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# BIOACTIVE COMPOUNDS FROM AGRI-FOOD BY-PRODUCTS. EXTRACTION AND APPLICATION IN MICROENCAPSULATION

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Universitat
de les Illes Balears

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Doctoral Programme of Chemical Science and Technology

# BIOACTIVE COMPOUNDS FROM AGRI-FOOD BY-PRODUCTS. EXTRACTION AND APPLICATION IN MICROENCAPSULATION

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# PAPERS LIST

This doctoral thesis titled "**Bioactive compounds from agri-food by-products. Extraction and application in microencapsulation**" whose author is Mónica María Umaña Zamora, is presented as a compendium of papers, listed as follows :

- Umaña, M., Dalmau, M. E., Eim, V. S., Femenia, A., & Rosselló, C. (2019). Effects of acoustic power and pH on pectin-enriched extracts obtained from citrus by-products. Modelling of the extraction process. Journal of the Science of Food and Agriculture, 99(15), 6893–6902. https://doi.org/10.1002/jsfa.9975. Journal impact factor (2019) 2.614. Q1 (8/58) Agriculture, Multidiciplinary.
- Umaña, M., Eim, V., Garau, C., Rosselló, C., & Simal, S. (2020). Ultrasound-assisted extraction of ergosterol and antioxidant components from mushroom by-products and the attainment of a β-glucan rich residue. Food Chemistry, 332, 127390. https://doi.org/10.1016/j.foodchem.2020.127390. Journal impact factor (2019) 6.306. Q1 (6/139) Food science & technology.
- Umaña, M., Turchiuli, C., Eim, V., Rosselló, C., & Simal, S. (2021). Stabilization of oil-in-water emulsions with a mushroom (Agaricus bisporus) by-product. Journal of Food Engineering, 307, 110667. https://doi.org/10.1016/j.jfoodeng.2021.110667. Journal impact factor (2019) 4.499. Q1 (16/139) Food science & technology.
- Umaña, M., Turchiuli, C., Rosselló, C., & Simal, S. (2021). Addition of a mushroom by-product in oil-in-water emulsions for the microencapsulation of sunflower oil by spray drying. Food Chemistry, 343, 128429. https://doi.org/10.1016/j.foodchem.2020.128429. Journal impact factor (2019) 6.306. Q1 (6/139) Food science & technology.
- Umaña, M., Wawrzyniak, P., Rosselló, C., Llavata, B., & Simal, S. Evaluation of the addition of artichoke by-products to O/W emulsions for oil microencapsulation by spray drying. LWT-Food Science and Technology. Under review. Journal impact factor (2019) 4.006. Q1 (28/139) Food science & technology.

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**Annex 3**: Model thesis supervision certificate

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Dra. Carmen Rosselló Matas, of the University of the Balearic Islands

I DECLARE:

That the thesis titles *Bioactive compounds from agri-food by-products. Extraction and application in microencapsulation,* presented by Mónica María Umaña Zamora to obtain a doctoral degree, has been completed under my supervision and meets the requirements to opt for an International Doctorate.

For all intents and purposes, I hereby sign this document.

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Palma de Mallorca, 25 March 2021

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Dra. Susana Simal, of the University of the Balearic Islands

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For all intents and purposes, I hereby sign this document.

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Palma de Mallorca, 25 March 2021

*A mis padres, mis hermanas*

*y al amor de mi vida, Juan*

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## Introduction

# ABSTRACT

The agri-food industry generates a large amount of waste and by-products which creates a serious environmental problem. Hence in 2020, the European Union adopted a circular economy plan that includes food waste reduction as a key undertaking. In this economic model, the products and materials are intended to be kept in use for as long as possible. Thus, the use of agri-food residues as a source of bioactive compounds has recently gained considerable attention. However, the natural resistance of plants and fungi cell walls hinders the solid-liquid extraction of these compounds and has driven the industry to search for new technologies to intensify the process. The application of high-power ultrasound is one of those intensification methods with known advantages, but there is still a need to investigate its effect under different extraction conditions. Moreover, after the extraction of high-value molecules, there remains a significant amount of residue that may still well contain compounds with bioactivity and/or interesting technological properties.

In this context, the general objectives of this thesis were to propose alternatives for the exploitation of agri-food by-products through the extraction of their bioactive compounds using high-power ultrasound; and to explore the application of these by-products and the residues of the extraction process in lipid microencapsulation, taking advantage of their emulsifying and inherent antioxidant capacity, and therefore, proposing an integral valorization.

By-products from three different sources were investigated for this thesis (a fruit, a mushroom, and a vegetable): orange (peel and pulp), mushroom (stalks and residues obtained after bioactive compounds extraction), and artichoke (bracts).

The orange and mushroom by-products were used to perform solid-liquid extractions of bioactive compounds. The orange by-product was investigated as a source of pectins, a polysaccharide mainly composed of galacturonic acid with several technological applications and healthy properties. The mushroom stalks were used as a source of ergosterol (a provitamin of vitamin D), phenolic compounds, and antioxidant activity. In both studies, extraction kinetics were obtained under relatively mild conditions, such as low temperature (25 °C) and using non-aggressive solvents. Thus, an organic acid (citric acid) at two pHs (1.5 and 2.0) was used in the case of extraction of pectins from orange by-products, and two concentrations of ethanol in water (70 and 96 % v/v) in the extractions of bioactive compounds from mushroom by-products. The ultrasound-assisted extraction was compared with conventional mechanically agitated extraction. For all the bioactive compounds extracted from both by-products, higher extraction yields were observed with the application of high-power ultrasound compared with mechanical agitation under any conditions. However, for some of the bioactive compounds, the solvent had a considerable effect on the extraction yields. For instance,

the extraction yields of pectins after 30 min of extraction were up to 178 % higher at pH 1.5 than at pH 2.0. Moreover, the extraction yields obtained with high-power ultrasound (542-794 W/L) were 105-147 % higher at pH 1.5 and 40-80 % higher at pH 2.0 than those obtained with mechanical agitation (82 rpm), indicating that the efficiency of the ultrasound-assisted extraction was affected by the pH of the solvent. The degree of methylation of the pectins was significantly higher when extracting at pH 1.5 (55 ± 1 %) than at pH 2.0 (46 ± 4 %). The application of high-power ultrasound for long periods (up to 60 min) might have promoted the extraction of non-pectic polysaccharides such as hemicelluloses or even cellulose. The second-order rate model properly simulated the pectins extraction kinetics obtaining a mean relative error (MRE) ≤ 7.4 %. The maximum yield (Ymax) increased with the application of high-power ultrasound by 103-175 % (542-794 W/L) at pH 1.5 and by 34-70 % pH 2.0, compared with mechanical agitation. Similarly, the initial extraction rate (h) increased with the high-power ultrasound (542-794 W/L) by 42-63 % at pH 1.5 and by 6-68 % at pH 2.0.

In the case of the bioactive compounds extracted from mushroom stalks at 25 °C, the ergosterol extraction yields were up to 213 % higher ($p < 0.05$) in 96 % ethanol than in 70 % ethanol. However, no significant effect of the ethanol concentration was observed in the extraction yields of the phenolic compounds, while the antioxidant activity extraction yields (according to CUPRAC assay) were slightly higher (up to 11 % higher) in 70 % ethanol than in 96 % ethanol. For ergosterol, the extraction yields obtained with the high-power ultrasound (182-321 W/L) were 123-200 % higher in 70 % ethanol and 16-20 % higher in 96 % ethanol compared to those obtained with mechanical agitation (130 rpm). For the phenolic compounds, the extraction yield with high-power ultrasound (182-321 W/L) was 20-27 % higher in 70 % ethanol and 27- 46 % higher in 96 % ethanol. These increases caused by the high-power ultrasound were 17-25 % in 70 % ethanol and 10-19 % in 96 % ethanol for the antioxidant activity. Interestingly, similar ergosterol extraction yields were observed after 30 min with mechanical agitation in 96 % ethanol to those with 321 W/L in 70 % ethanol (about 36 and 34 % of the initial ergosterol, respectively). The highest ergosterol extraction yield was obtained with ethanol 96 % and 321 W/L (about 45 % of the initial ergosterol of the mushroom stalks). In the case of the phenolic compounds, the highest extraction yield was obtained with 321 W/L (average of both ethanol concentration of 52.5 ± 2.2 % of the initial phenolic compounds). For the antioxidant activity, the highest yield was about 55 % of the initial antioxidant activity, obtained with 321 W/L in 70 % ethanol. The antioxidant activity and the ergosterol and phenolic contents of the extracts were highly correlated (Pearson's correlation coefficients ≥ 0.96). The extraction kinetics of bioactive compounds from mushroom stalks were satisfactorily modelled using the Weibull model (MRE ≤ 7.8 %). The equilibrium yield ($Y_{eq}$) significantly ($p < 0.05$) increased with the high-power ultrasound (182-321 W/L) by 115-203 % in 70 % ethanol and 12-21 % in 96 % ethanol for

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ergosterol; by 18-32 % in 96 % for the phenolic compounds; and by 17-30 % in 70 % ethanol and 11-19 % in 96 % ethanol for the antioxidant activity.

The solid mushroom residue obtained after the extraction process was characterized as being rich in other valuable components: high content of polysaccharides such as β-glucans (average of 12.2 ± 1.7 g/100 dm (dry matter)), a macromolecule with several healthy properties, and proteins (average of 19.3 ± 2.1 g/100 g dm).

The second part of the work for this thesis consisted of the assessment of agri-food by-products' application in oil microencapsulation by spray drying. Thus, the mushroom residues obtained after the extraction of bioactive compounds (mushroom concentrate), and a flour obtained from artichoke bracts (by drying (to a moisture content of 3.6 g/100 g dm), grinding, and sieving < 0.09 mm), were used. These materials were added to oil-in-water emulsions in different concentrations substituting a commercial emulsifier (Tween®20) and partially replacing a common wall material (maltodextrin). The emulsions were compared with a control containing Tween®20 as an emulsifier and only maltodextrin as wall material. The emulsions containing the mushroom concentrate or the artichoke flour presented significantly ($p < 0.05$) higher viscosity and better stability than the control, but this effect depended on the concentration of these materials in the emulsion. Thus, in the case of the mushroom concentrate, the emulsions containing the highest amount of this material (5.0 and 7.5 % w/w) presented a droplet size distribution similar to the control (average median droplet diameters ($d_{50}$) of 2.4 ± 0.4 μm), better stability against droplet size variation and migration than the control (for about 48 h), and a shear-thinning behaviour. However, emulsions containing 1.5 and 3.0 % w/w of the mushroom concentrate, presented significantly ($p < 0.05$) larger droplets ($d_{50}$ about 5.2 and 3.4 μm, respectively) and flocculated easily. Quantitative information about the flocculated oil was obtained with optical microscopy combined with image analysis. Thus, emulsions with 1.5 and 3.0 % of mushroom concentrate exhibited about 23 and 16 % of the oil volume flocculated, probably due to bridging flocculation. These values were 3, 9, and 6 % for the control emulsion and the emulsions with 5.0 and 7.5 % of mushroom concentrate, respectively.

Regarding the study of the emulsions containing the artichoke flour, the control emulsion and the emulsions containing 1.0-2.0 % w/w of this material presented small droplets (average $d_{50}$ of 2.7 ± 0.1 μm). However, about 13 % of the oil volume was flocculated in the emulsion with 1.0 % of the artichoke flour, indicating that bridging flocculation also occurred with a low concentration of this material. Only 5 % of the oil volume was flocculated in the emulsion with 2.0 % of artichoke flour, and this emulsion showed better stability against droplet size variation than the control emulsion (for about 24 h). The resulting stability with the mushroom concentrate (5.0 and 7.5 %) and the artichoke flour addition (2.0 %) was due to the viscosity increase, steric hindrance, and probably a Pickering effect since the insoluble proportion of these materials was

high (about 55 and 57 %, respectively) and was in the form of solid particles suspended in the emulsion.

After the spray drying of the emulsion containing 5.0 % of the mushroom concentrate, the powder presented a high oil encapsulation efficiency (about 89 %). Similarly, the presence of artichoke flour (2.0 %) in the initial emulsions resulted in a significantly ($p < 0.05$) higher encapsulation efficiency after spray drying (about 79 %) than the control. Further, the oil encapsulated with the mushroom concentrate or the artichoke flour exhibited better oxidative stability during spray drying and controlled storage (35 °C and 50 % relative humidity) than the control for about 1 and 2 months, respectively. This indicates that the antioxidant activity of these natural materials protected the oil from oxidation (about 23 and 60 mg of Trolox equivalent (TE)/g dm according to CUPRAC assay for mushroom concentrate and artichoke flour, respectively). The microcapsules obtained with these natural materials presented significantly ($p < 0.05$) lower moisture content and solubility in water, larger particles, and perceptible colour changes compared with the control. Other parameters such as the density, porosity, and glass transition temperature (Tg) were not significantly ($p > 0.05$) affected by the presence of the mushroom concentrate or the artichoke flour.

To sum up, this thesis has proposed alternatives for the exploitation of agri-food by-products by intensifying the extraction of bioactive compounds through the application of high-power ultrasound and providing one of the first comprehensive assessments of the application of agri-food by-products in lipid microencapsulation by spray drying.

# RESUMEN

La industria agroalimentaria genera gran cantidad de residuos y subproductos que representan un importante problema medioambiental. Por esta razón, en el año 2020, la Unión Europea aprobó un plan de economía circular que incluye, como una de sus acciones clave, la reducción del desperdicio de alimentos. En este modelo económico se pretende mantener los productos y materias primas en la cadena el mayor tiempo posible. Por ello, en los últimos años se está prestando especial atención al uso de residuos agroalimentarios como fuente de compuestos bioactivos. Para facilitar la extracción sólido-líquido de estos compuestos, la industria alimentaria precisa intensificar el proceso mediante la aplicación de nuevas tecnologías capaces de reducir la resistencia natural a la transferencia de masa ofrecida por las paredes celulares de los tejidos vegetales o fúngicos. La aplicación de ultrasonidos de potencia es uno de los métodos de intensificación que ha demostrado su eficacia, pero aún, es necesario estudiar sus efectos sobre diferentes matrices y condiciones de extracción. Además, tras la extracción de las moléculas de alto valor, queda un residuo que aún puede contener compuestos con bioactividad y/o propiedades tecnológicas interesantes. De cara a una valorización integral, estos residuos deben ser evaluados para su aplicación en procesos industriales.

En este contexto, el objetivo general de esta tesis fue proponer alternativas para la valorización integral de subproductos y residuos. Concretamente se propone, por una parte, revalorizar los subproductos agroalimentarios mediante la extracción acústica de algunos de sus compuestos bioactivos; y por otra, explorar el uso de estos subproductos y de los residuos del proceso de extracción en procesos de microencapsulación de lípidos, aprovechando su capacidad emulsionante y antioxidante.

En esta tesis se trabajó con subproductos de tres materias primas diferentes (una fruta, un hongo y una verdura): naranja (piel y pulpa), champiñón (tallos y residuos obtenidos después de la extracción de compuestos bioactivos) y alcachofa (brácteas).

Los experimentos de extracción sólido-líquido de compuestos bioactivos se realizaron con subproductos de naranja y champiñón. Se consideró al subproducto de naranja como fuente de pectinas, un polisacárido compuesto principalmente por ácido galacturónico con diferentes aplicaciones tecnológicas y propiedades saludables. Los tallos de los champiñones se valorizaron como fuente de ergosterol (una provitamina de la vitamina D), compuestos fenólicos y actividad antioxidante. En ambos estudios, las cinéticas de extracción se realizaron en condiciones relativamente suaves, como baja temperatura (25 °C) y disolventes no agresivos. Concretamente, se utilizó como disolvente un ácido orgánico (ácido cítrico) a dos pHs (1.5 y 2.0) en el caso de la extracción de pectinas de subproductos de naranja, y dos concentraciones de etanol en agua (70 y 96 % v/v) en las extracciones de compuestos bioactivos a partir de

subproductos de champiñón. Los experimentos de extracción acústica se compararon con extracciones convencionales llevadas a cabo mediante agitación mecánica. Para las mismas condiciones de operación, en ambos subproductos y para todos los compuestos bioactivos extraídos, se observaron mayores rendimientos de extracción en las experiencias en las que se aplicaron ultrasonidos de potencia que en los procesos realizados con agitación mecánica. También se observó que, en el caso de algunos de los compuestos bioactivos, el solvente utilizado afectó en gran medida a los rendimientos de extracción. Por ejemplo, después de 30 min de extracción, los rendimientos de extracción de pectinas fueron hasta un 178 % más elevados a pH 1.5 que a pH 2.0. Además, los rendimientos de extracción obtenidos aplicando ultrasonidos de potencia (542-794 W/L) en relación con los obtenidos con agitación mecánica (82 rpm), fueron 105-147 % superiores a pH 1.5 y 40-80 % a pH 2.0, lo que indicó que la eficacia de la extracción acústica se vio afectada por el pH del disolvente. Asimismo, el grado de metilación de las pectinas fue significativamente superior al extraer a pH 1.5 (55 ± 1 %) que a pH 2.0 (46 ± 4 %). Además de los rendimientos, los ultrasonidos de potencia también afectaron la composición de los extractos, ya que la aplicación de energía acústica durante 60 min provocó la extracción de polisacáridos no pécticos como hemicelulosas o incluso celulosa. Las cinéticas de extracción de pectinas pudieron simularse adecuadamente mediante el uso de un modelo cinético de segundo orden, obteniendo un error relativo medio (MRE) ≤ 7.4 %. El rendimiento máximo (Ymax) aumentó con la aplicación de ultrasonidos de potencia en comparación con el de los experimentos realizados con agitación mecánica en 103-175 % (542-794 W/L) a pH 1.5 y en 34-70 % a pH 2.0. De manera similar, la tasa de extracción inicial (h) aumentó con la aplicación de potencia acústica (542-794 W/L) en un 42-63 % a pH 1.5 y en un 6-68 % a pH 2.0.

En el caso de los compuestos bioactivos extraídos a partir de tallos de champiñón a 25 °C, los rendimientos de extracción de ergosterol fueron hasta un 213 % más elevados en etanol al 96 % que en etanol al 70 %. Sin embargo, no se observó ningún efecto significativo de la concentración de etanol en el rendimiento de extracción de los compuestos fenólicos, mientras que el rendimiento de extracción de la actividad antioxidante (según el ensayo CUPRAC) fue ligeramente superior (hasta un 11 % más alto) en etanol al 70 % que en 96 %. Los rendimientos de extracción de ergosterol obtenidos mediante la aplicación de potencia acústica (182-321 W/L), en comparación con los obtenidos con agitación mecánica (130 rpm), fueron un 123-200 % superiores en etanol al 70 % y un 16-20 % en etanol al 96 %. En el caso de la extracción de compuestos fenólicos, los rendimientos obtenidos mediante la aplicación de ultrasonidos de potencia (182-321 W/L), en relación con los obtenidos con agitación mecánica, fueron un 20-27 % superiores en etanol al 70 % y un 27-46 % en etanol al 96 %. Con respecto a la actividad antioxidante, los rendimientos obtenidos mediante la aplicación de energía acústica (182-321 W/L), al compararlos con los obtenidos mediante agitación mecánica,

fueron 17-25 % más elevados en etanol al 70 % y 10-19 % en etanol al 96 %. Cabe resaltar que la aplicación de energía acústica (321 W/L) permitió obtener rendimientos de extracción de ergosterol en 70 % de etanol, similares a los obtenidos en experimentos con agitación mecánica con una concentración de etanol más alta (96 %) (aproximadamente 34 y 36 % del ergosterol inicial, respectivamente). Entre todas las condiciones evaluadas, el rendimiento de extracción de ergosterol más alto se obtuvo con etanol al 96 % y 321 W/L (aproximadamente el 45 % del ergosterol inicial presente en los tallos de champiñón). En el caso de los compuestos fenólicos, el rendimiento más elevado se obtuvo con 321 W/L (rendimiento promedio de ambas concentraciones de etanol de 52.5 ± 2.2 % de los compuestos fenólicos iniciales). Con respecto a la actividad antioxidante, el rendimiento de extracción más alto fue de aproximadamente el 55 % de la actividad antioxidante inicial, y se obtuvo con 321 W/L en etanol al 70 %. Se observó una alta correlación entre la actividad antioxidante de los extractos y el contenido de ergosterol y de compuestos fenólicos (coeficientes de correlación de Pearson ≥ 0.96). Las cinéticas de extracción de los compuestos bioactivos obtenidos a partir de tallos de champiñón se pudieron simular adecuadamente utilizando el modelo de Weibull (MRE ≤ 7.8 %). El rendimiento de extracción en el equilibrio ($Y_{eq}$) aumentó con la aplicación de ultrasonidos de potencia (182-321 W/L) un 115-203 % en etanol al 70 % y un 12-21 % en etanol al 96 % para el ergosterol; en un 18-32 % en etanol al 96 % para los compuestos fenólicos; y en un 17-30 % en etanol al 70 % y un 11-19 % en etanol al 96 % para la actividad antioxidante.

Después del proceso de extracción de compuestos bioactivos del champiñón, quedó, como residuo, un concentrado que todavía contenía moléculas valiosas como β-glucanos (12.2 ± 1.7 g/100 b.s (base seca)), una macromolécula con varias propiedades saludables, y proteínas (19.3 ± 2.1 g /100 g b.s).

La segunda parte de esta tesis consistió en evaluar la viabilidad del uso de subproductos agroalimentarios en el proceso de microencapsulación de lípidos mediante secado por atomización. Se utilizaron los residuos de champiñón obtenidos tras la extracción de compuestos bioactivos (concentrado de champiñón), y una harina obtenida de brácteas de alcachofa (obtenida mediante secado (3.6 g/100g b.s de humedad), molido y tamizado (<0.09 mm)). Se añadieron estos materiales a emulsiones de aceite en agua en diferentes concentraciones, sustituyendo un emulsionante comercial (Tween®20) y reemplazando parcialmente un material de pared común (maltodextrina). Las emulsiones se compararon con una emulsión control que se preparó con Tween®20 como emulsionante y solo maltodextrina como material de pared. Las emulsiones que contenían el concentrado de champiñón o la harina de alcachofa presentaron valores de viscosidad significativamente ($p < 0.05$) más altos que la emulsión control, así como también mejor estabilidad, siendo este efecto dependiente de la concentración de dichos materiales en la emulsión. Así, en el caso del concentrado de champiñón, las emulsiones que contenían la mayor cantidad de este material (5.0 y 7.5 % p/p)

presentaron una distribución de tamaño de gotas de aceite similar a la del control (una mediana del diámetro de las gotas ($d_{50}$) de 2.4 ± 0.4 µm). Estas emulsiones también presentaron mejor estabilidad frente a la variación del tamaño y migración de las gotas que el control (durante aproximadamente 48 h) y un comportamiento pseudoplástico. Sin embargo, las emulsiones que contenían 1.5 y 3.0 % p/p de concentrado de champiñón, presentaron gotas significativamente más grandes ($p < 0.05$) ($d_{50}$ de 5.2 y 3.4 µm, respectivamente) y floculaban fácilmente. El nivel de floculación del aceite se cuantificó mediante microscopía óptica combinada con análisis de imagen. Las emulsiones con 1.5 y 3.0 % de concentrado de champiñón presentaron, aproximadamente, un 23 y 16 % del volumen de aceite floculado, probablemente debido al fenómeno de "bridging flocculation" o floculación por puente. En la emulsión control y en las emulsiones que contenían cantidades más elevadas del concentrado de champiñón, los niveles de floculación fueron inferiores, se observó un 3, 9 y 6 % del volumen de aceite floculado para la emulsión control y las emulsiones con 5.0 y 7.5 % de concentrado de champiñón, respectivamente.

