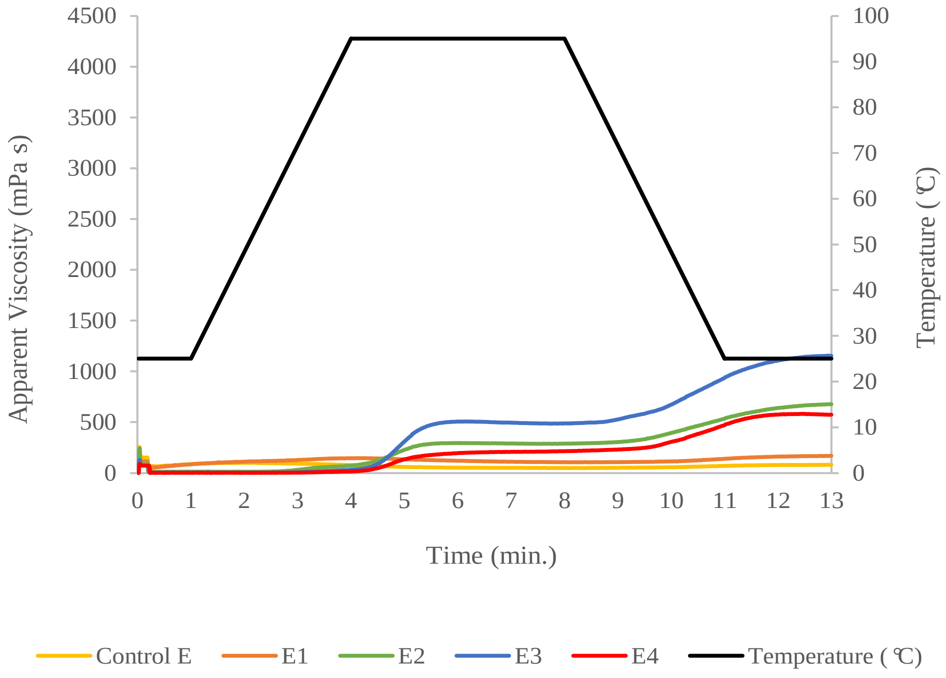

Figure 5. Rapid Visco Analyzer profiles of formulated extruded samples containing different levels of tiger nut flour.

Table 2. Main physical and nutritional characteristics of extruded samples made from formulations with increasing concentrations of tiger nut flour (E1: 10%; E2: 30%; E3: 50%; E4: 70%).

Parameters	Extruded Samples				
	Control E	E1	E2	E3	E4
Apparent Viscosity					
Onset temperature (°C)	50.5 ± 0.0 [a]	50.4 ± 0.1 [a]	70.4 ± 0.5 [b]	79.9 ± 2.5 [c]	87.2 ± 0.6 [d]
Peak Time (min)	2.2 ± 0.0 [a]	4.2 ± 0.2 [b]	6.5 ± 0.2 [c]	6.2 ± 0.0 [c]	7.15 ± 0.1 [d]
Peak viscosity (mPa·s)	100 ± 10 [a]	150 ± 0 [b]	300 ± 0 [d]	510 ± 10 [e]	210 ± 0 [c]
Trough viscosity (mPa·s)	50 ± 0 [a]	100 ± 0 [b]	290 ± 0 [d]	480 ± 10 [e]	210 ± 0 [c]
Breakdown (mPa·s)	50 ± 0 [e]	40 ± 0 [d]	10 ± 0 [b]	20 ± 0 [c]	0 ± 0 [a]
Final viscosity (mPa·s)	80 ± 0 [a]	170 ± 0 [b]	680 ± 0 [d]	1160 ± 10 [e]	580 ± 0 [c]
Total Setback (mPa·s)	30 ± 0 [a]	70 ± 0 [b]	390 ± 0 [d]	670 ± 0 [e]	370 ± 0 [c]
Quality Parameters					
Diameter (mm)	10.19 ± 0.28 [d]	9.27 ± 0.27 [c]	3.46 ± 0.12 [b]	2.53 ± 0.03 [a]	2.51 ± 0.03 [a]
Expansion ratio	16.63 ± 0.92 [d]	13.78 ± 0.81 [c]	1.92 ± 0.14 [b]	1.02 ± 0.02 [a]	1.01 ± 0.02 [a]
Bulk density (g/cm^3)	0.19 ± 0.02 [a]	0.21 ± 0.01 [a]	0.62 ± 0.01 [b]	0.69 ± 0.02 [c]	0.65 ± 0.02 [b]
True density (g/cm^3)	1.52 ± 0.01 [d]	1.54 ± 0.01 [e]	1.50 ± 0.01 [c]	1.44 ± 0.01 [b]	1.35 ± 0.00 [a]
Total Pore volume (cm^3/g)	0.34 ± 0.01 [d]	0.35 ± 0.00 [e]	0.33 ± 0.01 [c]	0.30 ± 0.01 [b]	0.26 ± 0.00 [a]
Firmness (g)	712 ± 146 [c]	922 ± 261 [d]	242 ± 52 [a]	367 ± 39 [b]	313 ± 73 [ab]
Work of Shear (g·s)	11 ± 3 [b]	19 ± 6 [c]	2 ± 1 [a]	4 ± 1 [a]	4 ± 1 [a]
Proximate Composition					
Moisture (g/100 g)	6.67 ± 0.07 [b]	6.12 ± 0.04 [a]	6.14 ± 0.06 [a]	6.20 ± 0.05 [a]	6.24 ± 0.02 [a]
Protein (g/100 g)	4.65 ± 0.13 [a]	4.87 ± 0.07 [b]	5.25 ± 0.09 [c]	5.57 ± 0.06 [d]	5.83 ± 0.04 [e]
Fat (g/100 g)	0.05 ± 0.01 [a]	1.47 ± 0.01 [b]	6.62 ± 0.02 [c]	14.11 ± 0.04 [d]	19.32 ± 0.14 [e]
Ash (g/100 g)	0.36 ± 0.00 [a]	0.67 ± 0.02 [b]	0.93 ± 0.01 [c]	1.22 ± 0.02 [d]	1.52 ± 0.01 [e]
Carbohydrates (g/100 g)	88.26	86.87	81.07	72.90	67.09
Total Soluble Phenolics Gallic acid Equivalent (µg/mg)	10 ± 0.00 [a]	110 ± 0.00 [b]	240 ± 0.01 [c]	550 ± 0.02 [d]	720 ± 0.03 [e]
Antioxidant Capacity Trolox Equivalent (µg/g)	533 ± 17 [a]	596 ± 15 [b]	730 ± 10 [c]	852 ± 10 [d]	937 ± 9 [e]