En el caso del estudio de las emulsiones que contenían la harina de alcachofa, la emulsión control y las emulsiones que contenían 1.0-2.0 % p/p de este material presentaron gotas pequeñas ($d_{50}$ promedio de 2.7 ± 0.1 µm). Sin embargo, la emulsión que contenía 1.0 % de la harina de alcachofa presentó aproximadamente un 13 % del volumen de aceite floculado, lo que indica que cuando la concentración de este material fue excesivamente baja también se produjo "bridging flocculation". En el caso de la emulsión con un 2.0 % de harina de alcachofa, sólo el 5 % del volumen de aceite estaba floculado. Esta emulsión presentó, durante aproximadamente 24 h, mayor estabilidad frente a la variación del tamaño de las gotas que la emulsión control. La adición del concentrado de champiñón (5.0 y 7.5 %) y de la harina de alcachofa (2.0 %) generaron emulsiones más estables como consecuencia del aumento de viscosidad, impedimento estérico y probablemente un efecto Pickering ya que la proporción insoluble de estos materiales era elevada (aproximadamente 55 y 57 %, respectivamente).

Después del secado por atomización de la emulsión con un 5.0 % de concentrado de champiñón, el polvo resultante presentó una elevada proporción del aceite encapsulado (aproximadamente 89 %). De manera similar, la presencia de harina de alcachofa (2.0 %) en las emulsiones permitió obtener, después del secado por atomización, una elevada eficiencia en la encapsulación de aceite (aproximadamente 79 %), significativamente ($p < 0.05$) más alta que la que se observó después de secar la emulsión control preparada para ese estudio. Además, el aceite encapsulado con el concentrado de champiñón o la harina de alcachofa presentó mayor estabilidad oxidativa durante el secado, y también en el almacenamiento controlado (35 ° C y 50 % de humedad relativa) durante alrededor de 1 y 2 meses, respectivamente, que el control. Estos resultados indican que la actividad antioxidante del concentrado de champiñón y de la harina de alcachofa (alrededor de 23 y 60 mg de equivalente de

Trolox/ g b.s según el ensayo CUPRAC para el concentrado de champiñón y la harina de alcachofa, respectivamente) redujo la oxidación del aceite. Las microcápsulas obtenidas con estos materiales naturales presentaron menor contenido de humedad y solubilidad en agua ($p < 0.05$), partículas más grandes ($p < 0.05$) y cambios de color perceptibles en comparación con el control. Otros parámetros como la densidad, la porosidad y la temperatura de transición vítrea (Tg) no se vieron afectados significativamente por la presencia del concentrado de champiñón o la harina de alcachofa.

En resumen, esta tesis ha propuesto diferentes alternativas para la valorización de subproductos agroalimentarios, por un lado, intensificando la extracción de sus compuestos bioactivos mediante la aplicación de una tecnología sostenible como es la aplicación de ultrasonidos de potencia, y por otro, presentando por primera vez el uso de subproductos agroalimentarios en el proceso de microencapsulación lipídica por atomización.

# RESUM

La indústria agroalimentària genera gran quantitat de residus i subproductes que representen un important problema mediambiental. Per aquesta raó, l'any 2020, la Unió Europea va aprovar un pla d'economia circular que inclou, com a una de les seves accions clau, la reducció del desaprofitament d'aliments. En aquest model econòmic es pretén mantenir els productes i matèries primeres en la cadena el major temps possible. Per això, en els últims anys s'està parant especial esment a l'ús de residus agroalimentaris com a font de compostos bioactius. Per a facilitar l'extracció sòlid-líquid d'aquests composts, la indústria alimentària necessita intensificar el procés mitjançant l'aplicació de noves tecnologies capaces de reduir la resistència natural de les parets cel·lulars del teixits vegetals o fúngics a la transferència de massa. L'aplicació d'ultrasons de potència és un dels mètodes d'intensificació que ha demostrat una bona eficàcia, però encara és necessari estudiar els seus efectes sobre diferents matrius i condicions d'extracció. A més, després de l'extracció de les molècules d'alt valor, queda un residu que pot tenir composts amb bioactivitat i/o propietats tecnològiques interessants. Des del punt de vista d'una valorització integral, aquests residus han de ser avaluats per a la seva aplicació en processos industrials.

En aquest context, l'objectiu general d'aquesta tesi va ser proposar alternatives per a la valorització integral de subproductes i residus. Concretament es proposa, per una part, revaloritzar els subproductes agroalimentaris mitjançant l'extracció acústica d'alguns dels seus composts bioactius; i per una altra, explorar l'ús d'aquests subproductes i dels residus del procés d'extracció en processos de microencapsulació de lípids, aprofitant la seva capacitat emulsionant i antioxidant.

En aquesta tesi es va treballar amb subproductes de tres matèries primeres diferents (una fruita, un fong i una verdura): taronja (pell i polpa), xampinyó (tiges i residus obtinguts després de l'extracció de compostos bioactius) i carxofa (bràctees).

Els experiments d'extracció sòlid-líquid dels composts bioactius es van realitzar amb subproductes de taronja i xampinyó. Es va considerar el subproducte de taronja com a font de pectines, un polisacàrid compost principalment per àcid galacturònic amb diferents aplicacions tecnològiques i propietats beneficioses per la salut. Les tiges dels xampinyons es van valorar com a font d'ergosterol (una provitamina de la vitamina D), composts fenòlics i activitat antioxidant. En els dos estudis, les cinètiques d'extracció es van realitzar en condicions relativament suaus, com a baixa temperatura (25 °C) i dissolvents no agressius. Concretament, es va utilitzar com a dissolvent un àcid orgànic (àcid cítric) a dos pHs (1.5 i 2.0) en el cas de l'extracció de pectines de subproductes de taronja, i dues concentracions d'etanol en aigua (70 i 96 % v/v) en les extraccions de composts bioactius a partir de subproductes de xampinyó. Els experiments d'extracció acústica es van comparar amb extraccions convencionals realitzades mitjançant agitació

mecànica. Per a les mateixes condicions d'operació, en els dos subproductes i per tots els compostos bioactius estudiats, es van observar majors rendiments d'extracció en les experiències amb ultrasons de potència que en els processos realitzats amb agitació mecànica. També es va observar que, en el cas d'alguns dels compostos bioactius, el solvent utilitzat va afectar en gran mesura als rendiments d'extracció. Per exemple, després de 30 min d'extracció, els rendiments d'extracció de pectines van ser fins a un 178 % més elevats a pH 1.5 que a pH 2.0. A més, els rendiments d'extracció obtinguts aplicant ultrasons de potència (542-794 W/L) en relació amb els obtinguts amb agitació mecànica (82 rpm), van ser de 105-147 % superiors a pH 1.5 i 40-80 % a pH 2.0, la qual cosa va indicar que l'eficàcia de l'extracció acústica es va veure afectada pel pH del dissolvent. Així mateix, el grau de metilació de les pectines va ser significativament superior extretes a pH 1.5 (55 ± 1 %) que a pH 2.0 (46 ± 4 %). A més dels rendiments, els ultrasons de potència també van afectar la composició dels extractes, ja que l'aplicació d'energia acústica durant 60 min va provocar l'extracció de polisacàrids no pèctics com hemicel·luloses o, fins i tot, cel·lulosa. Les cinètiques d'extracció de pectines van poder simular-se adequadament mitjançant l'ús d'un model cinètic de segon ordre, obtenint un error relatiu mitjà (MRE) ≤ 7.4 %. El rendiment màxim (Ymax) va augmentar amb l'aplicació d'ultrasons de potència en comparació amb el dels experiments realitzats amb agitació mecànica en 103-175 % (542-794 W/L) a pH 1.5 i en 34-70 % a pH 2.0. De la mateixa manera, la taxa d'extracció inicial (h) va augmentar amb l'aplicació de potència acústica (542-794 W/L) en un 42-63 % a pH 1.5 i en un 6-68 % a pH 2.0.

En el cas dels compostos bioactius extrets a partir de tiges de xampinyó a 25 °C, els rendiments d'extracció d'ergosterol van ser fins a un 213 % més elevats en etanol al 96 % que en etanol al 70 %. No obstant això, no es va observar cap efecte significatiu de la concentració d'etanol en el rendiment d'extracció dels compostos fenòlics, mentre que el rendiment d'extracció de l'activitat antioxidant (segons l'assaig CUPRAC) va ser lleugerament superior (fins a un 11 % més alt) en etanol al 70 % que en 96 %. Els rendiments d'extracció d'ergosterol obtinguts mitjançant l'aplicació de potència acústica (182-321 W/L), en comparació amb els obtinguts amb agitació mecànica (130 rpm), van ser un 123-200 % superiors en etanol al 70 % i un 16-20 % en etanol al 96 %. En el cas de l'extracció de compostos fenòlics, els rendiments obtinguts mitjançant l'aplicació d'ultrasons de potència (182-321 W/L), en relació amb els obtinguts amb agitació mecànica, van ser un 20-27 % superiors en etanol al 70 % i un 27-46 % en etanol al 96 %. Respecte a l'activitat antioxidant, els rendiments obtinguts mitjançant l'aplicació d'energia acústica (182-321 W/L), en comparar-los amb els obtinguts mitjançant agitació mecànica, van ser 17-25 % més elevats en etanol al 70 % i 10-19 % en etanol al 96 %. Cal ressaltar que l'aplicació d'energia acústica (321 W/L) va permetre obtenir rendiments d'extracció d'ergosterol en 70% d'etanol similars als obtinguts en experiments amb agitació mecànica amb una concentració d'etanol més alta (96 %) (aproximadament 34 i 36 % del ergosterol inicial, respectivament). Entre totes les condicions avaluades, el

rendiment d'extracció d'ergosterol més alt es va obtenir amb etanol al 96 % i 321 W/L (aproximadament el 45 % de l'ergosterol inicial present en les tiges de xampinyó). En el cas dels compostos fenòlics, el rendiment més elevat es va obtenir amb 321 W/L (rendiment mitjà de les dues concentracions d'etanol de 52.5 ± 2.2 % dels compostos fenòlics inicials). Respecte a l'activitat antioxidant, el rendiment d'extracció més alt va ser d'aproximadament el 55 % de l'activitat antioxidant inicial, i es va obtenir amb 321 W/L en etanol al 70 %. Es va observar una alta correlació entre l'activitat antioxidant dels extractes i el contingut d'ergosterol i de compostos fenòlics (coeficients de correlació de Pearson ≥ 0.96). Les cinètiques d'extracció dels compostos bioactius obtinguts a partir de tiges de xampinyó es van poder simular adequadament utilitzant el model de Weibull (MRE ≤ 7.8 %). El rendiment d'extracció en l'equilibri ($Y_{eq}$) va augmentar amb l'aplicació d'ultrasons de potència (182-321 W/L) un 115-203 % en etanol al 70 % i un 12-21 % en etanol al 96 % per l'ergosterol; en un 18-32 % en etanol al 96 % per els compostos fenòlics; i en un 17-30 % en etanol al 70 % i un 11-19 % en etanol al 96 % per l'activitat antioxidant.

Després del procés d'extracció de compostos bioactius del xampinyó, va quedar, com a residu, un concentrat que encara contenia molècules valuoses com β-glucans (12.2 ± 1.7 g/100 b.s (base seca)), una macromolècula amb diverses propietats beneficioses per a la salut, i proteïnes (19.3 ± 2.1 g /100 g b.s).

La segona part d'aquesta tesi va consistir en avaluar la viabilitat de l'ús de subproductes agroalimentaris en el procés de microencapsulació de lípids mitjançant assecat per atomització. Es van utilitzar els residus de xampinyó obtinguts després de l'extracció de compostos bioactius (concentrat de xampinyó), i una farina obtinguda de bràctees de carxofa (obtinguda mitjançant assecat (3.6 g/100g b.s d'humitat), molt i tamisat (<0.09 mm)). Es van afegir aquests materials a emulsions d'oli en aigua en diferents concentracions, substituint un emulsionant comercial (Tween®20) i reemplaçant parcialment un material d'encapsulació (maltodextrina). Les emulsions es van comparar amb una emulsió control que es va preparar amb Tween®20 com a emulsionant i només maltodextrina com a material d'encapsulació. Les emulsions que contenien el concentrat de xampinyó o la farina de carxofa van presentar valors de viscositat significativament ($p < 0.05$) més alts que l'emulsió control, així com també millor estabilitat, essent aquest efecte dependent de la concentració d'aquests materials en l'emulsió. Així, en el cas del concentrat de xampinyó, les emulsions que contenien la major quantitat d'aquest material (5.0 i 7.5 % p/p) van presentar una distribució de grandària de gotes d'oli similar a la del control (una mitjana del diàmetre de les gotes (d50) de 2. 4 ± 0. 4 µm). Aquestes emulsions també van presentar millor estabilitat enfront de la variació de la grandària i migració de les gotes que el control (durant aproximadament 48 h) i un comportament pseudo-plàstic. No obstant això, les emulsions que contenien 1.5 i 3.0 % p/p de concentrat de xampinyó, van presentar gotes significativament més grans ($p < 0.05$) (d$_{50}$ de 5.2 i 3.4 µm, respectivament) i floculaven fàcilment. El nivell de floculació de l'oli es va quantificar mitjançant microscòpia òptica

combinada amb anàlisi d'imatge. Les emulsions amb 1.5 i 3.0% de concentrat de xampinyó van presentar, aproximadament, un 23 i 16 % del volum d'oli floculat, probablement a causa del fenomen de "bridging flocculation" o floculació per pont. En l'emulsió control i en les emulsions amb quantitats més elevades del concentrat de xampinyó, els nivells de floculació van ser inferiors, es va observar un 3, 9 i 6 % del volum d'oli floculat per a l'emulsió control i les emulsions amb 5.0 i 7.5 % de concentrat de xampinyó, respectivament.

En el cas de l'estudi de les emulsions que contenien la farina de carxofa, l'emulsió control i les emulsions que contenien 1.0-2.0 % p/p d'aquest material van presentar gotes petites ($d_{50}$ mitjana de 2.7 ± 0.1 μm). No obstant això, l'emulsió que contenia 1.0 % de la farina de carxofa va presentar aproximadament un 13 % del volum d'oli floculat, la qual cosa indica que quan la concentració d'aquest material va ser excessivament baixa també es va produir "bridging flocculation". En el cas de l'emulsió amb un 2.0 % de farina de carxofa, només el 5 % del volum d'oli estava floculat. Aquesta emulsió va presentar, durant aproximadament 24 h, major estabilitat enfront de la variació de la grandària de les gotes que l'emulsió control. L'addició del concentrat de xampinyó (5.0 i 7.5 %) i de la farina de carxofa (2.0 %) van generar emulsions més estables a conseqüència de l'augment de viscositat, impediment estèric i probablement un efecte Pickering, ja que la proporció insoluble d'aquests materials era elevada (aproximadament 55 i 57 %, respectivament).

Després de l'assecat per atomització de l'emulsió amb un 5.0 % de concentrat de xampinyó, la pols resultant va presentar una elevada proporció d'oli encapsulat (aproximadament 89 %). De manera semblant, la presència de farina de carxofa (2.0 %) en les emulsions va permetre obtenir, després de l'assecat per atomització, una elevada eficiència en l'encapsulació d'oli (aproximadament 79 %), significativament ($p < 0.05$) més alta que la que es va observar després d'assecar l'emulsió control preparada per a aquest estudi. A més, l'oli encapsulat amb el concentrat de xampinyó o la farina de carxofa va presentar major estabilitat oxidativa durant l'assecat, i també durant l'emmagatzematge controlat (35 ° C i 50 % d'humitat relativa) al voltant d'1 i 2 mesos, respectivament, que el control. Aquests resultats indiquen que l'activitat antioxidant del concentrat de xampinyó i de la farina de carxofa (al voltant de 23 i 60 mg d'equivalent de Trolox/ g b.s segons l'assaig CUPRAC per al concentrat de xampinyó i la farina de carxofa, respectivament) va reduir l'oxidació de l'oli. Les microcàpsules obtingudes amb aquests materials naturals van presentar menor contingut d'humitat i solubilitat en aigua ($p < 0.05$), partícules més grans ($p < 0.05$) i canvis de color perceptibles en comparació amb el control. Altres paràmetres com la densitat, la porositat i la temperatura de transició vítria (Tg) no es van veure afectats significativament per la presència del concentrat de xampinyó o la farina de carxofa.

En resum, aquesta tesi ha proposat diferents alternatives per a la valorització de subproductes agroalimentaris, d'una banda, intensificant l'extracció dels seus composts bioactius mitjançant l'aplicació d'una tecnologia sostenible com és l'aplicació d'ultrasons de potència, i de l'altra, presentant per primera vegada l'ús de subproductes agroalimentaris en el procés de microencapsulació lipídica amb assecat per atomització.

# NOMENCLATURE

**Parameters and analysis**

| | |
|---|---|
| a* | CIELab coordinate (redness/greenness) |
| AA | Antioxidant activity (mg TE/g dm) or (g TE/g dm) |
| Abs 234 nm | Absorbance at 234 nm |
| ABTS | 2,2'-azino-bis(3-ethylbenzo- thiazoline-6-sulphonic acid |
| Ara | Arabinose |
| b* | CIELab coordinate (yellowness/blueness) |
| BS | Backscattering |
| C | Concentration (mg/g dm) (Chapter 2) |
| CCI | Carr's compressibility index |
| CI | Confidence intervals (95 %) (Chapters 1 and 2) |
| CI | Creaming index (Chapters 3 and 5) |
| Cp | Specific heat capacity of the solvent (J/kg K) |
| CUPRAC | Cupric Reducing Antioxidant Capacity |
| $d_{10}$ | Percentile 10 % |
| $d_{50}$ | Percentile 50 %, median droplet diameter |
| $d_{90}$ | Percentile 90 % |
| DE | Dextrose equivalent |
| DhaA | 3-deoxy-D-lyxo-heptulosaric acid |
| dm | Dry matter |
| DM | Degree of methylation |
| DSC | Differential scanning calorimetry |
| dTGA | Derivate of thermogravimetric analysis |
| FAME | Fatty acids methyl esters |
| FRAP | Ferric reducing antioxidant power |
| Fuc | Fucose |
| g | Gravity acceleration |
| GA | Gallic acid |

| | |
|---|---|
| GAE | Gallic acid equivalent |
| Gal | Galactose |
| GalA | Galacturonic acid |
| GDP | Gross domestic product |
| Glc | Glucose |
| h | Initial extraction rate (% $min^{-1}$) |
| HG | Homogalacturonans |
| HMP | High methoxyl pectins |
| HLB | Hydrophilic-Lipophilic Balance |
| HR | Hausner ratio |
| k | Extraction rate constant ($\%^{-1}$ $min^{-1}$). |
| KdoA | 3-deoxy-D-manno-octulosonic acid |
| $L^*$ | CIELab coordinate (Luminosity) |
| LMP | Low methoxyl pectin |
| m | Mass (kg) |
| Man | Mannose |
| MRE % | Mean relative error (%) |
| n | Number of samples |
| $N_h$ | Number of height positions of the scan (Turbiscan) |
| O/W | Oil-in-water emulsions |
| P | Ultrasound power (W) |
| PI | Prediction limits (95 %) |
| PUFAs | Polyunsaturated fatty acids |
| r | Radius of the droplet |
| R | Ratio of $A_{1740}$ over the sum of $A_{1740}$ and $A_{1630}$ |
| RG I | Rhamnogalacturonans I |
| RG II | Rhamnogalacturonan II |
| RH | Relative humidity (%) |
| Rha | Rhamnose |
| S | Solubility (%) |

| | |
|---|---|
| SA | Specific absorbance of conjugated dienes at 234 nm |
| S | Standard deviation |
| SE | Standard error |
| SEM | Scanning electron microscopy |
| SH | Layer formed at the bottom of the test tube |
| t | Time |
| TE | Trolox equivalent |
| Tg | Glass transition temperature (°C) |
| TGA | Thermogravimetric analysis |
| TH | Total height of the sample in the test tube |
| TPC | Total phenolic compounds |
| TSI | Turbiscan stability index |
| UA | Uronic acids |
| UAE | Ultrasound-assisted extraction |
| US | Power ultrasounds |
| V | Volume ($m^3$) |
| VAR % | Percentage of explained variance (%) |
| $v_{stokes}$ | Velocity of the droplets' migration |
| W | Water content  (kg water/kg wet matter) |
| W/O | Water-in-oil emulsions |
| Wg | Oil mass in volume of organic solvent (g/100 ml) |
| wm | Wet matter |
| Xyl | Xylose |
| Y | Yield of extraction (%) |
| Z | Height limits of the cell (mm) (Turbiscan) |

**Organization and institutions**

| | |
|---|---|
| AEI | Spanish Research Agency |
| EFSA | European Food Safety Authority |
| ERDF | European Regional Development Fund |

| | |
|---|---|
| EU | European Union |
| FAO | Food and Agricultural Organization of the United Nations |
| HORECA | HOtels, Restaurants, and Coffee shops |
| INIA | National Institute of Research and Agri-food Technology |
| MAPA | Ministry of Agriculture, Fisheries, and Food of Spain |
| MITECO | Ministry for Ecological Transition and the Demographic Challenge of Spain |

**Subscripts**

| | |
|---|---|
| 0 | Initial |
| A | Apparent |
| b | Bulk |
| calc | Calculated |
| eq | Equilibrium |
| exp | Experimental |
| L | Loose |
| max | Maximum |
| min | Minimum |
| T | True |
| t | Tap |
| w | Water |

**Greek symbols**

| | |
|---|---|
| α | Kinetic reaction constant of the Weibull model (Chapter 2) |
| α | Mass of powder (kg) (Chapter 4) |
| β | Shape parameter of the Weibull model |
| γ | Shear rate ($s^{-1}$) |
| ΔBS | Backscattered light referred to the initial state (t=0 h) |
| ΔE | Total colour change |
| ε | Porosity (%) |

| | |
|---|---|
| η | Viscosity (mPa·s) |
| ρ | Density (kg/m$^3$ or g/mL) |

**Samples and conditions**

**Chapter 1**

| | |
|---|---|
| AG | Mechanically agitated extraction (82 rpm) |
| US1 | Ultrasound-assisted extraction 542 ± 4 W/L |
| US2 | Ultrasound-assisted extraction 794 ± 4 W/L |

**Chapter 2**

| | |
|---|---|
| MAE | Mechanically agitated extraction (130 rpm) |
| UAE | Ultrasound-assisted extraction |
| UAE1 | Ultrasound-assisted extraction 182 ± 7 W/L |
| UAE2 | Ultrasound-assisted extraction 321 ± 14 W/L |

**Chapter 3**

| | |
|---|---|
| C | Control emulsion with MD and Tween®20 (without MC) |
| MC | Mushroom by-products concentrate |
| MC1.5 | Emulsion containing 1.5 % w/w of MC |
| MC3 | Emulsion containing 3.0 % w/w of MC |
| MC5 | Emulsion containing 5.0 % w/w of MC |
| MC7.5 | Emulsion containing 7.5 % w/w of MC |
| MD | Maltodextrin |

**Chapter 4**

| | |
|---|---|
| C | Control emulsion with MD and Tween®20 (without MC) |
| CE | Emulsion containing MD as wall material and ergosterol enriched oil |
| M | Emulsion containing MD and MC as wall material |
| MC | Mushroom concentrate |
| MD | Maltodextrin |
| ME | Emulsion containing MD and MC as wall material and ergosterol enriched oil |

**Chapter 5**

| | |
|---|---|
| AB | Artichoke bracts flour |
| AB1 | Emulsion containing 1.0 % w/w of AB |
| AB2 | Emulsion containing 2.0 % w/w of AB |
| C | Control emulsion with MD and Tween®20 (without AB) |
| MD | Maltodextrin |
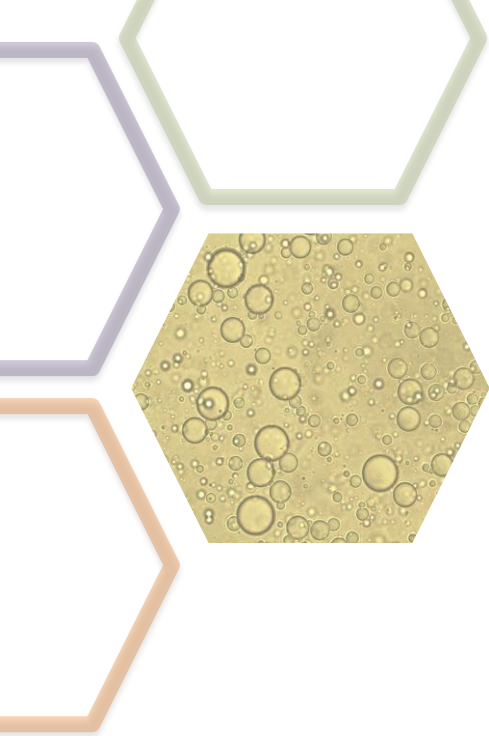
INTRODUCTION

# 1. INTRODUCTION

## 1.1. Residues from the agri-food industry

An approximation, calculated by the Food and Agricultural Organization of the United Nations (FAO) in 2011, suggested that roughly a third of the world's food is lost or wasted every year. In a report published by the FAO in 2019, food loss was defined as the decrease in the quantity or quality of food resulting from decisions and actions by food suppliers in the chain, excluding retail, food service providers, and consumers. Whilst food waste covers the decreases in the quantity or quality of food in the last stages of the chain (retailers, food services, and consumers).