Means with different letters within the same parameter differ significantly ($p < 0.01$).

The data in Table 2 indicate that the addition of TN in the formulations produced significant ($p < 0.01$) changes in the developed extrudates. The diameters, expansion ratios, true densities, and total pore volumes progressively decreased ($p < 0.01$) as a consequence of the increased content of TN in the samples. Formulations E3 and E4 recorded the lowest values in terms of diameter and expansion ratio, as shown in Figure 4 and Table 2. The reduction in expansion with the progressively higher inclusion of TN in the samples might be attributed to a reduction in the starch content and an increase in the insoluble fiber [18], fat and protein, which are known as factors that negatively correlate with expansion [32]. In literature, studies agree with the results presented in this work, the expansion ratio decreased when TN flour was blended with rice flour [15] and also when lentil [33], mango peel [34], and partially defatted hazelnut flour, high in fiber and protein [35] were used in combination with corn flour and rice grits, respectively. Berrios et al. [21] have previously indicated that bulk density was inversely related to expansion ratio, which agrees with the results from this study. Overall, with the increase of the TN flour in the formulation a rise of the bulk density of the extrudates was observed. In particular, between the control and sample E1 there was a slight increase that was even more pronounced between the samples E2 and E4, most likely due to the excessive fat content in those samples containing more TN in their formulation. A similar trend in BD was observed by previous researchers in extrudates made with corn and soybean hull [36]. The determination of true density and porosity of the extrudates provided additional quality parameters on the properties of the developed products, as these parameters provide insight into the structural properties of the dried materials. Addition of TN significantly reduced ($p < 0.01$) the true density of the extrudates. A similar trend was observed for the total pore volume of extrudates with higher inclusion of TN in their formulation (Table 2). During extrusion, as the melt exit the die, numerous small air cells are generated by the rapid release of the high pressure [26]. The pressure difference out of the die causes the water flash off with the formation of internal pores of varying sizes that are responsible for the product expansion. The high fat content in samples with the greatest inclusion of TN in their formulation, decreased the melt viscosity of the extruded material, which caused a reduction in die pressure, resulting in less expanded products with higher bulk densities (Table 2) [37]. This phenomenon is clearly shown in Figure 4. Extrudate control E had larger diameter and more air pockets, while sample E4, with the highest amount of TN, showed a cross-section devoid of air pockets or porous structure. Internal microstructure of the extrudates are of important consideration in the production of snack-type products as this structural pore formation is an important quality parameter associated with the crispiness and crunchiness of expanded products [37].