In the European Union (EU) alone, it has been estimated that about 88 million tons of food residues are generated every year, including both edible food and inedible parts associated with food (Stenmarck et al., 2016). Specifically, Spain has reported a volume of food residues of about 7.7 million tons in a year, being the seventh country of the EU that wastes the greatest amount of food, according to the Ministry of Agriculture, Fisheries, and Food of Spain (MAPA).

This loss and waste of food have an enormous negative impact on the environment as finite natural resources such as water and soil are employed for its production. Moreover, these residues generate a high $CO_2$ footprint which was estimated to be around 3300 million tons of $CO_2$ globally in 2007 (FAO, 2013). More specifically, in the EU, food loss and waste generates about 8 % of the total greenhouse emission (Copa-Cogeca et al., 2020). Besides the environmental impact, there is also a huge economic cost. According to the FAO, vegetables were the major contributors to the global economic cost of food wastage (23 %), followed by meat (21 %), fruits (19 %), and cereals (18 %) (FAO, 2013). In the EU, the total cost of food loss and waste is estimated to be around 143 billion euros (Copa-Cogeca et al., 2020).

It has been estimated that in the EU, most of the waste is generated in homes (53 %) and manufacturing processes (19 %) followed by food services (12 %), primary production (11 %), and wholesale and retail (5 %) (Stenmarck et al., 2016). The FAO has previously reported that most affluent societies impose too restrictive quality standards of size or aesthetics, which are responsible for a large amount of the food wasted at the end of the chain (FAO, 2013). The most recent data of the FAO has reported that about 14 % of the food produced worldwide in 2016 was lost in the chain excluding the retail stage (food loss index). Meanwhile, the calculation of the food waste index, which covers retail and consumption, is still under development (FAO, 2019).

Apart from the edible material, the biomass inedible for humans is also a result of the inefficiency of the food production system and contributes to the environmental impact (Alexander et al., 2017). There is no official term for the inedible material related to food

production, but generally, it is considered a by-product of food crop processing and includes leaves, peels, barriers, seeds, bracts, pulp, stems, stalks, pomaces, roots, and so forth (Sagar et al., 2018). In some products, industrial processes cause exceedingly high quantities of discarded material. For instance, after extracting the juice of citrus fruits, by-products such as peel, pulp, and seeds account for 50-55 % of the weight of the initial fruits (Berk, 2016). Even higher percentages of by-products are observed in products such as artichokes since the non-edible material (including bracts, leaves, and stems) represents about 60-80 % of the total weight (Pandino et al., 2011; Ruiz-Aceituno et al., 2016). In the mushroom industry, products with irregular dimensions and/or shape, along with stalks, account for about 20-35 % of the weight of fresh mushrooms depending on the size of the production (Heleno et al., 2016b).

To simplify, and given the lack of official definitions, the word *residues* will be used to include both the food waste and losses along the supply chain and the inedible parts associated with food production.

## 1.1.1. Circular economy

In the EU, the food sector is the main manufacturing activity, accounting for about 294,000 companies, of which 95 % are small and medium-sized enterprises. The Spanish food industry ranks fourth in terms of revenue in the EU and is the leading manufacturing branch of the industrial sector in Spain, with more than 30,000 companies (MAPA, 2020).

The importance of the agri-food industry along with the environmental, economic, and ethical problems arising from food residues have driven the international community to design new strategies to reduce them. For instance, the EU has adopted a circular economy action plan which includes food and water as key product value chains (European commission, 2020).

This plan is based on the reduction, re-use, recovery, and recycling of materials and energy, enhancing the value and extending the life of the products (Faustino et al., 2019). It sets out to find new processes to make use of some food parts that were previously disregarded, if possible for consumption and if not, for other uses (Copa-Cogeca et al., 2020).

Specifically in Spain, the ministry responsible for ecological transition and the demographic challenge (MITECO) has established quantitative objectives to be achieved by 2030:

- Reduce the national consumption of materials by 30 % of GDP (Gross Domestic Product), taking 2010 as a reference.
- Reduce waste generation by 15 % compared to the residue generated in 2010.

- Reduce the generation of food residues in the entire food chain: 50 % reduction per capita at the household and retail consumption level and 20 % in the production and supply chains from 2020.
- Increase the re-use of municipal waste by 10 %.
- Improve water use efficiency by 10 %.
- Reduce the emission of greenhouse gases to below 10 million tons of $CO_2$ equivalent.

The agri-food industry has been chosen as a priority sector in the circular economy plan in Spain. In this context, the MAPA has developed the strategy "more food, less waste", which aims to reduce food residues and maximize the value of discarded food. The approach is through research and innovation, promoting agreements and collaboration with technological institutes and universities to develop the new technologies needed to reduce food residues (MAPA, 2018).

In fact, various researchers have recently proposed alternatives to maximize the value of food parts that are usually discarded. For instance, Castro et al. (2020) have produced a flour from orange juice by-products that can substitute wheat flour in the elaboration of biscuits. Pasqualone et al. (2020) have investigated the use of almond skins in the production of functional biscuits. Campos et al. (2020) and Skendi et al. (2020) have proposed a transition of fruit and cereal by-products, respectively, from a linear to a circular economy, by using them as a source of valuable bioactive compounds such as antioxidants.

## 1.2. Bioactive compounds in fruits, vegetables, and fungi residues

Bioactive compounds are extra-nutritional components that provide additional health benefits beyond the basic nutritional value of the product (Hamzalioğlu and Gökmen, 2016). These compounds can be found in small quantities in foods, mainly in fruits, vegetables, and whole grains (Santos et al., 2019). Among these foods, mushrooms are also worth mentioning. While not being vegetables, since they belong to the *fungi* kingdom, they have a unique composition including several bioactive compounds.

Recently, particular attention has been directed to the recovery of bioactive compounds from different agri-food residues (Carciochi et al., 2017). Peel, seeds, pulp, leaves, and pomace have been the fruit and vegetable by-products most commonly investigated as raw matter for the extraction of bioactive compounds in the last 5 years (Coelho et al., 2019; Hatami et al., 2020; Riciputi et al., 2018). Other by-products as bracts, straws, press cake, and kernels have also been investigated to a lesser extent (Pataro et al., 2017; Sabater et al., 2018; Sophonputtanaphoca et al., 2018; Wong et al., 2015). In the case of mushroom residues used as a source of bioactive compounds, they have consisted mainly of products discarded in the industry because of their irregular shape or size (Aguiló-Aguayo et al., 2017; Heleno et al., 2016b; Silva et al., 2020). However,

some studies have been also carried out using only mushroom stipes or stalks (Ahmed et al., 2017; Gil-Ramírez et al., 2013; Lin et al., 2010; Van Ba et al., 2017).

Several investigations have shown these agri-food residues to contain a wide range of bioactive compounds, including vitamins, minerals, proteins, antioxidants, polyphenols, lipids, polysaccharides among others (Carciochi et al., 2017; Faustino et al., 2019). Residues from fruits and vegetables are considered as a particularly rich source of cell wall material such as pectin, cellulose, and hemicellulose (Minjares-Fuentes, 2017). Some residues, such as those from artichokes, are also rich in an important storage polysaccharide called inulin (Glibowski and Skrzypczak, 2017; Yahia et al., 2018). Meanwhile, mushrooms are a rich source of polysaccharides like chitin, hemicelluloses, glucans, and heteroglycans (Ahmed et al., 2017; Chakraborty et al., 2019).

## 1.2.1.Polysaccharides

Polysaccharides are complex carbohydrate polymers formed by more than two monosaccharides linked by covalent glycosidic bonds. They are natural macromolecules usually located in the primary cell walls of plants and fungi. Their function in living organisms is usually related to either structure or storage (Lee, 2017; Muhamad et al., 2017).

### 1.2.1.1.Fruit and vegetable polysaccharides

The plant cell walls are composed mainly of polysaccharides which maintain the texture and hardness of the cell. The composition of the cell wall depends on the type or part of the plant, but the main structural polysaccharides that can be found in the primary wall are cellulose, hemicellulose, and pectins (Figure 1). Some cells also form a secondary wall that can contain lignin (Lerouxel et al., 2006; Voragen et al., 2009; Yahia et al., 2018).
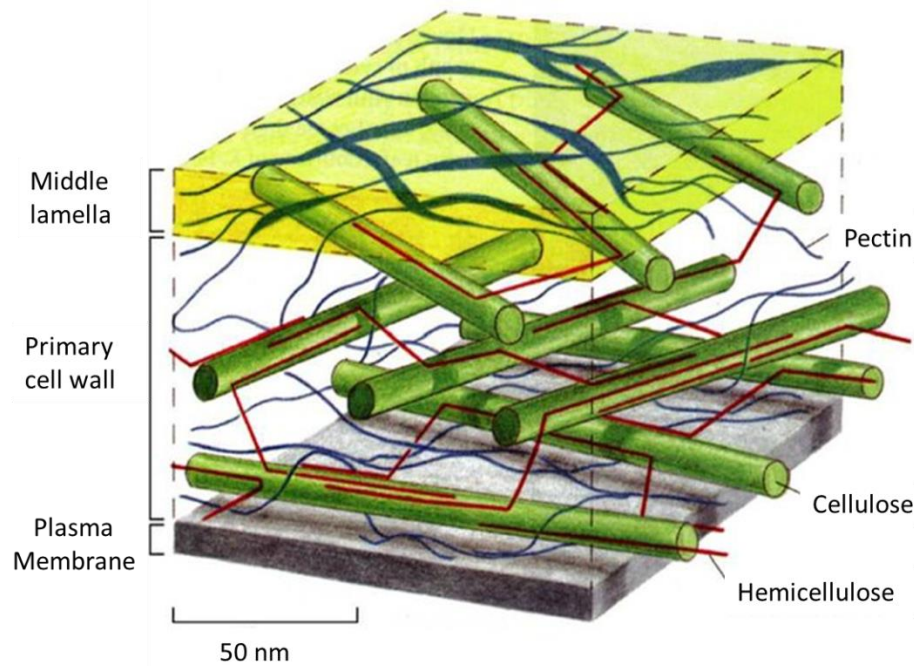
**Figure 1.** Diagram of the plant cell wall structure (Scheller and Ulvskov, 2010)

- *Pectic polysaccharides*

Pectic polysaccharides or pectins can be found in the cell wall and, also, in the middle lamellae of many plants (Figure 1). They are complex polysaccharides mainly composed of galacturonic acid that are methyl esterified to a variable extent (Minjares-Fuentes, 2017; Yahia et al., 2018). The main types of pectins are known as homogalacturonans (HG) and rhamnogalacturonans I (RG I). There are also relatively minor amounts of rhamnogalacturonan II (RGs II) (Lerouxel et al., 2006). These pectic polysaccharides are represented in the schema of Figure 2.

HG is a linear homopolymer consisting of α-(1-4) linked *D*-galacturonic acid residues. It is known as the "smooth" region of pectins and accounts for approximately 60–65 % of the total pectin amount (Chan et al., 2017; Dranca and Oroian, 2018). HG might be joined by one or two α-1,2-linked l-rhamnopyranose units. As well, but less frequently, galacturonic acid figures may be substituted at the C-2 or C-3 positions with residues of xylose or apiose, which produces domains known as xylogalacturonan or apiogalacturonan, respectively (Voragen et al., 2009).

RG-I is considered the "hairy" region of pectins. RG-I contains 100 or more repeating units of the disaccharide α-1,2-linked-l-rhamnose-α-1,4-d-GalA (Chan et al., 2017). Rhamnose residues in RG I can be substituted at the C-4 position with neutral sugar side chains. These neutral sugars are predominantly galactose and arabinose, but glucose, mannose, fucose, xylose, and galacturonic acid have also been found covalently linked to the backbones as side chains (Arnous and Meyer, 2009; Chan et al., 2017).

Rhamnogalacturonan II (RG II) is a highly conserved and complex structure in plants, not structurally related to RG I, despite their names. The structure is a distinct region within HG with four different side chains. These side chains contain eleven different glycosyl residues including galacturonic acid, rhamnose, arabinose, galactose, glucuronic acid, and some rare sugars such as apiose, 2-*O*-methyl-D-fucose, 2-*O*-methyl-D-xylose, 3-*C*-carboxy-5-deoxy-l-xylose (aceric acid), 3-deoxy-D-manno-octulosonic acid (KdoA), and 3-deoxy-D-lyxo-heptulosaric acid (DhaA) (Chan et al., 2017; Voragen et al., 2009; Yapo, 2011).

An important characteristic of pectic polysaccharides is the esterification by methyl groups (at C-6) and/or acetyl groups (at O-2 and/or O-3) of galacturonic acid residues of HG. The degree of substitution of methyl groups is known as the degree of methylation (DM). Based on the DM, pectins are classified into two categories: high-methoxyl pectins (HMP, DM>50 %) and low-methoxyl pectins (LMP, DM<50 %). This parameter affects the potential pectin application in the industry (Chan et al., 2017) because it influences the conditions that must be applied for pectins to form a gel.
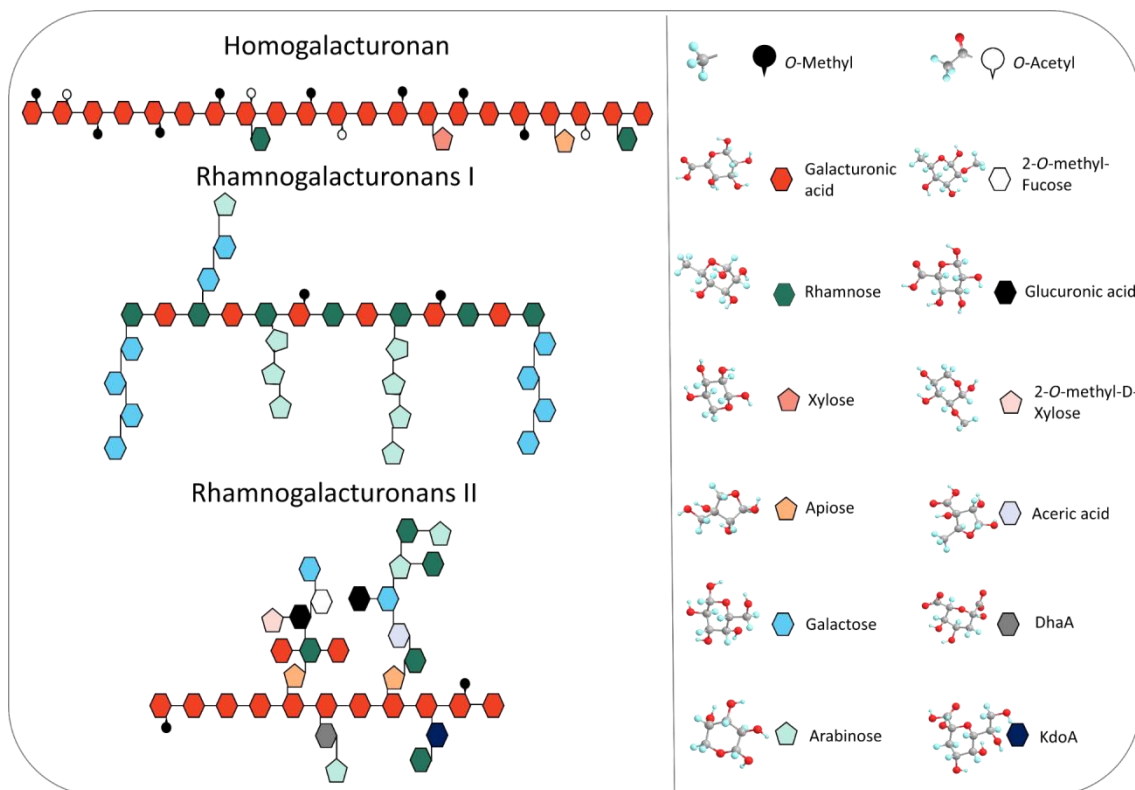


**Figure 2.** Schematic representation of pectic polysaccharides

- *Cellulose*

Cellulose is the most abundant structural component of primary cell walls in green plants, algae and some bacteria. It is a homopolymer composed of glucose monomers, which are linked via β-(1→4) glycosidic linkage (Mudgil, 2017). Cellulose chains are held in a crystalline lattice within the microfibril. The lattice is stabilized by intramolecular

and intermolecular hydrogen bonds conferring tensile strength on the cell walls (T. Brett and W. Waldron, 1990). A schematic representation of cellulose structure is shown in Figure 3.
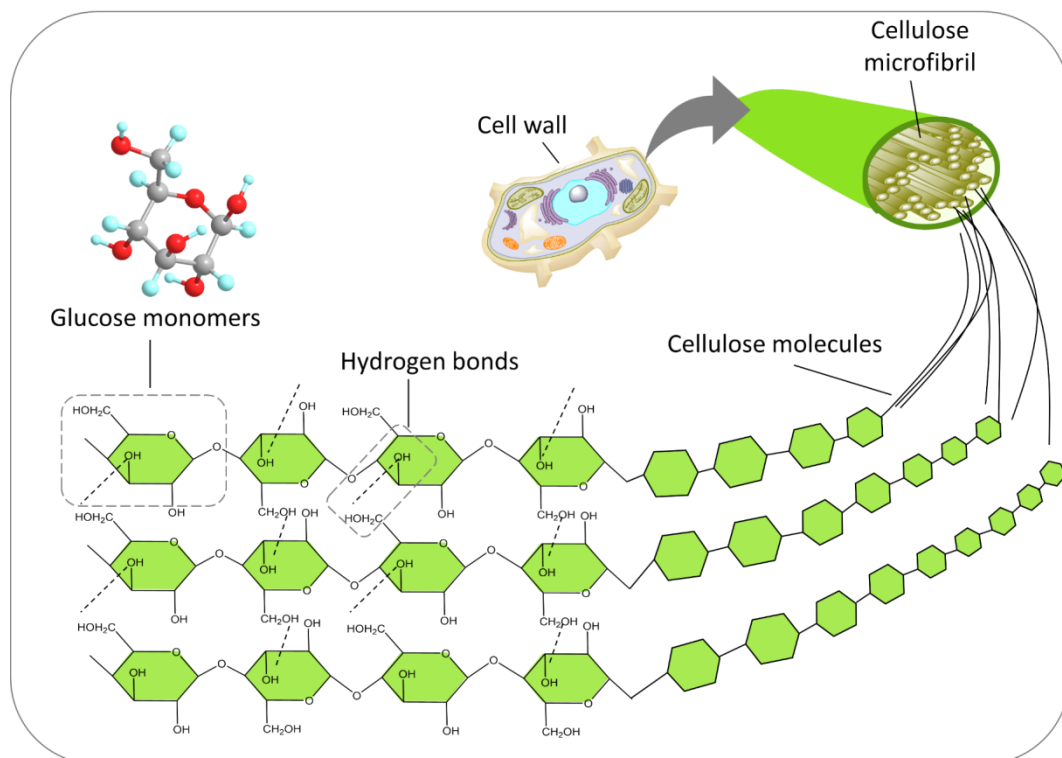


**Figure 3**. Schematic representation of cellulose

- *Hemicelluloses*

Hemicelluloses are the second most abundant chemical constituent of the cell wall of plants after cellulose (Li et al., 2013). They include several heteropolymers derived from monosaccharides such as xylose, galactose, mannose, glucose, and arabinose. The backbones are composed of β-(1→4)- glucose, mannose, or xylose. The differing classes of hemicellulose are named according to their main monosaccharide unit (Ebringerová, 2005; Frassoldati and Ranzi, 2019).

They are usually classified into three groups of structurally different polysaccharide types: xyloglucans, xylans, and mannans. Xyloglucans is the most abundant hemicellulose in the primary cell walls of all higher plants. Xyloglucans' backbone consists of β−(1→4) linked glucose with xylose residues attached at C-6. Some of the xylose attached to the backbone may exhibit galactose residues linked to C−2, and more rarely arabinose residues may be observed attached to xylose. Galactose monomers can also be linked to fucose (Ebringerová, 2005; Li et al., 2013; Minjares-Fuentes, 2017).

Xylans are a diverse group of polysaccharides with the common characteristic of a backbone of β-(1→4)-linked xylose residues. A common type of xylans is

glucuronoxylans which have glucuronic acid residues attached to xylose at position 2 (Ebringerová, 2005; Scheller and Ulvskov, 2010).

Mannans include different types of polysaccharides present in the soft tissues of plants. They have a backbone consisting of mannose (mannans), or mannose and glucose in a non-repeating pattern (glucomannans) that may have galactose attached to mannose (C-6) (galactomannan or galactoglucomannan) (Minjares-Fuentes, 2017; Scheller and Ulvskov, 2010). A schematic representation of the main types of hemicelluloses is shown in Figure 4.
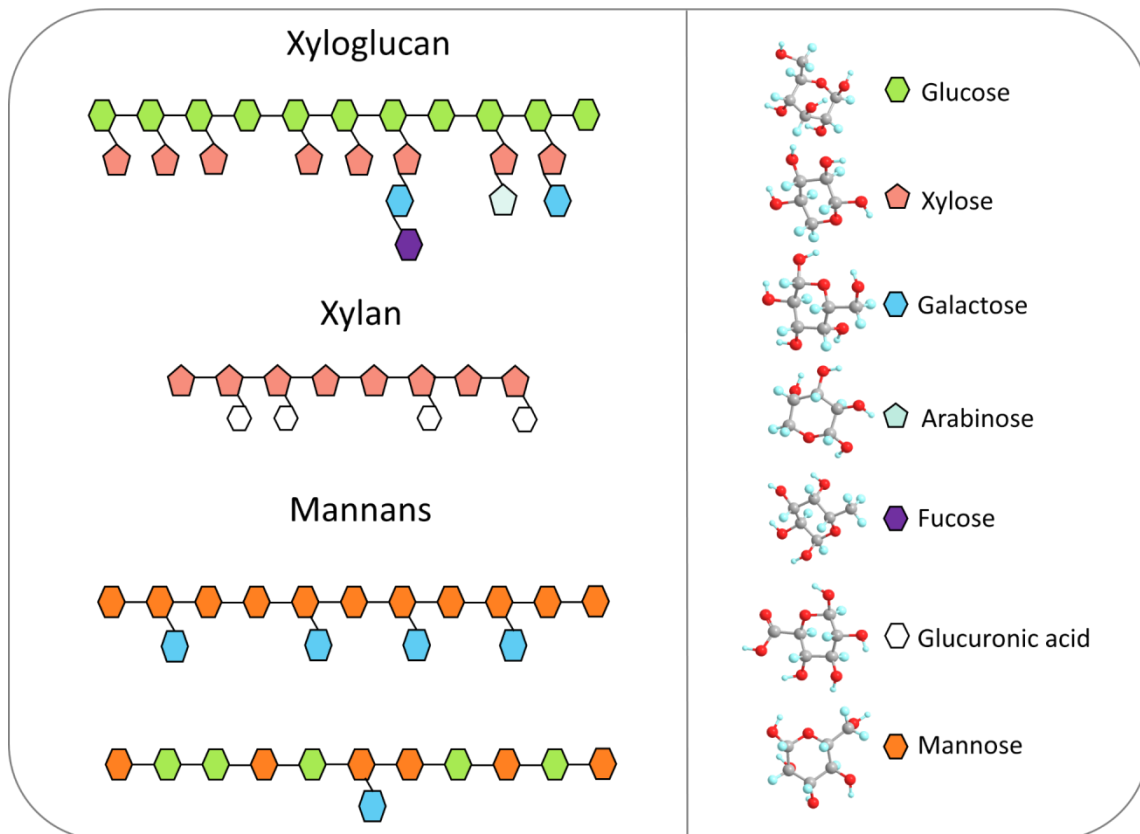


**Figure 4.** Schematic representation of the main types of hemicelluloses

- *Storage polysaccharides in fruits and vegetables*

Besides structural polysaccharides, plant cells also have storage polysaccharides which are the main source of calories for some plant organs. Most of these polysaccharides are intracellular, and starch is the most common, followed by inulin (Glibowski and Skrzypczak, 2017).  Inulin is the global name for all β -(2,1) fructans and is built up of 2–60 fructose units with one terminal glucose molecule (Meyer and Blaauwhoed, 2009).

### 1.2.1.2. Fungi polysaccharides

Fungi are composed of microscopic structures called hyphae (singular hypha), with the shape of a cylindrical tube. They are partitioned into cells by transverse walls of septa (singular septum). To carry out their different functions, such as spreading through soil

or growing erect, hyphae must generate high pressure and therefore have strong cell walls. Mushroom cell walls consist of three major biopolymers, which are cross-linked to each other: chitin, β-glucans, and mannoproteins. A schematic representation of the fungi structure and its cell wall is represented in Figure 5.
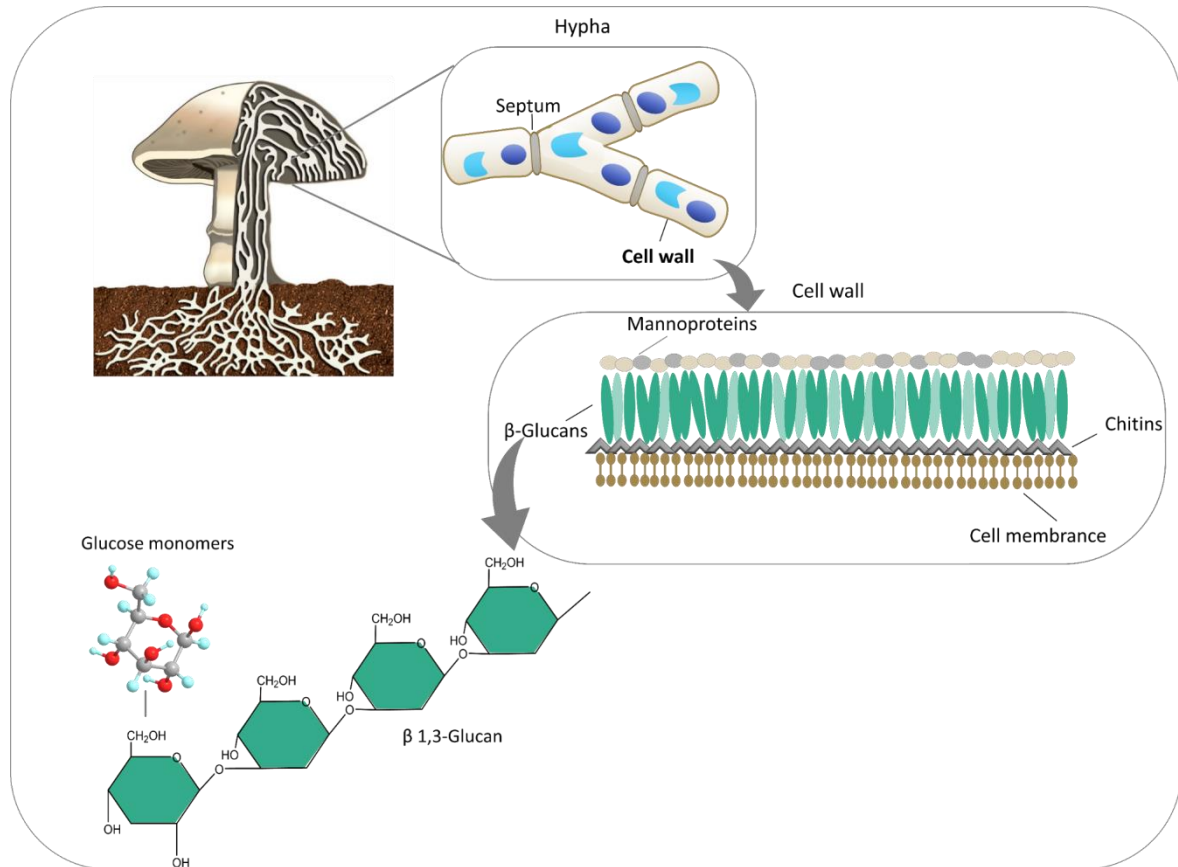


**Figure 5.** Schematic representation of the fungi structure

- *Chitin*

Chitin is an important component of the fungi cell wall, comprising approximately 10 % of the cell wall components. It is a polymer of β-(1→4) branched N-acetylglucosamine units, and contributes to the mechanical strength of the cell wall (Ohno, 2007; Paudel et al., 2016).

- *Glucans*
  - β-Glucans

β-Glucans from fungi are built of β-(1→3) and (1→6) linked D-glucose monomers (Zhu et al., 2015). These polysaccharides have been classified into two groups: linear and branched β-Glucans. Linear β-Glucans can be built from a single type of linkage ( β-(1→3) (which would be the most abundant in fungi) or they could be built from more than one type.

Given the microfibrillar structure of the most abundant β 1,3-glucans (Figure 5), it could be deduced that they have an important role in the architecture and resistance of the cell wall (Ruiz-Herrera and Ortiz-Castellanos, 2019). In the case of branched β-glucans, β -(1→3) with branches of β -(1→6) are the most common.

o α-Glucans

The most common α-glucans in fungi is glycogen which consists of glycosyl units joined by α-(1→4) and α-(1→6) bonds. Glycogen is a material of energy reserve that is accumulated in the form of grains in the cytoplasm. There are also α-glucans in which glucose can be linked exclusively by α-(1→3) bonds, or both α-(1→3) and α-(1→4). (Ruiz-Herrera and Ortiz-Castellanos, 2019).

- *Mannoproteins*

Mannoproteins are defined as glycoproteins that contain 15 to 90 % mannose by weight. They are found on the surface of the cell wall and can be connected via either covalent or non-covalent linkages to β-glucans (Butkhup et al., 2018; Kang et al., 2018).

### 1.2.1.3. Polysaccharide applications in the industry

Polysaccharides have an enormous impact in different areas of industry (such as food, cosmetics, and pharmaceutical) (Lapasin et al., 1995). These macromolecules stand out because of their healthy and nutritional properties as well as the functional characteristics that make them interesting for several technological applications.

Polysaccharides belong to an important nutritional group called dietary fibre, which is defined as storage and cell wall polysaccharides that cannot be hydrolyzed by human digestive enzymes (Van Horn et al., 2001). Dietary fibre is usually fermented in the large intestine, helping to maintain the microflora (Tosh and Yada, 2010), and is usually classified according to its solubility in water. Thus, soluble fibre consists of polysaccharides as pectins, some β-glucans, and inulin; while non-soluble fibre includes cellulose, hemicellulose, and lignin (Mudgil and Barak, 2019). These two types of fibre have distinct physiological functions. Insoluble fibre promotes the movement of material through the digestive system. Soluble fibre, on the other hand, helps to decrease blood cholesterol and regulate glucose in the blood. It has been found to lower the risk of ischemic heart disease, possibly through decreasing cholesterol levels. It has also been associated with a lower risk of diabetes type 2 and colorectal cancer (Park, 2016). More specifically, pectins have been found to moderate the glycemic index and help to control energy intake by slowing the gastric transit (Wicker and Kim, 2015). Other properties such as antitumor, anti-inflammatory, and immunostimulatory have also been associated with pectins (Khedmat et al., 2020). Another example would be inulin which has considerable prebiotic action and low caloric value, among other healthy properties (Shoaib et al., 2016).

Recently, fungi polysaccharides have attracted a lot of attention. For instance, β-glucans coming from different species of mushrooms have been investigated because of their hypocholesterolemic, immunomodulatory, antioxidant, and antitumoral activity (Khan et al., 2018; Morales et al., 2020).

Several studies have concluded that dietary fibre is an essential constituent of a healthy diet (Park, 2016). According to the European Food Safety Authority (EFSA), in adults, the recommended amounts of dietary fibre range from 25 to 38 g/day. Daily dietary fibre intakes vary broadly among different European countries (12-29 g/day). However, it is reasonable to deduce that many citizens do not consume enough fibre (EFSA, 2016). The addition of polysaccharides to foods such as bakery, dairy, meat, or snack products has been investigated recently as an alternative way of improving dietary fibre consumption (Ballester-Sánchez et al., 2020; Das et al., 2020; Keerthana et al., 2020).  It is interesting because apart from their diverse physiological functions, polysaccharides have important functional properties. Most notable is their ability to modify the characteristics of aqueous environments, acting as thickeners, emulgents, stabilizers, gelling agents among others (Lapasin et al., 1995). Thus, polysaccharides play an important role in controlling the texture of food, as well as affecting its appearance and other sensorial characteristics (Sandford and Baird, 1983).

For instance, pectins are known as gelling agents in the food industry. A pectin gel is formed when portions of the HG chain are crossed-linked to construct a three-dimensional crystal network in which water and solutes are trapped (Willats et al., 2006). The gel formation depends on the pectin type, thus HMP forms gels in the presence of high concentrations of sugars. This is because sugar molecules remove water molecules binding to the hydroxyl groups of HMP chains, resulting in the interconnection of the HMP chain. On the other hand, LMP forms gels in the presence of divalent ions (such as $Ca^{+2}$), typically at pH values in the range 3–5, since junction zones are formed by divalent ions cross-linking between free carboxyl groups. Due to these properties, pectin is used in the production of jams and jellies (HMP) and also to stabilize acidified milk drinks and yoghurts (LMP) (Chen et al., 2021; Li et al., 2019; Willats et al., 2006).

Similarly, it has been observed that β-glucans coming from mushrooms can form a network structure through chain segment association, trapping solutes or particles such as oil droplets (Veverka et al., 2018). Generally, polysaccharides are able to thicken aqueous systems, increasing viscosity and hindering the undesired movement of particles. Furthermore, polysaccharides in their native state may be attached to proteins that provide the structure of amphiphilic properties enhancing its emulgent capacity (Shao et al., 2020).

## 1.2.2.Antioxidant compounds

In biological systems, reactive oxygen species and/or reactive nitrogen species can lead to the oxidation of lipids and proteins in cells (Xu et al., 2017). To survive, cells prevent natural oxidation by producing antioxidants, which are substances that can delay the autoxidation onset or slow its rate. Antioxidants inhibit the formation or interrupt the propagation of free radicals by one (or more) mechanisms: 1) scavenging species that initiate peroxidation, 2) chelating metal ions preventing them from generating reactive species or decomposing lipid peroxides, 3) quenching $*O_2$ avoiding the formation of peroxides, 4) interrupting the autoxidative chain reaction, and/or 5) reducing localized $*O_2$ concentrations (Asimi et al., 2013). The effectiveness of an antioxidant is related to many factors, including its chemical characteristics such as activation energy, rate constants, oxidation-reduction potential, ease of antioxidant loss or destruction, and solubility properties (Fennema, 1996).

### 1.2.2.1.Antioxidant compounds in fruits and vegetables

The main natural antioxidants present in fruits and vegetables are phenolic compounds, carotenoids, and vitamins (vitamin E and C) (Xu et al., 2017).

- Phenolic compounds

Phenolic compounds are found in plants as secondary metabolites (Naczk and Shahidi, 2004). This means they do not take part in the photosynthetic or respiratory metabolism but are responsible for the plant's defence against pathogens and predators. They also contribute to the colour and other sensorial characteristics of fruits and vegetables (Vuolo et al., 2018).

The phenolic compound structure consists of an aromatic ring with one or more hydroxyl substituents. They show a great diversity of structures, ranging from quite simple molecules (monomers and oligomers) to polymers. Furthermore, phenolic compounds mostly occur naturally as conjugates with mono and polysaccharides (Cheynier, 2005; Vuolo et al., 2018). Flavonoids and several classes of nonflavonoids generally stand out among plant phenolic compounds.

  o Flavonoids

Flavonoids constitute the largest group of phenolic compounds in plants; more than 6000 compounds have been identified within this group. They are made up of two phenolic rings and an oxygenated heterocycle; a generic structure can be seen in Figure 6. Depending on the state of oxidation of the heterocyclic pyran ring, flavonoids can be classified into different groups such as anthocyanidins, flavonols, and flavanols.
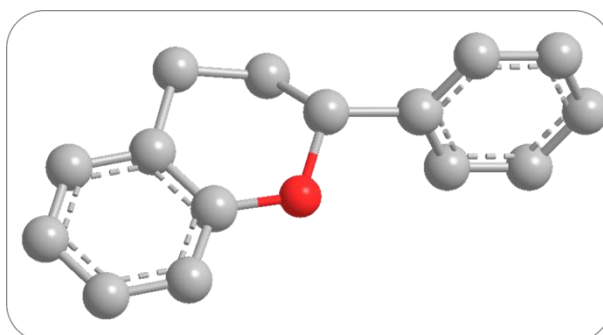
**Figure 6.** Generic chemical structure of flavonoids

o   Non-flavonoids

Nonflavonoids, on the other hand, are mostly simple molecules, such as phenolic acids. These compounds are subdivided into derivatives of hydrobenzoic acids, such as gallic acid, and derivatives of hydrocinnamic acids, such as caffeic acid (Dai and Mumper, 2010). The chemical structure of the main phenolic acids can be observed in Figure 7. Caffeic acid is the most abundant phenolic acid in many fruits and vegetables. It is often esterified with quinic acid as in chlorogenic acid (Sato et al., 2011) which has shown great antioxidant activity. Stilbenes and lignans, on the other hand, are less common types of nonflavonoids (Dai and Mumper, 2010).



Hydroxibenzoic acid                    Hydroxicinnamic acid

| Hydroxibenzoic acids | $R_1$ | $R_2$ | $R_3$ | $R_4$ | Hydroxicinnamic acids |
|---|---|---|---|---|---|
| ρ-Hydroxibenzoic | H | H | OH | H | ρ-Cummaric |
| Protocatechuic | H | OH | OH | H | Caffeic |
| Vanillic | H | $OCH_3$ | OH | H | Ferulic |
| Syringic | H | $OCH_3$ | OH | $OCH_3$ | Sinapic |
| Gallic | H | OH | OH | OH | |
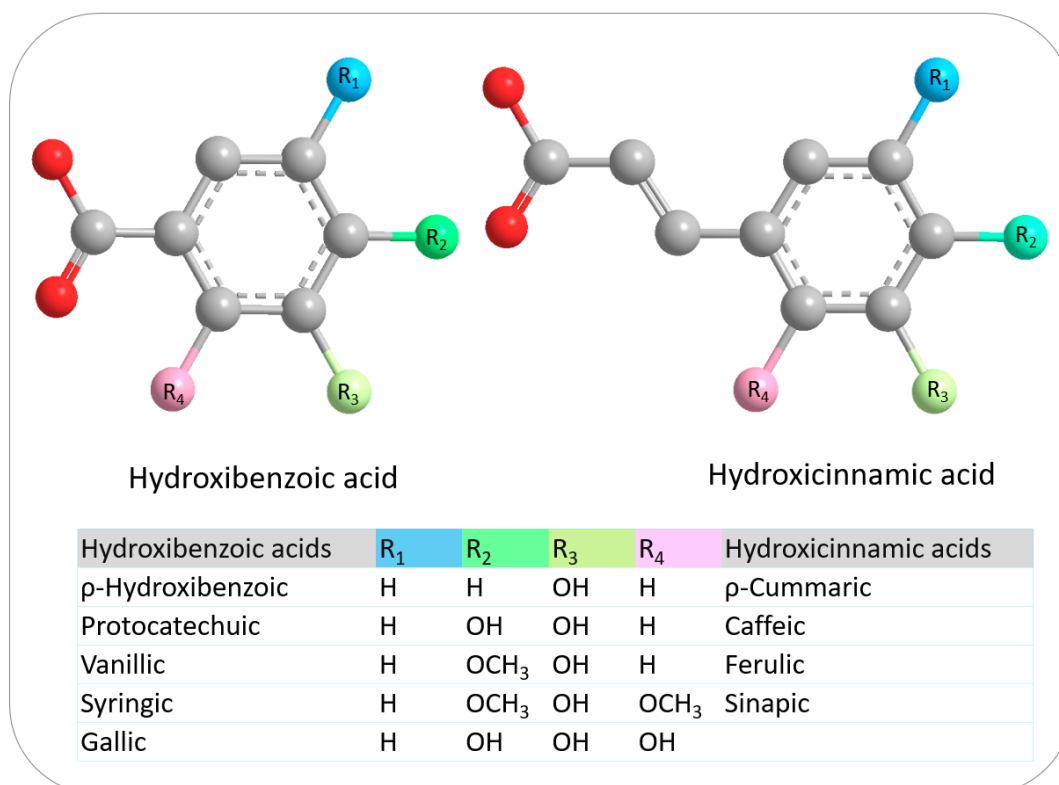
**Figure 7.** Chemical structure of the main phenolic acids

*1.2.2.2.Antioxidant compounds in fungi*

Secondary metabolites of higher fungi (mushrooms) have not been explored as much as those of plants. However, there is an increasing interest in mushrooms as a source of antioxidant compounds (Chen and Liu, 2017). They are rich in phenolic compounds, carotenoids, vitamins E (tocopherol), and C, and a provitamin of vitamin D called ergosterol (Corrêa et al., 2018; Sánchez, 2017). Ergosterol stands out among the rest of the bioactive compounds because mushrooms are one of the few sources of this provitamin. Moreover, some authors state that ergosterol exists only in the fungi kingdom (Guan et al., 2016).

- Phenolic compounds

Phenolic acids are the most abundant phenolic compounds in mushrooms. As with those of plants, phenolic acids of mushrooms can also be classified as hydroxybenzoic acids or hydroxycinnamic acids. The phenolic acids present in Figure 7 have been detected in both plants and mushrooms (Dinesh, 2021).

However, there is some controversy about the presence of flavonoids in mushrooms. Several studies have determined the total flavonoid content in mushrooms using colourimetric methods, while Gil-Ramírez et al. (2016) have demonstrated that mushrooms cannot synthesize flavonoids since they do not have the required enzymes. Moreover, phenolic acid nonflavonoids have shown a positive response to some colourimetric methods used for the determination of flavonoids (Nowacka-Jechalke et al., 2018).

- Ergosterol

Ergosterol is the main sterol in higher fungi (mushrooms). It is analogous to the cholesterol of mammalian cells and thus has similar functions in the strengthening of cell membranes, modulation of membrane fluidity, and intracellular transport (Barreira et al., 2014). Ergosterol has been associated with the growth and maturation of mushrooms and hyphae formation. Its structure, presented in Figure 8, has two double bonds in the sterol ring, which differentiates it from other sterols such as those coming from plants (Bell, 2006).

With regard to its bioactivity, ergosterol has great importance as it can be converted into vitamin $D_2$ (ergocalciferol) after photolysis and thermal rearrangement (Bell, 2006). Vitamin D has important functions for human health as it facilitates the synthesis of calcium and enhances its absorption, reducing the risk of osteomalacia. It is also necessary for correct muscle function (Cardwell et al., 2018), as well as being associated with healthy properties such as antioxidant, anti-inflammatory, and anti-hyperlipidemic activities (Barreira et al., 2014; Kuo et al., 2011; Shao et al., 2010; Shu et al., 2006).
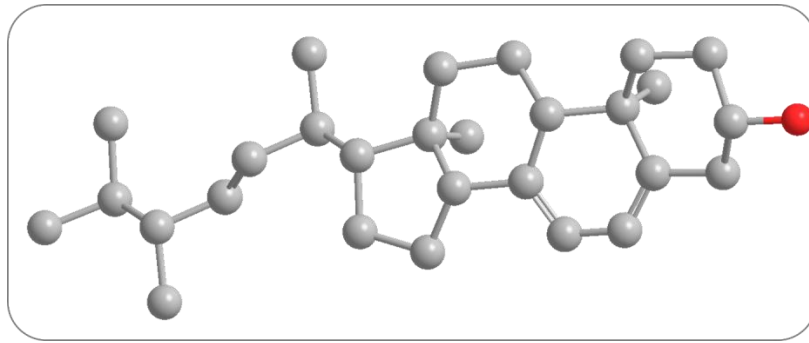
**Figure 8.** Chemical structure of ergosterol

### 1.2.2.3. Antioxidant compounds in industry

Food product deterioration is closely connected to oxidation reactions. Industrially produced foods are not immediately consumed, and so there is a constant search for strategies to ensure these products maintain their quality and safeness through transport and storage to delivery to consumers. The addition of antioxidant compounds is one of the most commonly applied strategies to prevent or delay oxidative damage (Lourenço et al., 2019).