Texture was evaluated by measuring the firmness (force required to break the extruded rod) and work of shear (area under the curve). The results in Table 2 show significant effect ($p < 0.01$) of TN on the texture of the developed extrudates. The sample with 10% of TN presented the highest firmness and shear force values. This textural effect may be due to a higher integration of the matrix components and the amylose-lipid complex formation. In fact, the interaction of fatty acids or long-chain alcohols with amylose double helices forms the amylose-lipid complexes that cause functional modifications in the physical and chemical behavior of the starch [38]. Given that, the interactions between amylose and fatty acids increase the elasticity of the starch matrix, the structure resulted to be more resistant to breakdown [39]. Conversely, both texture parameters were reduced with an increase of TN in the formulations. This could be explained by the reduction of starch content as the addition of TN increased in the formulations, while fat content greatly increased resulting in extrudates with softer and more brittle texture. This same trend was also reported in a previous study on extrudates containing different levels of cassava-TN mixtures [15]. Moreover, the outcomes about the maximum force value from this study (from 242 g to 922 g) are in the same range of those reported by Kareem et al. [15] (from 251 g to 1272 g).

The proximate analysis of the raw blends determined that the TN addition significantly ($p < 0.01$) increased the protein, fat and ash content compared to the control sample with no TN addition. It was also observed that extrusion process reduced the protein and fat amount in the extruded snacks.

Protein losses ranged from 11% to 16% but those were much greater for the fat content. The control suffered the greatest fat loss followed by sample E1 (150%). This reduction of fat most likely occurred at the die opening as free oils [40]. As melt exits the die, a rapid temperature and pressure drop occur, resulting in rapid expansion of the water molecules into steam, and in this research, a small quantity of liquified fat was observed at the die, especially for the samples E4 and E5. Another possible explanation of the fat reduction could be the formation of new complexes with amylose or protein that trap the lipid, making their extraction more difficult with conventional method [40]. After extrusion, all the samples, (except the control) presented a slightly higher ash content with respect to their raw counterparts. From a nutritional standpoint, it is well known that GF foodstuffs are poorer in minerals and protein content, while their saturated fat amount result to be higher compared to their gluten-containing counterparts [41]. TN included in GF free snacks production may represent a valuable alternative when it comes to cover these nutritional deficiencies. Moreover, the lipidic profile mostly characterized by polyunsaturated fatty acids [42], may help to reduce the quantity of the saturated ones. Although, its high fat content imposes a correct evaluation when deciding to employ TN in GF food product development.

3.3. Total Soluble Phenolics and Total Antioxidant Capacity of Extruded Snacks

The total soluble phenolic content and the antioxidant capacity were evaluated to assess the nutritional improvement provided by TN (Table 2). The control sample showed low amount of total soluble phenolics, but it had antioxidant activity, which might be related to the reaction of some peptides or amino acids with the DPPH. There was a significant increase in total soluble phenolic and antioxidant capacity when rice flour was replaced with TN in extruded samples. The TSP in the extrudates varied between 10 to 720 μg GAE/mg DW. Adebowale et al. [43] using a cassava-based formulation containing increasing TN, reported a slightly higher TSP (370 to 890 μg GAE/mg DW values), which might be due to the presence of different spices (onion, ginger, chili pepper) in the formulations. TAC results (Table 2) reflected similar trend as that of TSP. The amount of TAC significantly ($p < 0.01$) increased with addition of TN in the extruded products. Those increases may be related with the presence, in the tiger nut cell wall, of some antioxidant monomeric phenols, such as p-hydroxybenzoic acid, vanillic acid, p-hydroxybenzaldehyde, vanillin, p-trans-coumaric acid, trans-ferulic acid, p-cis-coumaric acid, cis-ferulic acid that may contribute to improve the antioxidant capacity [42]. Another possible explanation may come from the presence of tocopherols in TN that have been described as the most important natural group of antioxidants found in vegetable oils [44].

4. Conclusions

This study presents the potential use of TN to produce novel gluten-free extruded snacks, providing an attractive alternative to consumers with celiac disease. Pasting profile analysis showed that TN inclusion, into the rice-based formulated flours, increased the onset temperature and delayed the peak viscosity while in the extruded flours. Progressive addition of TN in the formulations promoted a reduction in diameter, expansion ratio, true density, and total pore volume in the extrudates, while their bulk densities increased. Furthermore, TN incorporation was responsible for an increase in ash, protein, and total phenol content, which is an added value to the developed snack. This study demonstrated that extrudates with 10% TN in the formulation showed the best overall texture profile. A future study is proposed using specialty starches to further promote expansion of the TN-based snacks.

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