Therefore, both natural and synthetic antioxidants are widely used in the food industry as food additives. In fact, antioxidants constitute one of the functional classes of food additives according to the EFSA (EU, 2008). In this regulation, antioxidants are defined as "*substances which prolong the shelf-life of foods by protecting them against deterioration caused by oxidation, such as fat rancidity and color changes*". These compounds are usually added to fats and oils since lipid oxidation is highly undesirable, leading as it does to rancidity and potential toxicity (Kebede and Admassu, 2019). Moreover, lipid oxidation is one of the main causes of deterioration of a variety of foods including meat-based products (de Paiva et al., 2021). Antioxidants are also added to mayonnaise, dairy products, vegetable oils, animals fats, and so forth (Shahidi, 2015).

There are hundreds of compounds, both natural and synthesized, which have shown antioxidant activity (Fennema, 1996). However, nowadays consumers have shown a clear preference for food products free of synthetic ingredients since there is a concern that long-term intake of non-natural additives could be harmful to human health (Román et al., 2017).

Further, epidemiological investigations have repeatedly demonstrated an inverse relationship between the risk of chronic human diseases and the consumption of a polyphenolic rich diet (Pandey and Rizvi, 2009). Anti-inflammatory, anti-ageing, anti-atherosclerosis, and anticancer are some of the biological activities observed in natural antioxidant compounds (Bollati et al., 2006; Wojdyło et al., 2018; Zhang et al., 2015).

## 1.3.Solid-liquid extraction

Solid-liquid extraction is a mass transfer operation based on the preferential dissolution of one or more of the components of a solid mixture in a liquid solvent (Berk, 2009). This separation process is widely used in the food industry. Among other applications, it has an important role in the isolation of bioactive compounds coming from fruits, vegetables, and fungi tissues (González-Centeno, 2013; Sun et al., 2019).

### 1.3.1.Fundamentals

The solid-liquid extraction involves two stages, an initial washing-out step, which corresponds to a rapid dissolution of the solute from the solid surface or broken cells; and the second stage of diffusion, which consists of a transfer of solute molecules from inside the solid to the solvent. The diffusion stage is a much slower and more limited step for most of the solid-liquid processes (Cheung and Wu, 2013; Sturzoiu and Stroescu, 2011).

One of the key factors in the extraction of bioactive compounds is the election of a suitable solvent, as several of its characteristics such as polarity, pH, or even toxicity could affect the process (Minjares-Fuentes, 2017). The election of the extraction solvent should depend on the target compound. For instance, pectin is usually extracted with acids such as HCl, $HNO_3$, or $H_2SO_4$ (Colodel et al., 2018; Tang et al., 2011; Wang et al., 2015). Acid solvents allow the extraction of insoluble pectin that is tightly bound to the cell matrix of the plant material,  improving the extraction yields (Assoi et al., 2014). Another example would be some antioxidant molecules such as phenolic compounds or sterols; in these cases, the polarity of the solvent needs to be adjusted to get higher extraction yields (Gil-Ramírez et al., 2013; Yang et al., 2009). However, the effluents of these extraction procedures generate elevated management costs and a serious environmental problem (Minjares-Fuentes, 2017). Moreover, the use of toxic solvents is highly detrimental to the further application of the extracted compounds in the food or pharmaceutical industries. From this point of view, replacing toxic solvents with aqueous or, at least, less harmful solvents would be a great advantage.

In the extraction of bioactive compounds from fruit and vegetables or fungi materials, there is a natural resistance of the cell walls to the solvent penetration, which hinders the mass transfer (Cissé et al., 2012). The diffusivity of the solute into the solvent could be increased by using higher temperatures. However, this means higher energy consumption and the loss of bioactive compounds that are susceptible to temperature (Cacace and Mazza, 2003; Diaz et al., 2007; Kadakal and Artik, 2008; Sharma et al., 2015). Thus, other alternatives should be evaluated to increase extraction yields.

### 1.3.2. Intensification of extraction processes

Intensifying a conventional process consists of modifying it by developing new technologies to reduce energy needs, increase yields, and/or increase the quality of the

final product. This is an area of great potential in the food industry because, besides improving the process efficiency, it also has consequent environmental benefits (Rodríguez Barragán et al., 2014).

Specifically, in the case of extraction processes, the intensification is pursued through sustainable techniques that increase the extraction yields. Among the techniques evaluated in recent years are microwave-assisted extraction (Heleno et al., 2016b), ultrasound-assisted extraction (González-Centeno et al., 2014), supercritical fluid extraction (Pinto et al., 2020), pulsed electric field extraction (Xue and Farid, 2015), and pressurized liquids extraction (Gil-Ramírez et al., 2013). All these methodologies aim to reduce the amount of solvent and energy requirements, shorten the extraction time, increase the extraction yield and improve the quality of the extracts (Wang and Weller, 2006).

The ultrasound-assisted extraction (UAE) technique is probably one of the most studied at both laboratory and industrial scale. This might be due to its great versatility, the capacity to extract using less or no organic solvent, its simplicity of operation, its relatively cheap equipment, the ability to work at lower temperatures, and the preservation of the biological activity of the extracted compounds (Cheung et al., 2013; Kumar et al., 2021).

### 1.3.1.1. High-power ultrasound

Acoustic waves are defined as mechanical waves that require a material medium to propagate. Like any other kind of wave, acoustic waves can also be characterized by several parameters, the frequency, intensity, power, power density, and attenuation being some of the main ones. Frequency is the number of cycles or vibrations completed in a unit of time, usually expressed in hertz (Hz). The intensity is defined as the energy transmitted per unit of time through a unit of area perpendicular to the propagation direction and can be expressed as $W/m^2$. The acoustic power is the total energy transmitted by the ultrasonic source to the medium per unit of time, calculated from the product of the intensity and radiant surface area, and is expressed as W. The power density is the amount of power dissipated per unit of volume of a given medium expressed as W/L. The attenuation is the loss of energy as the waves pass through a medium (García-Pérez, 2007; Sivakumar and Pandit, 2001).

According to their frequency, acoustic waves are usually classified by taking the human audible frequency as a reference, thus, placing it between 20 Hz and 20 kHz (Figure 9). Lower frequencies are known as infrasound and higher frequencies as ultrasound (Cárcel et al., 2012).
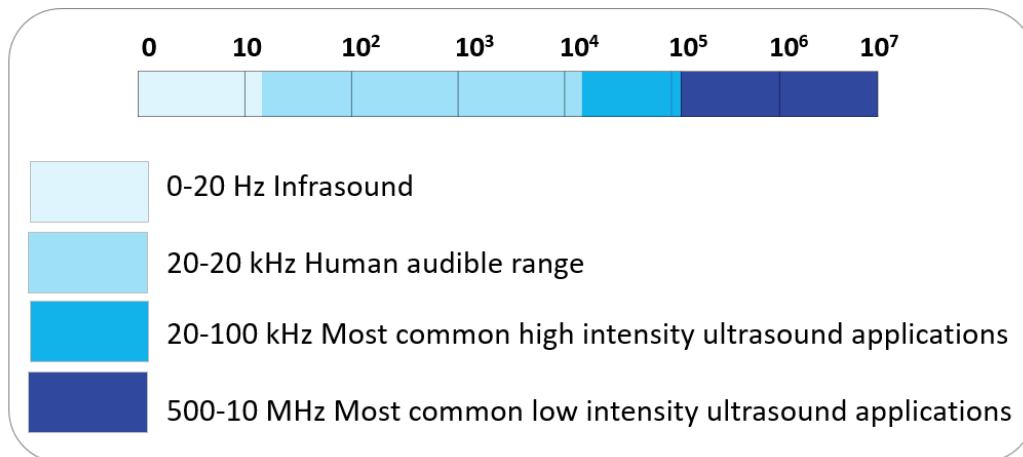
**Figure 9.** Sound classification according to its frequency

Ultrasound is classified into low and high-intensity ultrasound. Low-intensity ultrasound has frequencies higher than 100 kHz and intensities below 1 W/cm$^2$. They are transmitted through a medium without causing any changes to it, but the ultrasound waves suffer changes in their properties (velocity, attenuation, frequency spectrum, and so forth). These variations are used in diagnosis applications such as medical diagnosis, industrial monitoring and control, and depth sounding among others (Cárcel et al., 2012; Musielak et al., 2016). On the other hand, when ultrasound waves have intensities higher than 1 W/cm$^2$ and frequencies in the range of 20-100 kHz, they can be classified as "high-power ultrasound" or "high-intensity ultrasound". This type of ultrasound induces changes in the physical, chemical, and biochemical properties of the medium where it is applied.

When acoustic waves pass through a liquid, they cause a fluctuation of pressure. During compression cycles, ultrasound waves generate positive pressure leading the molecules to approach each other. And during rarefaction, the negative pressure caused by ultrasound waves tends to pull the molecules apart. In the case of high-intensity ultrasound waves, the negative pressure during rarefaction exceeds the attracting force that keeps the molecules together and pulls them apart creating cavitation nano/microbubbles (Esclapez et al., 2011; Kumar et al., 2021).

During the cycles of rarefaction, the cavitation bubbles grow, since there is a flux of gas from the liquid to the bubble. And during the compression, the opposite occurs, as the flux of gas goes from the bubble to the liquid. Therefore, the bubble' size oscillations could be regular if the flux of gas bubble-liquid is similar in both directions. This is known as stable cavitation. However, this is only possible when the ultrasound intensity is not high enough to provoke transitory cavitation, which consists of the progressive growth of the bubble resulting in a violent collapse when the bubble reaches a critical size (González-Centeno, 2013). These violent implosions generate pressures about 100 atm and temperatures about 4700 °C. Thus, cavitation bubbles last for only a few cycles

before collapsing and giving rise to smaller bubbles that could be a new cavitation nucleus (Esclapez et al., 2011; Kumar et al., 2021). A schematic representation of this process can be seen in Figure 10.
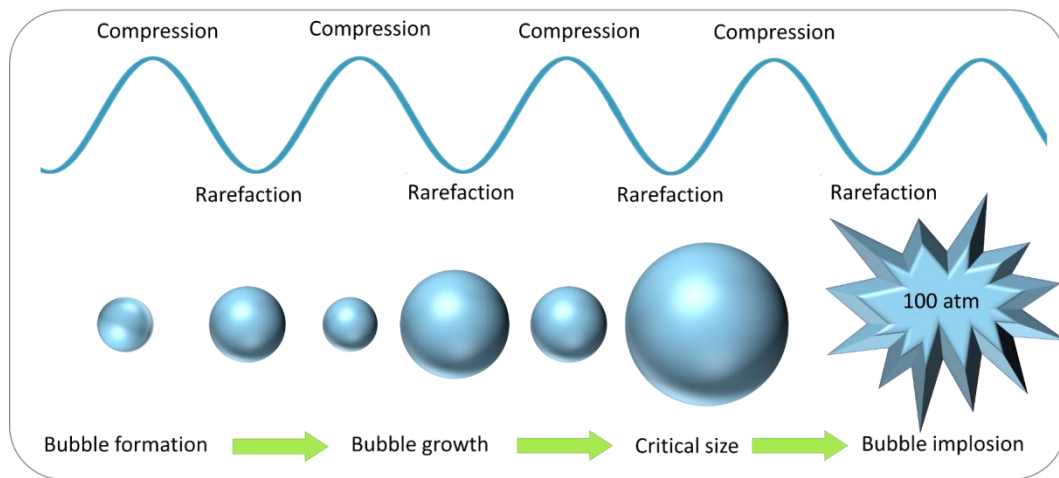


**Figure 10.** Schematic representation of cavitation bubble formation and implosion (García-Pérez, 2007)

The implosion of cavitation bubbles provokes several mechanisms such as shear forces and turbulences (Wen et al., 2018). Moreover, in a solid-liquid system, when the cavitation bubbles collapse asymmetrically close to a solid surface, they generate microjets that hit the solid surface and produce an injection of fluid inside the solid (Kek et al., 2013). This phenomenon can cause important changes in the solid characteristics such as erosion of the surface, microfractures or even particle breakdown (Vilkhu et al., 2008). A schematic representation of cavitation bubbles implosion close to a solid surface can be seen in Figure 11.

Generally, the production of ultrasound consists of converting any other kind of energy into acoustic energy. Thus, the equipment used to transmit high-intensity ultrasound is composed of three fundamental parts: a generator of energy, a transducer, and a transmitter. The generator transforms the electric energy into the desired frequency. The transducer converts the electric energy into acoustic energy with the required characteristics of intensity and frequency. Finally, the transmitter radiates the acoustic energy to the medium (García-Pérez, 2007).
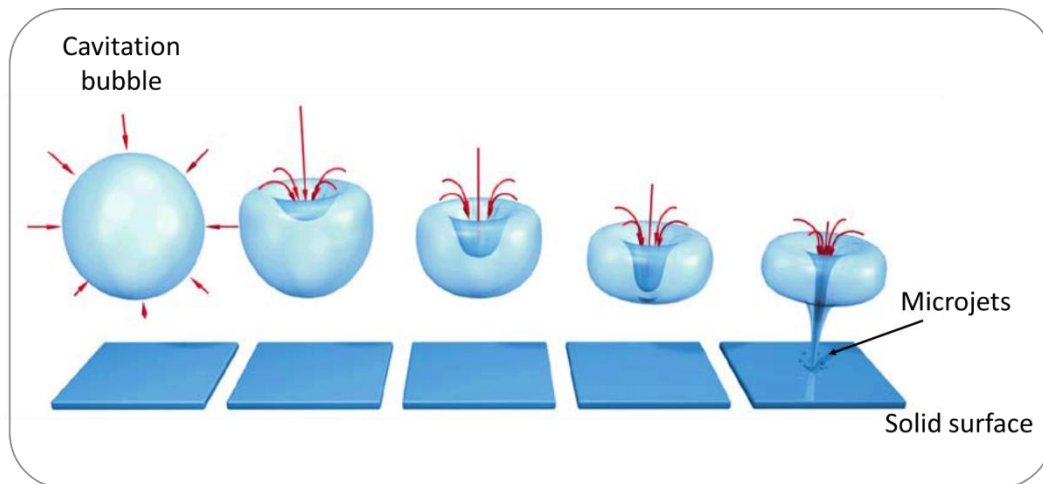
**Figure 11.** Microjet formation as a result of bubble cavitation in a solid-liquid interface when applying high-power ultrasound (González-Centeno, 2013)

The two main types of systems for ultrasound application to liquids are ultrasound baths and probe systems. Ultrasound baths are probably the most widely used equipment and consist of a stainless-steel container with the transducer placed in the base. Their main limitation is the little power they supply to the medium (García-Pérez, 2007).

On the other hand, probe systems use a metal probe to transmit the energy to the medium. The applied power depends on the vibration amplitude at the probe tip which can be controlled by varying the electrical power supplied by the generator or amplifier. The shape and design of the probe are of great importance; therefore many systems are designed to work with interchangeable probes (García-Pérez, 2007).

### 1.3.1.2. Ultrasound-assisted extraction of bioactive compounds

Several studies have demonstrated that ultrasonic technology improves the performance of such food industry operations as extraction, dehydration, emulsification, modification of food components, among others (Fu et al., 2020). The intensification of the solid-liquid extraction of bioactive compounds from natural materials is one of the main applications of high-power ultrasound. Traditional extraction techniques including Soxhlet, maceration, heat reflux, and solid-liquid extraction by mechanical agitation, have many limitations such as high energy requirements, and solvent consumption, long periods of processing time, and low extraction yields (Ojha et al., 2020; Wen et al., 2018).

Ultrasound assistance can enhance and accelerate the solid-liquid extraction process because of mechanisms originating in the cavitation effect. According to Mason et al. (2011), there are four mechanisms caused by ultrasound that can improve solid-liquid extraction efficiency:

- *Breakdown of cells.* Acoustic cavitation provides enough energy to break down the plant material as a result of microjet formation. This increases the

permeability of the cell walls or can even release the cell content. The mechanical action can also reduce the particle size of the solid allowing much more surface in contact with the extraction solvent.

- *Increase of the target compound solubility.* Facilitated by the localized increase of the temperature in the zone, the cavitation bubbles collapse.

- *Improve the solvent penetration.* The microjets cause a localized increase of pressure which can enlarge the cell pores and effectively push the solvent into the cell.

- *Improve diffusion.* Ultrasound causes the constant renewal of the layer of solvent that is in contact with the solid. This is due to the turbulence and microcurrents occurring as a result of the bubbles' collapse, letting fresh (or less loaded) solvent surround the solid surface and therefore, improving the diffusion of the target compounds.

The intensification of the solid-liquid extraction process by ultrasound assistance might also allow what is known as green extraction, a current trend in natural products research proposed by many authors (Chemat et al., 2019; Civan and Kumcuoglu, 2019; Scopel et al., 2020), which involves reducing solvents, energy, wastes, and environmental pollution while obtaining higher extraction yields (Wen et al., 2018).

The level of success of high-power ultrasound application might also be affected by a variety of other operating parameters. For example, several authors have investigated the ultrasound-assisted extraction of bioactive compounds varying extraction conditions such as solvent (Ana et al., 2018; Đurović et al., 2018; Heleno et al., 2016a), temperature (González-Centeno et al., 2015; Pinela et al., 2019; Riciputi et al., 2018), time (Martínez-Patiño et al., 2019; Pan et al., 2012; Pinela et al., 2019), particle size (Aguiló-Aguayo et al., 2017; Wu et al., 2018), solid-liquid ratio (Huang et al., 2019; Kou et al., 2018) and ultrasound power and/or amplitude (Dabbour et al., 2018; Martínez-Patiño et al., 2019; Pinela et al., 2019). More specifically, Ana et al. (2018) who extracted phenolic compounds from sour orange by ultrasound assistance, observed significantly ($p < 0.05$) higher extraction yields when extracting in 50 % v/v of aqueous ethanol than when extracting in pure water or 96 % v/v aqueous ethanol. Increasing the temperature usually results in higher extraction yields in conventional extraction. However, in the case of ultrasound application, one of the main advantages is that mass transfer can be increased even at low temperatures. For instance, González-Centeno et al. (2015), observed similar extraction yields of phenolic compounds from grape pomace, when applying ultrasound at 35 °C, to those obtained with mechanical stirring at 50 °C. Ultrasound assistance has also been found to be able to reduce extraction times. For instance, Heleno et al., (2016a) extracted ergosterol from mushrooms (*A. bisporus*) and observed that ultrasound-assisted extraction at 375 W for 15 min was as efficient as the Soxhlet extraction for 12 h. Moreover, some authors have observed that extending the extraction time when applying high-power ultrasound might result in the degradation

of some bioactive compounds. For instance, Surin et al., (2020) extracted polysaccharides from purple glutinous rice bran by applying high-power ultrasound (150 W) and reported that the antioxidant activity of the extracts decreased when the extraction was longer than 30 min. They explained that the mechanisms activated by the ultrasound allowed the release of the polysaccharides from the cells, but thereafter, it could degrade them into free sugars making them lose their antioxidant properties. Particle size has also been found to affect the extraction yield. For example, Aguiló-Aguayo et al. (2017) investigated the ultrasound-assisted extraction of β-glucan from mushroom residues using different particle sizes (>450, 355-250, 150-120 and <90 μm) and observed significantly ($p < 0.05$) higher extraction yields when using particles in the range of 355-250 μm. The solid-liquid ratio is important due to the cost and technical problems deriving  from the use of solvents. Kou et al. (2018) who extracted hydrophobic gingerols and hydrophilic polysaccharides using liquid-based ultrasound-assisted extraction (100-400 W), observed higher extraction yields of gingerols at a solid-liquid ratio of 1:20 than those obtained at 1:10 and 1:15, but no significant differences when using 1:25 and 1:30. Finally, Hosseini et al. (2019) reported higher extraction yields of pectins obtained from sour orange peel, when ultrasound was applied at 150 W than when it was applied at 50 and 100 W.

## 1.3.2. Mathematical modelling of solid-liquid extraction

Mathematical modelling has become an irreplaceable tool in the intensification of processes in the food industry (Červeňanský et al., 2018), enabling the prediction of the behaviour of a system under different operating conditions. Further, through mathematical modelling, it is possible to analyze a process or even control it and optimize it with better use of resources like time, energy, and raw materials  (Simal et al., 2006). However, it is a challenge to model a transformation process of natural materials, since biological systems are heterogeneous, complex, and sensitive (Rodríguez Barragán et al., 2014).

There are two main steps in the modelling of a system. The first one consists of the formulation, resolution, and calibration of the model, and in the second, the model is simulated and validated.

In the first step, the different stages are as follows (Dalmau, 2019):

- *Observation.* The variables and factors that affect the systems must be identified.
- *Formulation of a hypothesis.* The mathematical relation between the factors and the variables should be hypothesized. These mathematical relations can be phenomenological or empiric.
- *Mathematical approach.* A series of equations are proposed from the hypothesis that best describes the system.
- *Resolution of the model.* A method to solve the equations is applied. The election of the method depends on the complexity of the model.

- *Parametrical identification.* The parameters of the model are estimated by using an appropriate method.

Once the values of the parameters have been calculated, the model is simulated and validated.

- *Simulation*. The model is applied by using the same conditions of/as in the calibration.
- *Validation*. There are different methods to validate a model. One of the most used consists of comparing the experimental and the calculated values mathematically or graphically.

As stated before, the mathematical approaches of a model might be phenomenological or empiric. Phenomenological models are based on physical laws whilst empirical models are based on a simple fitting of experimental observations (González-Centeno, 2013; Vallespir, 2019). For this reason, empirical models lack physical meaning since they only aim to determine the relevant variables of a system and describe the relationships among them. There are also semi-empirical models that use both physical and empirical estimations to provide some physical meaning to the system.

Empirical models are, in fact, useful for certain industrial applications and for simplifying complex systems difficult to formulate and solve through phenomenological models. This is the case of solid-liquid extraction processes as empirical models have been widely used in the extraction of bioactive compounds because of their simplicity and satisfactory fit to the experimental data (Cheung and Wu, 2013). There are several empirical and semi-empirical models previously applied for the extraction of bioactive compounds from fruit and vegetables or fungi materials. Among them are the second-order rate model (Patil and Akamanchi, 2017), the Weibull model (Rodríguez et al., 2014) the parabolic diffusion, power-law model (Cheung and Wu, 2013), and the Peleg model (Aguiló-Aguayo et al., 2017) .

Ho et al. (2005) observed that a model based on a second-order extraction process was the most suitable to describe the kinetics of water-soluble compounds extraction from *tilia* sapwood. Since then, this model has been successfully used on several occasions to evaluate solid-liquid extraction processes (Abugabr Elhag et al., 2020; Patil and Akamanchi, 2017; Rakotondramasy-Rabesiaka et al., 2007; Su et al., 2014). The model considers that there are two phenomena occurring during a solid-liquid extraction, an intense dissolution of the target compound and a much slower stage of external diffusion (Ho et al., 2005). The second-order rate equation can be described as follows:

$$\frac{dY}{dt} = k(Ymax - Y)^2 \qquad\qquad \text{Eq. 1}$$

where Y is the extraction yield at a specific time t, Ymax is the maximum extraction yield, t the extraction time, and k the extraction rate constant. The initial extraction rate defined as h when t approaches 0, can be expressed as,

$$h = k\,Ymax^2$$

Eq. 2

The Weibull model has also been applied to solid-liquid extraction processes (González-Centeno et al., 2015; Rodríguez Barragán et al., 2014). It is a probability distribution function used to describe the behaviour of systems or events that have some degree of variability (Cunha et al., 1998). This model has great versatility and has been used in different areas of food engineering. For instance, besides solid-liquid extraction processes, it has been applied to describe microorganism inactivation (Chen et al., 2013), drying, rehydration kinetics (Zura-Bravo et al., 2013), and the release of bioactive compounds during digestion (Dalmau, 2019) among other applications. The Weibull model equation can be described as follows:

$$\frac{Y - Y_{eq}}{Y_0 - Y_{eq}} = e^{[-\left(\frac{t}{\alpha}\right)^{\beta}]}$$

Eq. 3

The term $Y_{eq}$ is the yield at equilibrium (or maximum yield) and Y is the extraction yield at time t; α is related to the reciprocal of the extraction rate constant, thus, a lower α indicates a faster extraction rate; and β is the shape parameter of the model, which represents a behaviour index of the material during the process. When β is equal to 1, the model corresponds to a first-order kinetic with a constant rate. Meanwhile, when β has a value above or below 1, this parameter denotes the concavity (increasing rate over time) or convexity (decreasing rate over time) of the curve (González-Centeno et al., 2015).

## 1.4. Microencapsulation by spray-drying

Microencapsulation is a process by which an active agent or ingredient can be stored within a shell, being coated with a continuous film of polymeric material. Thus, a microcapsule consists of an external wall shell composed of what is commonly known as wall material, and an internal core cavity for active agents or ingredients. The wall material can act as a physical barrier for active ingredients against hazardous environmental conditions like oxygen, moisture, acids, alkalinity, heat, and even interactions with other compounds. It can also provide an active agent with some desirable physical properties such as solubility and flowability. The particles produced in this process are in the range of micrometres to millimetres (Alihosseini, 2016; Salaün, 2016).

According to the forming mechanism of the shell, microencapsulation methods can be divided into three categories: physical, chemical, and physicochemical. In the physical methods, the formation of the shell occurs because of the evaporation of the solvent in which the wall material was dissolved along with the active agent to be encapsulated.

In chemical methods, monomers with small molecules polymerize to form the polymer shell around the active agent; and in physicochemical microencapsulation, the precipitation of the pre-dissolved shell-forming materials is provoked by varying the temperature, pH value, or electrolyte concentration (Fu and Hu, 2017).

### 1.4.1.Spray drying

Spray drying is the most commonly used technology in the food industry for microencapsulation due to its low cost and the available equipment. It is a physical method by which a liquid is atomized in a hot gas current to instantaneously obtain a powder (Gharsallaoui et al., 2007).

The unit operation of spray drying consists of three steps: the atomization of the feed sample into the drying chamber, the drying of liquid droplets, and the powder recovery (Shishir and Chen, 2017). A diagram of the general process of spray drying is shown in Figure 12.

- *Atomization*. The atomization step involves the distribution of the liquid into tiny droplets. This maximizes the surface volume area of liquid for efficient drying. The main types of commercial atomizers are rotary atomizers, single-fluid nozzle, and twin-fluid nozzles. In rotary atomization, the liquid feed is centrifugally accelerated to high velocities. The atomization energy is provided by the rotational speed, thus, the size of the droplets decreases with higher rotational speeds (Walters et al., 2014). Single-fluid nozzles are operated by pumping the fluid through a die or orifice under pressure. Finally, twin-fluid nozzles apply a high-speed gas stream, usually air, to blast the liquid into droplets. Single-fluid and twin-fluid nozzles produce smaller droplets with a larger distribution than rotary atomizers, thus are preferable for applications that require smaller particle sizes (Walters et al., 2014; Ziaee et al., 2019).
- *Drying*. The drying occurs when the atomized droplets and hot air interact, resulting in the evaporation of water or other solvents. A dry layer of the wall material develops, leaving a powder with low moisture content and water activity. This method can dry a product very quickly compared to other methods (Phisut, 2012; Shishir and Chen, 2017). Spray dryers can be classified according to the flow pattern within the drying chamber. There are three main types of air flow patterns, namely co-current, counter-current, and mixed flow.
  In the co-current types, the drying air and droplets move in the same direction from the top of the drying chamber to the bottom. In the counter-current setup, the feed and drying gas flow in opposite directions. The droplets' outlet temperature is higher in this arrangement than in the co-current, so it is not recommended for thermally sensitive compounds. The mixed flow is a combination of co-current and counter-current. The atomizer is placed in the middle of the drying chamber where the airflow is applied downward, and then

the solution is sprayed upward or downward depending on its thermal stability (Ziaee et al., 2019).

- *Powder recovery.* Dense particles are recovered at the base of the chamber and the finest ones pass through a cyclone to separate them from the humid air.
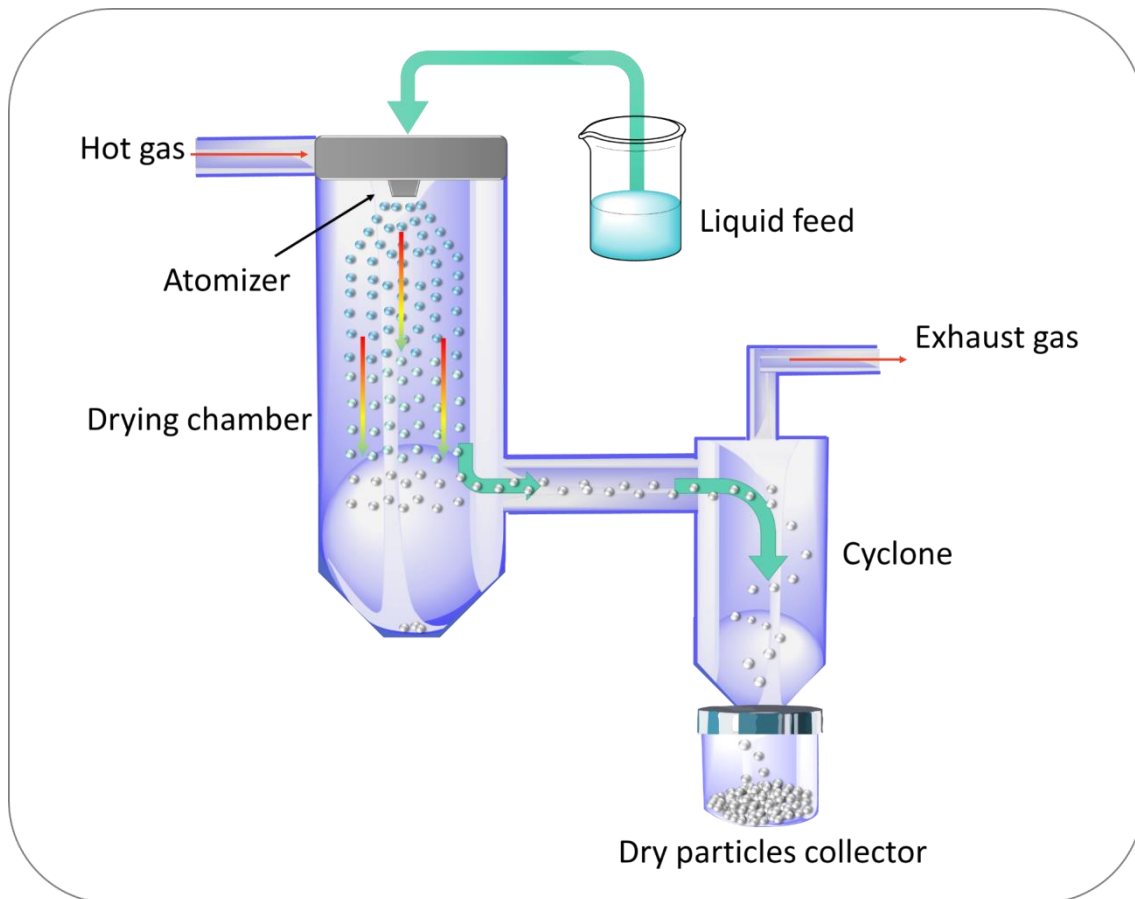


**Figure 12.** Schematic representation of a spray drying process

The obtained powder is made up of microparticles that originated from spherical droplets after shrinking. The characteristics of these particles such as their morphology, particle size distributions, moisture content, water activity, porosity, density, flow properties, and others, depend on the conditions of the spray drying process. Moreover, the parameters related to the efficiency of the process such as yield or encapsulation efficiency are also related to the spray drying conditions (Piñón-Balderrama et al., 2020).

The main parameters that affect the microencapsulation process by spray drying are (Piñón-Balderrama et al., 2020; Zbicinski et al., 2002):

- *The inlet temperature.* The temperature of the drying medium.
- *The feed rates.* The amount of liquid fed into the drying chamber per time (i.e. mL/min).

- *The outlet temperature.* The temperature of the sample when it leaves the drying chamber, which cannot be set up since it is a consequence of other factors such as the inlet temperature.
- *The aspirator rates.* The amount of compressed gas supplied to the drying chamber.

## 1.4.2. Microencapsulation of lipid compounds

In the food industry, many of the bioactive compounds or ingredients to be encapsulated consist of lipophilic compounds. These compounds are important because of their organoleptic and technological functions (aroma, flavours) and/or their healthy properties (vitamins, polyunsaturated fatty acids). However, lipids rich in unsaturated fatty acids are susceptible to oxidation, which is one of the most undesirable reactions in foods since it affects both the nutritional composition and the sensorial properties of the product.

Lipids oxidation involves three stages: initiation, propagation, and termination (Domínguez et al., 2019; Roman et al., 2013). Initiation occurs when reactive oxygen species abstract hydrogen from an unsaturated fatty acid. This results in an alkyl radical that tends to be stabilized by rearranging the double-bonds to form a conjugated diene. The alkyl radical reacts with $O_2$ to form peroxyl radicals and hydroperoxides. The hydroperoxide decomposition gives rise to hydroxy, peroxy and alkoxy radicals. During the propagation stage, the oxidation occurs by lipid-lipid interactions, since the peroxy radicals are highly reactive and abstract hydrogen from other lipids producing more alky radicals. Finally, the process that ensures termination efficiently is the decomposition of peroxy and alkoxy radicals to generate secondary products such as alkanes, alcohols and carbonyl compounds, which are responsible for the sensory deterioration of lipids (Domínguez et al., 2019).

Hence, the oxidation of lipids could be prevented by protecting lipophilic compounds from contact with oxygen in the first place. This is why microencapsulation is one of the main strategies applied by the food industry to minimize the degradation of lipophilic compounds, along with the addition of natural or synthetic antioxidants.

## 1.4.3. Emulsions for spray drying

The encapsulation of lipophilic compounds by spray drying involves the dispersion of these compounds in an emulsion which will be the feeding liquid of the spray drying process (Hernández Sánchez et al., 2016). Besides the spray drying conditions, the composition and characteristics of the feeding liquid have an important effect on the quality of the microcapsules.

Emulsions are unstable thermodynamic systems that consist of two immiscible liquids, one of which is dispersed in the other in the form of small droplets (Matos et al., 2018; McClements, 2005). Food emulsions can be conveniently classified in oil-in-water (O/W)

emulsions, which consist of oil droplets dispersed in an aqueous phase, for example, milk, dressings, and sauces. And water-in-oil (W/O) emulsions, which are systems of water droplets dispersed in an oil phase, for example, margarine and butter. The substance that makes up the droplets in an emulsion is called the dispersed or discontinuous phase, whilst the substance that makes up the surrounding liquid is called the continuous or external phase (McClements, 2005). To encapsulate lipid compounds by spray drying, the initial emulsion is usually an oil-in-water emulsion and should have compatible characteristics with this process (Hernández Sanchez et al., 2015; Lewandowski et al., 2016). These characteristics are described below.

### 1.4.3.1. Emulsion stability

The emulsion must be physically stable until and during spray drying (Janiszewska et al., 2015). Several physicochemical mechanisms may cause alterations in the emulsion properties. Some of the most common instability mechanisms are presented in Figure 13 (McClements, 2007, 2005; Pons, 2000):

- *Creaming and sedimentation*. Both phenomena have a common origin: the difference in density of the emulsion phases. Creaming describes the upward movement of droplets since they have a lower density than the continuous phase. If the dispersed phase has a higher density, droplets move downward, in the process known as sedimentation.

  The rate at which an isolated rigid spherical particle migrates in an ideal (Newtonian) liquid is given by Stokes' Law (Eq. 4) (McClements, 2007):

$$v_{stokes=} - \frac{2\,g\,r^2(\rho_2 - \rho_1)}{9\eta_1} \qquad \text{Eq. 4}$$

  where $v_{Stokes}$ is the immigration velocity, r is the radius of the droplet, g is the gravity acceleration, $\rho$ is the density, $\eta$ is the viscosity, and subscripts 1 and 2 refer to the continuous and dispersed phases, respectively. The $v_{Stokes}$ sign determines if the droplet is cream (+) or sediment (-). From this equation, it can be deduced that gravitational separation can be delayed in an emulsion by decreasing the density contrast between the phases, by reducing the size of the droplets, or by increasing the viscosity of the continuous phase.

- *Flocculation*. Flocculation is the process by which two or more droplets aggregate without losing their individuality. Two droplets flocculate because the repulsive forces required to oppose the attraction between droplets are not high enough. The main attractive forces in food emulsions are van der Waals, depletion, and hydrophobic interactions, whilst the main repulsive forces are electrostatic and steric.

- *Coalescence*. Coalescence is the process by which two or more droplets merge to form a bigger droplet. Coalescence may occur as a result of droplet collisions because repulsive interactions are not strong enough to stop them from

approaching each other. It can also occur because of two or more droplets remaining in contact for extended periods, i.e., in floccules.

- *Ostwald ripening*. This is the process whereby bigger droplets grow at the expense of smaller droplets which, eventually, disappear. This occurs through the mass transport of dispersed phase material through the continuous phase.
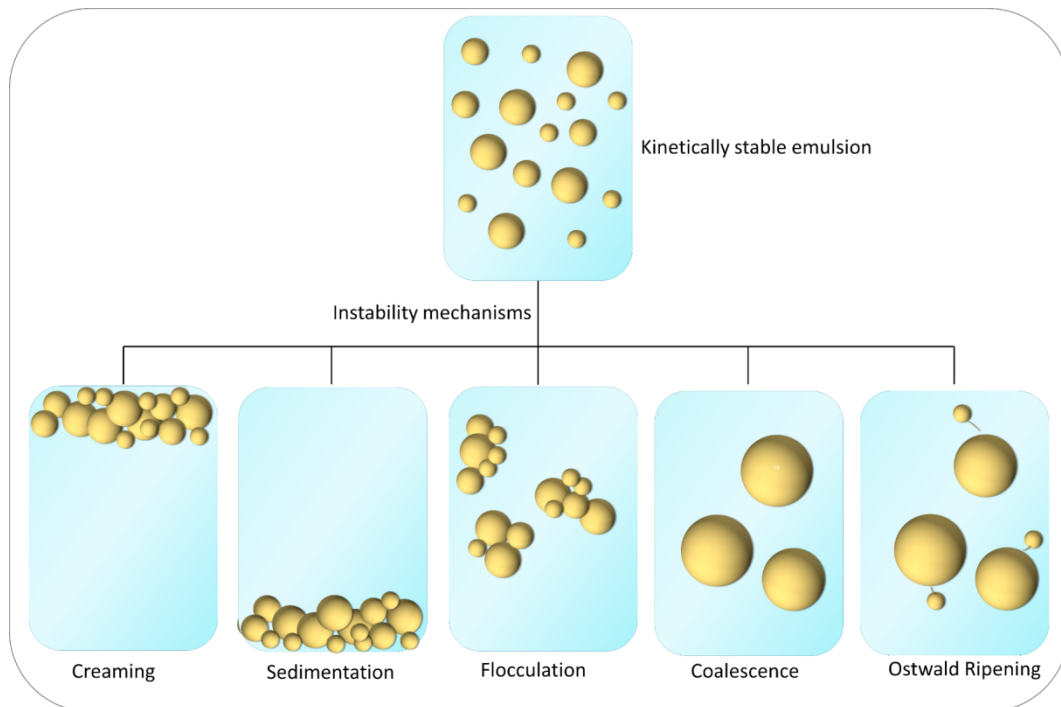


**Figure 13.** Schematic representation of the most common instability mechanisms that occur in emulsions

### 1.4.3.2. The droplet size distribution of the emulsion

The droplets' size in the initial emulsion is an important parameter that can affect encapsulation efficiency. For instance, it has been observed that the presence of oil droplets with diameters of 10 µm or larger results in low efficiency when using pilot-scale spray drying (Turchiuli et al., 2014). Droplets that are too large increase the non-encapsulated oil after spray drying, which results in lower oxidative stability (Linke et al., 2020). Thus, the droplet size should be in concordance with the size of the intended microcapsules and with the spray drying equipment. Generally, for spray drying, the initial emulsion droplets' distribution should be in the order of 1-100 µm diameter (Gharsallaoui et al., 2007).

A single value cannot accurately describe the size of the droplets in an emulsion. Thus, it is usually presented as a droplet size distribution. This consists of dividing the full droplet size range into a determined number of classes. The fraction of particles in a class can be defined as the number of droplets or as the volume of the droplets in each size class. Volume frequency is more sensitive to larger droplets and is normally preferred for emulsions. The droplet size distribution can be represented as a smooth

curve (McClements, 2005). The particle size at which half the droplets are smaller, and the other half are larger is named the *median* particle ($d_{50}$). Other percentiles as $d_{10}$ and $d_{90}$ are also used to describe a droplet size distribution. The spread of the distribution can be represented as the span of the emulsion and can be calculated as follows (Serfert et al., 2013):

$$Span = \frac{(d_{90} - d_{10})}{d_{50}}$$
Eq. 5

Emulsions prepared for spray drying should ideally present a monomodal droplet size distribution with a median diameter of about 2 μm and a span no larger than 2 to obtain microcapsules with a diameter of about 20 μm (Gallotti et al., 2020; Hernández Sanchez et al., 2015).

There are several methods for the determination of the oil droplet size distribution in an emulsion (e.g. laser diffraction and dynamic light scattering) (Schuster et al., 2012). Unfortunately, these methods usually require expensive equipment. However, microscopy is a simple and direct method that provides information about the microstructure of the emulsion. Among the various types of microscopes including optical, electron, or atomic force, optical microscopy is the most commonly used for obtaining information about the characteristics of the emulsion such as the presence of floccules (McClements, 2007). Optical microscopes are relatively inexpensive and are already available in many food technology laboratories.

Techniques such as laser diffraction can provide rapid information about the droplet size distribution but cannot directly distinguish between different types of destabilization phenomena, e.g., coalescence versus association of several droplets (flocculation). Microscopy, on the other hand, is an efficient technique for distinguishing different destabilization mechanisms. Another advantage is that microscopy requires only a small quantity of the sample (< 1 mL). An aliquot of the emulsion is placed on a microscope slide and subsequently observed. The microscope can be connected to a digital camera and computer to obtain images, which can be manipulated using imaging processing software to obtain valuable qualitative and quantitative information.

An appropriate image analysis must be applied to obtain reliable and systematic results. Image analysis allows the study of the shape and size of individual particles obtaining dimensions such as area and perimeter. However, a huge number of particles must be measured to obtain representative results (Dalmau, 2019). According to Schuster et al. (2012), at least 9000 droplets need to be measured to obtain a droplet size distribution of an emulsion which is expected to present a span of 2. The image analysis applied to emulsions, generally, consists of four steps: a) converting image to binary, b) creation of a border around each droplet, c) droplet size determination and c) statistical treatment (Freire et al., 2005; Hosseini et al., 2015; Schuster et al., 2012). The same procedure can

be applied to determine the particle size distribution of microcapsules obtained after the spray drying.

### 1.4.3.3. The viscosity of the emulsion

According to the Stokes equation (Eq. 4), creaming could be prevented by increasing the continuous phase viscosity. Moreover, a higher viscosity hinders the droplets' mobility, thus preventing phenomena such as flocculation and coalescence. However, this parameter can only be increased up to a certain limit, since it has been observed that the higher the emulsion viscosity, the larger the droplets obtained during atomization (Tonon et al., 2011) which is not desirable for some food applications. Further, some spray drying types of equipment are not able to pump liquids that are too viscous. For instance, lab-scale spray dryers are usually limited to a viscosity of 300 mPa·s (BUCHI, 2010).

The rheological behaviour of emulsions is also important for spray drying, especially when it comes to the viscosity variation with shear rates. Depending on their composition, initial emulsions can present Newtonian or non-Newtonian behaviour. Newtonian behaviour means that the emulsion viscosity does not change when the shear rate increases. Among non-Newtonian behaviours, shear-thinning is the most common in oil-in-water emulsions containing polysaccharides. It indicates that the viscosity decrease with shear rates increases (McClements, 2015). The values of viscosity at high shear rates allows the prediction of the viscosity during certain processing operations, such as pumping and spray drying (Koocheki et al., 2009). Thus, in shear-thinning emulsions, pumping efficiency is expected to be high since their viscosity decreases with the shear rate.

### 1.4.3.4. Composition of the emulsion

Finally, the initial emulsion should have a formulation that allows the microencapsulation by spray drying. This means that the emulsion must contain a wall material in an appropriate concentration (e.g. up to 35–60 g/100 g liquid feed) (Hernández Sanchez et al., 2015). The type of wall material in microencapsulation by spray drying is very important for encapsulation efficiency and core protection (Gharsallaoui et al., 2007). A wall material should have low hygroscopicity in order to protect the encapsulated agent from moisture (Delshadi et al., 2020). A high glass transition temperature (Tg) is desirable since it prevents the final powders from caking and facilitates storage (Ramakrishnan et al., 2018). Caking is a phenomenon whereby free-flowing particles aggregate to form larger lumps of particles; this usually reduces the powders' quality and core stability (Descamps et al., 2013). Wall materials should not react with the encapsulated component during processing and storage and should be low cost and food-grade (Hernández Sanchez et al., 2015). Besides all these characteristics, it is desirable that wall materials produce stable emulsions (Hernández

Sanchez et al., 2015). The most commonly used types of wall materials are described below (Can Karaca et al., 2015; Gharsallaoui et al., 2007).

- *Carbohydrates.* Carbohydrates such as starches and maltodextrins are widely used as encapsulating agents. They exhibit low viscosities at high solids content and good solubility in water. However, most of these types of materials lack the emulsifying capacity required for high microencapsulation efficiency.
- *Gums.* Gums are used in microencapsulation because of their film-forming capacity and emulsion stabilization properties. Among all gums, Arabic gum stands out due to its excellent emulsification properties, which have been attributed to the presence of a small protein fraction. Nevertheless, this material is expensive and supplies are limited.
- *Proteins*. The functional properties of proteins make them a good wall material for microencapsulation by spray-drying. Proteins adsorb at the oil-water interface and form stable emulsions. The most used proteins for encapsulating food ingredients are milk (or whey) proteins and gelatin. However, the consumer demand for alternatives to proteins from animals has led researchers to evaluate proteins coming from plants. Thus, proteins obtained from soy, pulses (e.g. pean) (Pierucci et al., 2007), and cereals (e.g. wheat) (Liao et al., 2012) have been found to have an amphiphilic nature and good film-forming capacity.

Since it is unlikely that a single wall material meets all the required criteria, they are used in combination (Carneiro et al., 2013; Premi and Sharma, 2017). Moreover, when the single or combined wall materials used for microencapsulation of lipid compounds do not present emulsifying properties, it is necessary to add an emulsifier. These molecules contain a lipophilic tail that interacts with the oil and a hydrophilic head that interacts with the water at the interface of the dispersed droplet (Miller, 2015). Emulsifiers maintain the stability of the emulsions by generating strong electrostatic and/or steric repulsive interactions (McClements and Gumus, 2016). The electrostatic repulsive interactions consist of electrically charged droplets that have the same sign and repulse each other. The attraction between lipid droplets suspended in water depends on the surface charge density, as well as the solution conditions, such as ionic strength and solvent type. Steric repulsive interactions occur when the emulsifier molecules have flexible molecular chains ("hairs") that remain in the continuous phase. When the surface of another particle comes close, there is an increase of free energy, and repulsion occurs. This volume-restriction effect can be large, but it is significant only if the surfaces have an exceptionally low hair density. If this is not the case, another mechanism can occur; the overlap of two droplets provokes an increase of hairs concentrations and then, an increase of osmotic pressure; therefore, water moves to the overlap region, which results in repulsion between the droplets (Fennema, 1996; McClements and Gumus, 2016). A schematic representation of electrostatic and steric repulsions is shown in Figure 14.
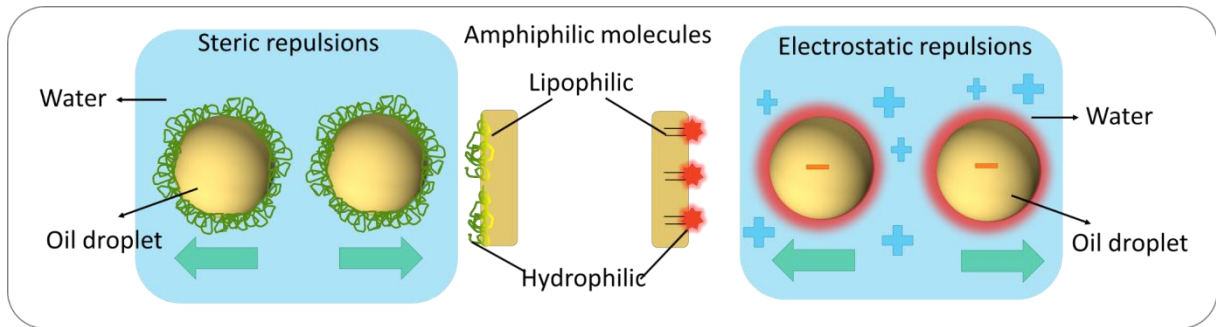
**Figure 14**. Schematic representation of steric and electrostatic repulsive interactions promoted by emulsifier to stabilize oil-in-water emulsions

Conventional emulsifiers include surface-active agents with low molecular weight, and polymers (Berton-Carabin and Schroën, 2015).

- *Low-molecular-weight emulsifiers.* These emulsifiers have molecular weights from about 250 g/mol, e.g. monoacylglycerols, to about 1,200 g/mol, e.g. polysorbates (Berton-Carabin and Schroën, 2015). They are classified as nonionic, anionic, and cationic, according to the nature of the hydrophilic part, and can be of either natural or synthetic origin (Fennema, 1996). The application of these types of surfactants depends on their hydrophilic-lipophilic balance (HLB). HLB represents the oil and water solubility of an emulsifier within a scale of 0–20 for non-ionic surfactant and up to 60 for ionic surfactant. Lower HLB values are an indication of high solubility in oil, and high HLB values indicate high water-solubility. Thus, emulsifiers with high HLB values are suitable for stabilizing oil-in-water emulsions (Premlal Ranjith and Wijewardene, 2006). For instance, the emulsifier commercially known as Tween®20 (polyoxyethylene sorbitan monolaurate with 20 ethylene oxide units) which is a nonionic type emulsifier, has an HLB of 16.7 and is considered a hydrophilic emulsifier being widely applied for oil-in-water emulsions (Dinarvand et al., 2005).

- *Polymers with amphiphilic nature*. In the case of biopolymers, proteins stand out and have been widely investigated for their emulsifying properties. Polysaccharide hydrocolloids, on the other hand, are not considered to be strong surface-active agents (Santipanichwong and Suphantharika, 2009). Some studies have successfully produced stable oil-in-water emulsions using mixtures of proteins and polysaccharides (Nicoletti Telis, 2018; Ray and Rousseau, 2013). The combination is interesting since proteins are known for their emulsification properties, and polysaccharides for their water-holding and thickening capacity (Dickinson, 2003). Moreover, protein-polysaccharides conjugates can be obtained through Maillard reactions. Some studies have demonstrated that these conjugates are able to stabilize emulsions because hydrophobic groups of the protein are attached to the oil droplets, and polysaccharides hydrophilic groups orient themselves in the aqueous phase stabilizing the emulsion through

a steric hindrance effect which inhibits coalescence (Setiowati et al., 2020). Nevertheless, it should be considered that in emulsions stabilized with polymers, two phenomena, bridging and depletion flocculation, can take place. At low polymer concentrations, a single polymer chain might adsorb onto two or more droplets, causing what is known as bridging flocculation. On the other hand, where there are no adsorbed polymers an osmotic imbalance occurs between the continuous phase and the spaces surrounding the oil droplets. This causes the solvent to flow out from the space between the oil droplets, resulting in a force of attraction between the oil droplets. This is known as depletion flocculation.

Besides low-molecular-weight surfactants and polymers, emulsions can also be stabilized by solid colloidal particles. This type of emulsions is known as Pickering emulsions. The solid particles are partially moistened by oil and water. It has been observed that Pickering emulsions show great stability against coalescence and Ostwald ripening compared to emulsions stabilized by low-molecular-weight surfactants and biopolymers (Berton-Carabin and Schroën, 2015; Mwangi et al., 2020). Different types of colloidal particles have been used in Pickering emulsions including inorganic particles such as silica (Sagar et al., 2018) and biological particles such as aggregates of soy proteins (Liu and Tang, 2013), starch (Yang et al., 2018), gelatin (Feng et al., 2020), cellulose (Dai et al., 2020) and others. They have also been produced using materials with a heterogeneous composition such as bamboo shoot dietary fibre (He et al., 2020), citrus fibre (Qi et al., 2020) and apple, oat, and citrus by-products (Huc-Mathis et al., 2021).

### 1.4.4. Natural compounds in lipid microencapsulation

Nowadays, consumers place a high value on the naturalness of their food. They consider its origin, the ingredients and technology used to produce it, and the properties of the final product, before buying and consuming (Román et al., 2017). Thus, there is an increasing demand for "clean label products". There is no official definition for such a concept, but recent investigations suggest that these consumers are looking for familiar ingredients, shorter ingredient lists, and ingredients that are minimally processed (Maruyama et al., 2021).

This situation has led the food industry to search for promising new ingredients coming from natural sources, materials that should improve the nutritional quality of the final products. Furthermore, there is a consumer demand for alternatives to animal-derived products because of great interest in vegan and vegetarianism as well as animal welfare concerns (Can Karaca et al., 2015; García-Gudiño et al., 2021).

Specifically, in lipid microencapsulation by spray drying, researchers have directed their attention to plant-derived compounds such as plant proteins and polysaccharides that can act as wall material and/or as emulsifiers to totally or partially replace commonly

used compounds (Berton-Carabin and Schroën, 2019; Can Karaca et al., 2015). Table 1 shows several studies that have evaluated the use of different materials coming from plants and mushrooms, in the microencapsulation of lipid compounds by spray drying. As can be seen, proteins coming from legumes such as soy or peas, or cereals such as barley, have been applied on several occasions to microencapsulation processes (Nesterenko et al., 2012; Pierucci et al., 2007; Rascón et al., 2011; Wang et al., 2011). However, in the last decade, the materials evaluated for lipid microencapsulation have been diversified. Thus, more recently, proteins used for lipid microencapsulation have also been obtained from other raw matters such as sunflowers, flaxseed, or rice (Gomes and Kurozawa, 2020; Le Priol et al., 2019; Pham et al., 2020). Moreover, other compounds besides proteins, such as polysaccharides, have been applied to these processes. This is the case of β-glucans which have been obtained from both cereals and mushrooms for their use as wall material (Gallotti et al., 2020; Salgado et al., 2015). The lack of solubility of these materials, especially proteins, has led researchers to combine them with commonly used wall materials such as maltodextrin (Gomes and Kurozawa, 2020; Karaca et al., 2013).

Generally, promising results have been obtained from these studies. For instance, Rusli et al. (2006) obtained encapsulation efficiencies of 80-94 % in tuna oil when using soy protein isolate (14-27 % dm (dry matter)) combined with glucose syrup. Similarly, Gomes and Kurozawa (2020) observed high encapsulation efficiencies (80-89 %) when encapsulating linseed oil using hydrolyzed rice protein (4.4 % dm) combined with maltodextrin. Besides the high encapsulation efficiencies, some studies have also reported that the addition of natural materials improves the oxidative stability of the encapsulated oil. For example, Gallotti et al. (2020) observed a lower content of primary oxidation indicators (conjugated dienes) in sunflower oil encapsulated with β-glucans obtained from *Pleurotus Ostreatus,* than that in oil encapsulated only with maltodextrin and Arabic gum. Shi et al. (2020) observed higher oxidative stability in tuna oil encapsulated with a green tea leaves powder, than in that encapsulated with maltodextrin. This powder (matcha) used by Shi et al. (2020), was a heterogeneous material mainly composed of proteins (about 30 % dm) and dietary fibre (about 40 % dm) and with a considerable presence of antioxidant compounds such as catechins (about 13 % dm). The authors explained that the higher oxidative stability of the oil encapsulated with matcha powder could be due to its high content of antioxidant compounds. This research demonstrated that naturally occurring heterogeneous compounds have great potential as wall material with inherent antioxidant activity.

The use of innovative natural material in oil microencapsulation also affects the characteristics of the initial oil-in-water emulsions. Francisco et al. (2020) produced orange essential oil microcapsules using soy protein isolate and pea protein isolate as emulsifiers. They observed that the emulsions were physically stable for about 2 h when using 2.4 % of any soy or pea protein isolate. Gomes and Kurozawa (2020) observed that

hydrolyzed rice protein favourably affected the surface charge of linseed oil droplets in oil-in-water emulsions, obtaining zeta potentials in a range of -44 to -55 mV. These values indicate that the proteins were able to be adsorbed onto the oil droplets thus improving the emulsion stability. In fact, several studies have used naturally occurring proteins and polysaccharides for the stabilization of oil-in-water emulsions prepared for different purposes, not only for spray drying. For instance, Maravić et al. (2019) produced oil-in-water emulsions stabilized with sugar beet fibre and modified maltodextrin. An increase in the stability against creaming was observed after 24 h when using sugar beet fibre (70-74 % creaming) rather than maltodextrin alone (90 % creaming). The capacity of sugar beet fibre to reduce emulsion creaming was mainly attributed to the increase of continuous phase viscosity. Zhu et al. (2020) evaluated the emulsifying capacity of eggplant pulp (containing 34 % fibre and 12 % protein) and reported that stable oil-in-water emulsions were obtained with a concentration of 1.50 % of the pulp. After 7 days, emulsions containing this material (1.50 %) presented no variation in the droplet size distribution and no creaming. Santipanichwong and Suphantharika (2009) studied the effect of β-glucans obtained from different sources (curdlan, barley, oat, and yeast) on the physical and rheological properties of egg yolk stabilized oil-in-water emulsion. They observed that the creaming stability during storage of these emulsions containing β-glucans was improved, possibly because of the increase in the viscosity of the continuous phase and/or a formation of a three-dimensional droplet network. Vasile et al. (2016) evaluated the use of a non-conventional exudate gum (*Prosopis alba*) comparing it with the better known Arabic gum. They concluded that this novel material was able to produce emulsions with smaller droplet diameters, higher zeta potentials, and lower values of polydispersity and creaming indexes during storage than Arabic gum; these results being explained by the higher content of proteins in *Prosopis alba* exudate. Huc-Mathis et al. (2021) stabilized oil-in-water emulsions using by-products of orange, oats, and apples and observed that the insoluble particles of these materials prevented the oil droplets' coalescence creating a Pickering effect, while the soluble compounds were adsorbed at the interface, reducing the droplet size, and acting as thickening agents from the continuous phase.

As can be seen, several investigations have been carried out using compounds with a natural origin as emulsifiers and/or as wall material. However, in most of the cases, these compounds were purchased as commercial concentrates which had probably been submitted to aggressive purification processes (Charve and Reineccius, 2009; Francisco et al., 2020; Le Priol et al., 2019; Locali et al., 2019; Maravić et al., 2019; Nesterenko et al., 2012; Pierucci et al., 2007; Rascón et al., 2011; Rusli et al., 2006; Salgado et al., 2015). The possibility of obtaining these materials from fruit, vegetable, or fungi residues is an interesting alternative for their reintroduction into a sustainable circular economy as renewable raw matter. Nonetheless, the application of any innovative material in the microencapsulation process needs to be evaluated. For

example, it is necessary to determine the effect of these novel materials on the characteristics of the oil-in-water emulsions such as viscosity, stability, and oil droplet size, and on the properties of the final microcapsules, especially encapsulation efficiency and the oxidative stability of the encapsulated oil.

**Table 1.** Literature review on the evaluation of the usefulness of fruits, vegetables, and fungi materials in the microencapsulation of lipids by spray drying.

| Natural material | Function | Lipid compound encapsulated | Reference |
|---|---|---|---|
| Soy protein isolate | Wall material combined with glucose syrup | Tuna oil with or without palm stearin | (Rusli et al., 2006) |
| Pea protein | Wall material combined or not with maltodextrin and sodium-carboxymethylcellulose | α-tocopherol | (Pierucci et al., 2007) |
| Soy protein isolates | Wall material | Limonene and 3 unsaturated aldehydes | (Charve and Reineccius, 2009) |
| Barley protein | Wall material | Fish oil | (Wang et al., 2011) |
| Soy protein isolate | Wall material | Paprika oleoresin | (Rascón et al., 2011) |
| Modified soy protein isolate | Wall material | α-tocopherol | (Nesterenko et al., 2012) |
| Pea protein isolate and pea protein | Wall material combined or not with maltodextrin and carboxymethylcellulose | Conjugated linoleic acid | (Costa et al., 2015) |
| Barley β-glucans and soy lecithin | Wall material | Resveratrol | (Salgado et al., 2015) |
| Barley β-glucan | Wall material combined or not with modified starch | Fish oil | (Kurek et al., 2018) |
| Barley β-glucans | Wall material combined with Arabic gum and maltodextrin | Sea-buckthorn oil | (Drozińska et al., 2019) |
| Protein isolate from pea, soybean, and brown rice and protein from hemp and sunflower | Wall material | Sunflower oil | (Le Priol et al., 2019) |

**Table 1** (continued)

| Natural material | Function | Lipid compound encapsulated | Reference |
|---|---|---|---|
| Soy protein isolate and pectin | Wall material combined with maltodextrin | Pink pepper oil | (Locali et al., 2019) |
| Pea and soy proteins | Emulsifiers | Orange essential oil | (Francisco et al., 2020) |
| β-glucans and proteins extracts obtained from *Pleurotus Ostreatus* | Emulsifier and wall material combined with maltodextrin | Sunflower oil and α-tocopherol | (Gallotti et al., 2020) |
| Rice protein submitted to enzymatic hydrolysis | Emulsifier and wall material combined with maltodextrin | Linseed oil | (Gomes and Kurozawa, 2020) |
| Pea protein isolate and sugar beet pectin | Wall material | Hemp seed oil | (Lan et al., 2020) |
| Polyphenol-adducted flaxseed protein isolate and flaxseed gum | Wall material | Flaxseed oil | (Pham et al., 2020) |
| Green tea (matcha) powder | Wall material combined or not with maltodextrin | Tuna oil | (Shi et al., 2020) |

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## 1.5.Research hypotheses

After considering the literature, the following research hypotheses were stated as the initial point of the experimental work:

- Agri-food by-products from plants and fungi are a potential source of bioactive compounds such as dietary fibre (polysaccharides), compounds with antioxidant activity, and provitamins.
- The extraction of bioactive compounds from plants and fungi by-products could be intensified by applying high-power ultrasound, even when working under mild conditions such as low temperature and without aggressive or toxic solvents.
- The solid-liquid extraction process assisted by high-power ultrasound could be evaluated by mathematical modelling.
- The solid residues obtained after the extraction of antioxidant compounds and provitamins from plants and fungi might be still rich in other valuable compounds such as polysaccharides and proteins.
- Agri-food by-products rich in polysaccharides and proteins could be useful for the stabilization of oil-in-water emulsions suitable for spray drying.
- Agri-food by-products with inherent antioxidant activity might be able to protect lipids from oxidation during spray drying and storage.

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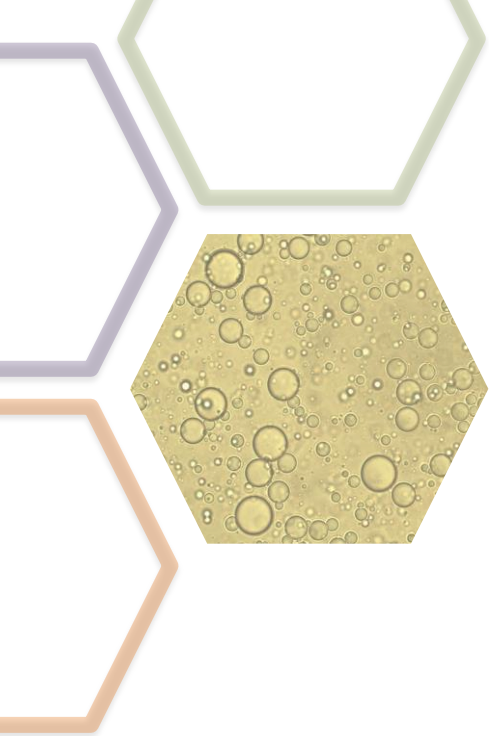
OBJETIVES

## 2. OBJECTIVES

The work presented in this thesis was carried out within the framework of two research projects developed by the Agri-Food Engineering Group at the University of the Balearic Islands. These projects, financially supported by the National Institute of Research and Agri-food Technology (INIA), ERDF, the Spanish research agency (AEI) and the Ministry of Science and Innovation (MCI) were the following:

- "Revalorización integral de subproductos en función de sus usos potenciales: Extracción de compuestos de interés mediante aplicación de US de potencia y estudios de bioaccesibilidad in vitro (RTA 2015-00060-C04-03)" within the coordinated project: "Gestión sostenible y revalorización de subproductos agroalimentarios para alimentación, energía y uso agronómico."
- "Integration of revalorised agro-food by-products into an enhanced circular economy model: Value-added vegan foods for the agro-industrial and HORECA sectors (PID2019-106148RR-C43)" within the coordinated project: "Integration of revalorised agro-food by-products into an enhanced circular Economy model (REVAL 2.0)."

These projects focused on the intensification of solid-liquid extraction of bioactive compounds from agri-food by-products through the application of power ultrasound; and on the transition of agri-food by-products, from a linear to a circular economy, by using them as renewable raw matters in the food industry.

Within this context, the main aim of the work was to propose alternatives for the exploitation of agri-food by-products through the extraction of their bioactive compounds using high-power ultrasound; and to explore the application of these by-products and the residues of the extraction processes in lipid microencapsulation, taking advantage of their emulsifying and inherent antioxidant capacity, and therefore, proposing an integral valorization.

Thus, in order to accomplish both general objectives, the following specific objectives were proposed:

- Evaluate the application of high-power ultrasound in the extraction of pectins from orange by-products (pulp and peel) using mild extraction conditions such as weak acid (citric acid) and low temperature (25 °C).
- Evaluate the application of power ultrasound in the extraction of a provitamin of vitamin D (ergosterol) and other antioxidant compounds from mushroom by-products using different concentrations of an organic solvent (ethanol) and low temperature (25 °C).

- Characterize the solid residues obtained after the ergosterol extraction from mushrooms in order to investigate their potential as a source of high-value polysaccharides and proteins.

- Assess the usefulness of mushroom residues, obtained after the ergosterol extraction, in the production of oil-in-water emulsions suitable for spray drying, and evaluate its effect on the characteristics of the solid microcapsules and the oxidative stability of the encapsulated oil.

- Evaluate the usefulness of a flour obtained from artichoke by-product (bracts) in the microencapsulation of lipids, analyzing its effect on the characteristics and stability of the oil-in-water emulsions, the final microcapsules, and the oxidative stability of the encapsulated oil.
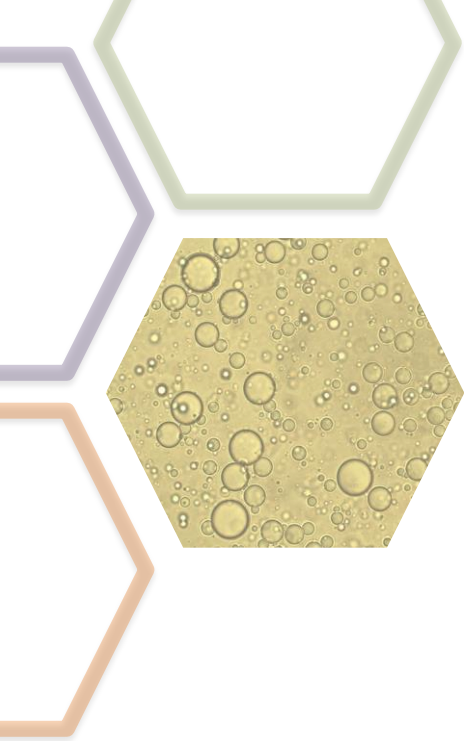
RESULTS AND DISCUSSION

# CHAPTER 1

## Effects of acoustic power and pH on pectin-enriched extracts obtained from citrus by-products. Modelling of the extraction process

Mónica M Umaña, María E Dalmau, Valeria S Eim, Antoni Femenia*

and Carmen Rosselló

*Doctoral thesis Mónica María Umaña Zamora*
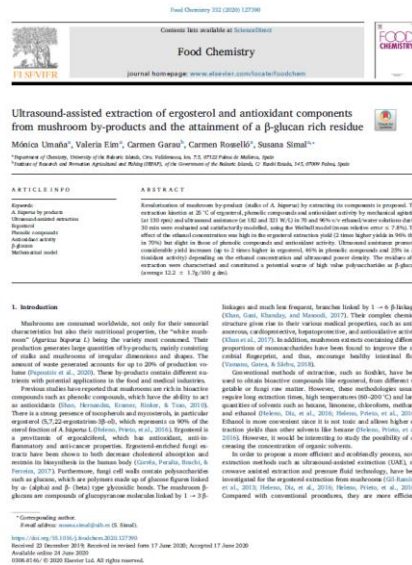
# CHAPTER 2

## Ultrasound-assisted extraction of ergosterol and antioxidant components from mushroom by-products and the attainment of a β-glucan rich residue

Mónica Umaña, Valeria Eim, Carmen Garau, Carmen Rosselló, Susana Simal

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# CHAPTER 3

## Stabilization of oil-in-water emulsions with a mushroom (*Agaricus bisporus*) by-product

Mónica Umaña, Christelle Turchiuli, Valeria Eim, Carmen Rosselló, Susana Simal

# CHAPTER 4

## Addition of a mushroom by-product in oil-in-water emulsions for the microencapsulation of sunflower oil by spray drying

Mónica Umaña, Christelle Turchiuli, Carmen Rosselló, Susana Simal

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**LWT**

**Evaluation of the addition of artichoke by-products to O/W emulsions for oil microencapsulation by spray drying**
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| | Beatriz Llavata |
| | Susana Simal, Dr. |

| Abstract: | This study aimed to evaluate the use of artichoke bracts in oil microencapsulation by spray drying. Thus, 1% and 2% w/w of this material was added to sunflower O/W emulsions to partially replace maltodextrin and substitute Tween®20. Emulsions were compared with a control containing only maltodextrin as wall material and Tween®20 as emulsifier. The emulsion containing 2% of artichoke exhibited higher (p<0.05) viscosity and stability against coalescence and flocculation (24 h) and 20% higher encapsulation efficiency after spray drying, compared with control. The three microcapsules showed similar microstructure, density, porosity, flow properties, and Tg. Microcapsules containing artichoke exhibited, on average, 15% larger particles, 19% lower moisture content, and 11% lower solubility, besides perceptible colour changes. Microcapsules containing artichoke (2%) showed lower oxidation indicators content (37%) after spray drying and over 2 months of controlled storage (35°C, 50% relative humidity) than control. After 90 days, decreases in linoleic acid were observed in all the samples (up to 24%), with increases of oleic and saturated fatty acids. The control showed the highest increase in saturated fatty acids (73%). Hence, artichoke bracts can be exploited for their application in lipid microencapsulation because of their emulsifier properties and the oxidative protection they provide. |

# CHAPTER 5

## Evaluation of the addition of artichoke by-products to O/W emulsions for oil microencapsulation by spray drying

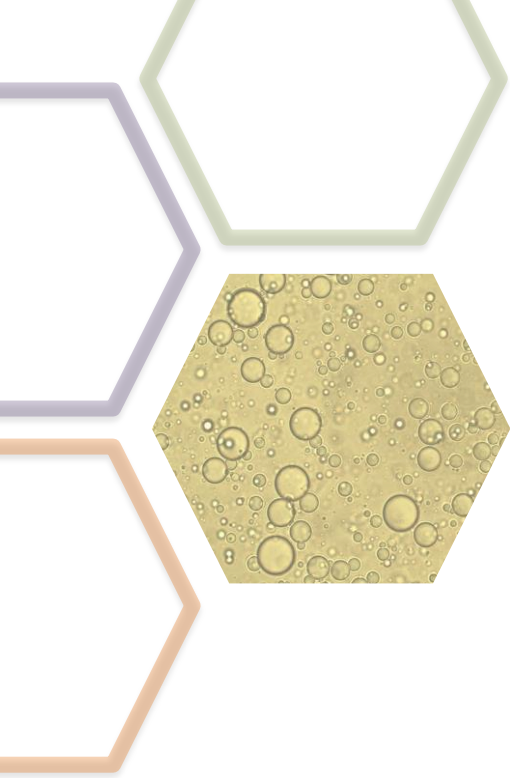Mónica Umaña, Paweł Wawrzyniak, Carmen Rosselló, Beatriz Llavata, Susana Simal

CONCLUSIONS

# 4. CONCLUSIONS

Based on the results obtained in this work, the following conclusions can be stated:

1.  The application of high-power ultrasound increased the extraction yield of pectic polysaccharides from orange by-products, this increase being highly dependent on the solvent pH.

    1.1.  The extraction yields obtained with high-power ultrasound (542-794 W/L) were significantly ($p<0.05$) higher (105-147 % at pH 1.5 and 40-80 % at pH 2.0) than those obtained with mechanical agitation (82 rpm). And the extraction yields were up to 178 % higher at pH 1.5 than at pH 2.0.

    1.2.  The pH of the solvent affected the degree of methylation (DM) of the extracted pectins, it being significantly ($p<0.05$) higher at pH 1.5 than at pH 2.0 (about 20 % higher).

    1.3.  Prolonged high-power ultrasound application up to 60 min might have promoted the extraction of non-pectic polysaccharides such as hemicelluloses or even cellulose.

    1.4.  The second-order rate model properly simulated the pectin extraction kinetics (mean relative error ≤ 7.4 %). The maximum extraction yield (Ymax) and the initial extraction rate (h) significantly ($p < 0.05$) increased up to 175 and 68 % respectively with the high-power ultrasound application.

2.  The application of high-power ultrasound increased the extraction yield of ergosterol, phenolic compounds, and antioxidant activity from mushrooms stalks. The ergosterol extraction yield was significantly ($p < 0.05$) higher in 96 % ethanol than in 70 % ethanol probably because of the low polarity of the ergosterol molecule.

    2.1.  The ergosterol, phenolic compounds, and antioxidant activity extraction yields increased 123-200 %, 20-27 % and 17-25 %, respectively in 70 % ethanol; and 16-20 %, 27-46 % and 10-19 %, respectively in 96 % ethanol, when applying high-power ultrasound (182-321 W/L).

    2.2.  The effect of the ethanol concentration was remarkable in the ergosterol extraction yield (up to 213 % higher in 96 % ethanol than in 70 % ethanol), negligible ($p > 0.05$) in the extraction yield of phenolic compounds and slight in the antioxidant activity extraction yield (up to 11 % higher in 70 % ethanol).

    2.3.  The highest extraction yields were obtained with 321 W/L of ultrasound power density, in 96 % ethanol for ergosterol (45 % of the initial content), in 70 % ethanol for the antioxidant activity (55 % of the initial antioxidant activity), and regardless of the ethanol concentration (70 or 96 %) for the phenolic compounds (average 52.5 ± 2.2 % of initial content).

    2.4.  The ergosterol and phenolic compounds contents and the antioxidant activity exhibited high correlations (Pearson's correlation coefficient ≥ 0.96).

2.5. The Weibull model satisfactorily simulated the extraction kinetics (MRE ≤ 7.8 %). The equilibrium yield ($Y_{eq}$) significantly ($p < 0.05$) increased with the high-power ultrasound application up to 203 % for ergosterol; 32 % for the phenolic compounds; and 30 % for the antioxidant activity.

3. The solid mushroom residue obtained after the extraction of ergosterol, phenolic compounds, and antioxidant activity (mushroom concentrate), rich in β-glucans and proteins, was useful to produce oil-in-water emulsions suitable for spray drying. The powders obtained after spray drying exhibited high oil encapsulation efficiency (∼89 %) and good oxidative stability.

3.1. The emulsions containing 5.0 and 7.5 % w/w of mushroom concentrate showed shear-thinning behaviour, higher ($p < 0.05$) viscosity (32 and 77 % higher, respectively), similar droplets size (average $d_{50}$ of 2.4 ± 0.5 μm), and better stability than the control emulsion (produced with maltodextrin as wall material and Tween®20 as an emulsifier). However, the emulsions containing lower amounts of mushroom concentrate (1.5 and 3.0 % w/w) exhibited undesirable characteristics such as a high presence of floccules (23 and 16 % oil volume, respectively) probably due to bridging flocculation.

3.2. The mushroom concentrate (5.0 and 7.5 % w/w) improved the emulsion stability through the viscosity increase, steric hindrance, and probably a Pickering effect. The Z-potential indicated that the electrostatic repulsion was not a relevant stabilizing force.

3.3. The use of mushroom concentrate (5.0 % w/w) in the emulsion formulation resulted in high encapsulation efficiency (about 89 %) after spray drying and prevented the oil oxidation during drying and controlled storage (35 °C and 50 % RH, 1 month) keeping other characteristics of the microcapsules within satisfactory values.

4. An artichoke by-product (flour obtained from artichoke bracts) rich in polysaccharides, proteins, and antioxidant compounds was successfully used to produce oil-in-water emulsions suitable for spray drying. The microcapsules obtained with this material showed higher encapsulation efficiency than the control and better oxidative stability.

4.1. The emulsions containing the artichoke flour (1.0 and 2.0 % w/w) showed higher ($p < 0.05$) apparent viscosity (39 and 63 % higher, respectively) and similar droplets size (average $d_{50}$ of 2.7 ± 0.1 μm) than the control (produced only with maltodextrin as wall material and Tween®20 as an emulsifier). The high oil flocculation (13 %) in the 1.0 % artichoke flour emulsion advised against this formulation.

4.2. The use of 2 % of artichoke flour in the emulsion formulation improved (p > 0.05) the droplet size stability, the encapsulation efficiency after spray drying, and prevented or decreased oxidation during drying and controlled storage (35 °C and 50 % RH, 2 months), maintaining other quality indicators within satisfactory values.

Overall, from the results obtained in this thesis, it could be concluded that it is possible to obtain bioactive compounds from different agri-food by-products and the extraction of these components could be intensified by the application of high-power ultrasound. Moreover, these materials could be applied to a food industry process taking advantage of their inherent emulsifying and antioxidant properties.
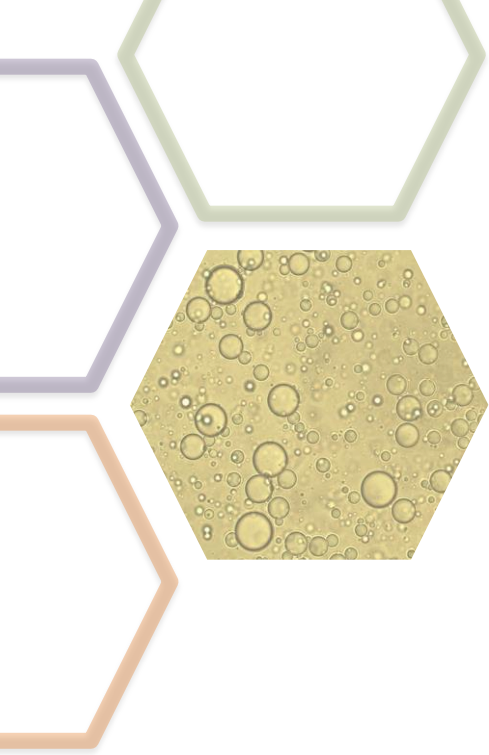
FUTURE WORK

## 5. FUTURE WORK

This thesis evaluated the intensification of the extraction of pectins from orange by-products by applying high-power ultrasound and covered both the extraction yields and chemical characteristics of the extracts, such as DM and monosaccharides composition. However, it would be interesting to determine the effect of high-power ultrasound on other parameters such as the molecular weight or technological properties (e.g gelling capacity, and emulsifying properties) of the extracted pectins for their future application as a food additive.

The extraction of ergosterol and phenolic compounds with antioxidant activity was successfully intensified by applying high-power ultrasound. It would be necessary to stabilize the liquid extracts to facilitate their addition to food matrices. Thus, these extracts could be transformed into powders through spray drying or complex coacervation.

The microencapsulation of lipids by spray drying, using two different by-products (mushroom concentrate and artichoke flour), was investigated obtaining promising results. As the success of a microencapsulation process by spray drying is highly dependent on the spray drying condition, it would be interesting to study the effect of the different operational variables (inlet temperature, feed rate, aspiration rate, and so forth) on the encapsulation efficiency and other technological properties of the powders. Moreover, similar studies could be conducted with a wider range of fruit, vegetable, and mushroom by-products to assess their potential as emulsifiers and/or wall materials in microencapsulation. In addition, it would be interesting to compare heterogeneous materials, such as those evaluated in this thesis, with purified compounds (e.g purified polysaccharides or proteins isolates) to determine whether the purification process is necessary or not. Other potential integrations of these new materials into novel food products of suitable quality to meet market requirements should also be explored.
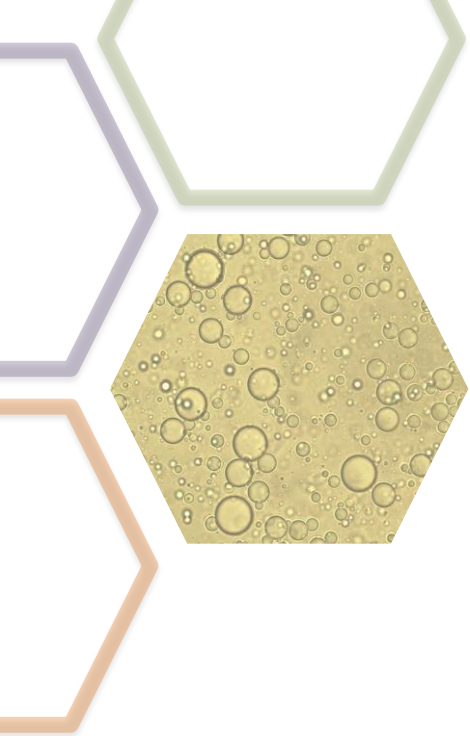
ANNEX

## ANNEX I

## Image analysis

Image analysis was used to obtain the droplet/particle size distribution of liquid emulsions and powders (Chapter 5). This analysis was applied to micrographs obtained with optical microscopy for liquid emulsions, and with SEM for powders, using the ImageJ 1.52a software (Rasband, 2020). In the case of the liquid emulsions, the image analysis was also used to determine the volume percentage of flocculated oil (Chapter 3 and 5).

Figure A1 illustrates the process used to obtain the droplet size distribution of oil-in-water emulsions with image analysis. Firstly, a scale was settled in the software using a standard of a known size. Images were transformed into the 8-bit type and into binary and, for some micrographs, it was necessary to enhance the contrast as well. Thereafter, a boundary for each droplet was constructed using the "watershed" command. Depending on the micrographs and the emulsions, in some cases, it was necessary to use the command "fill holes" before constructing the boundary. The "analyze particle" command was used to obtain the area of each droplet. A macro was used to perform this analysis automatically with several micrographs of the same sample, in such a way that at least 30,000 droplets were analyzed for each emulsion. From the droplets' area, the diameter and volume were calculated assuming that the droplets were spherical, and the total oil volume was estimated by summing the volume of every analyzed droplet. Thereafter, the full droplet size range was divided into classes according to the diameters of the droplets, and the % oil volume within each class was stipulated. Finally, the % volume of each class was plotted against the central position of the class to obtain the volume droplet size distribution.
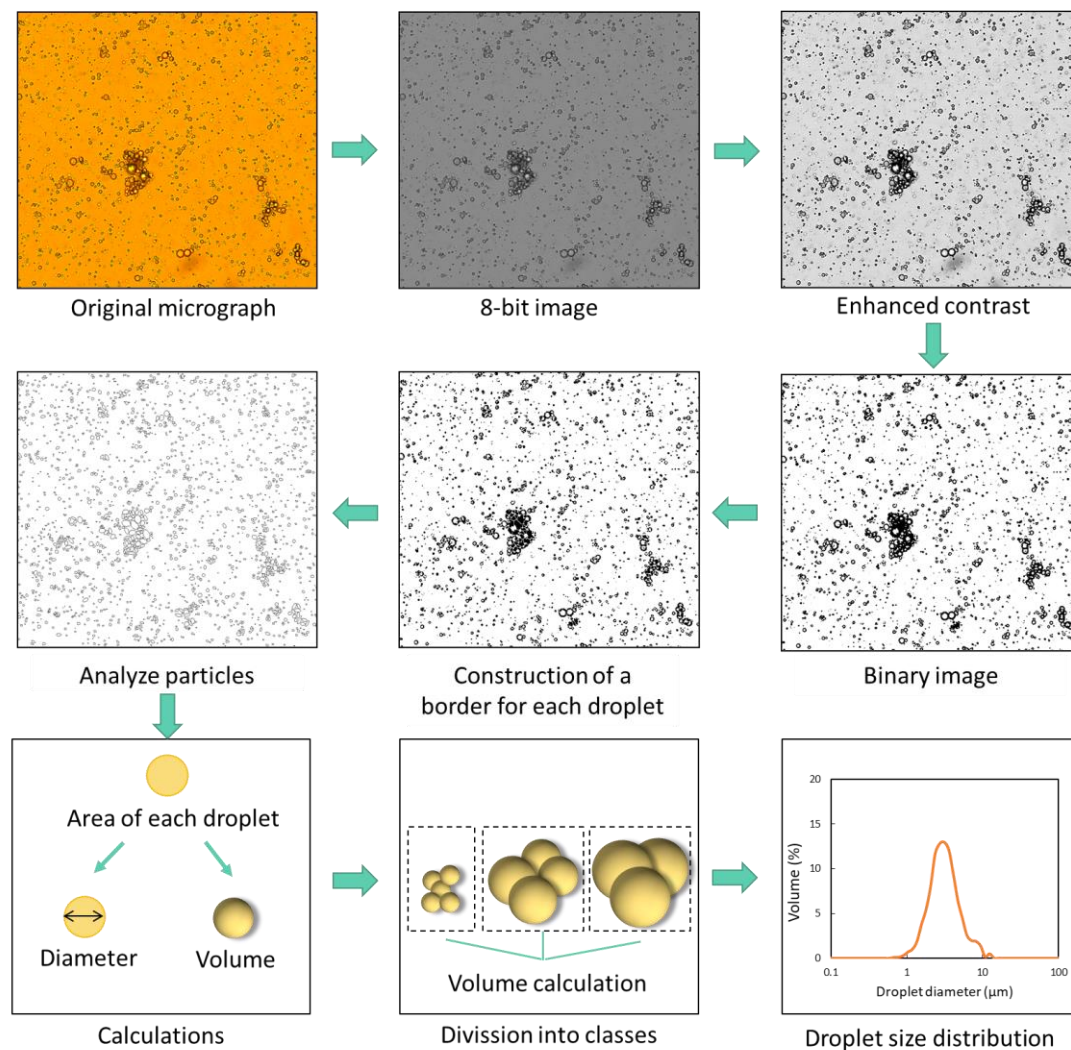
**Figure A1.** Schematic representation of the image analysis to determine the droplet size distribution of oil-in-water emulsions

Figure A2 shows the process used to calculate the flocculated oil in oil-in-water emulsions. This process was carried out with the same micrographs used to determine the droplet size distribution of the emulsion. The micrographs were transformed into binary and, thereafter, the "minimum filter" and the "find maxima" commands were used to discard the individual droplets that did not belong to the floccules. After these commands, the borders of the droplets were unclear, thus, the function "dilate" or/and the function "fill holes" were used, before reconstructing the boundaries. The area of each individual droplet, that belonged to floccules, was obtained automatically, and the volume of the droplets was calculated from the area. Finally, the percentage of flocculated oil was determined by dividing the volume of flocculated oil between the total oil volume which was estimated as described previously.
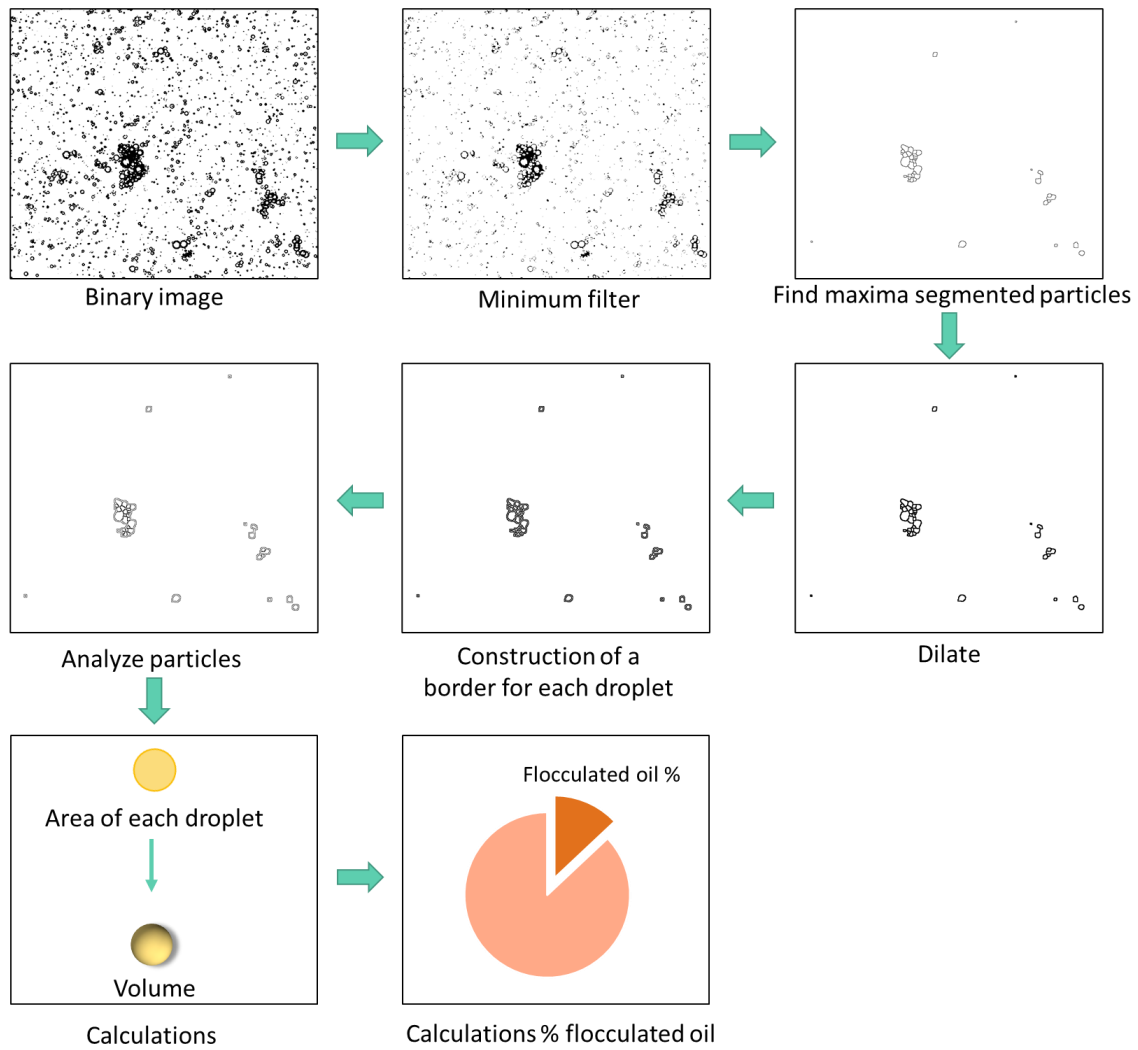
**Figure A2.** Schematic representation of the image analysis to determine the % of flocculated oil in oil-in-water emulsions.

## ANNEX II

## Contributions to congresses

From the studies of this doctoral thesis, the following contributions to national and international congresses were carried out:

1. **Title:** Extracción acústica de polisacáridos de subproductos de naranja, efecto de la potencia acústica y del pH
   **Authors:** Mónica Umaña, Esperanza Dalmau, Marina Calahorro, Remedios González, Valeria Eim
   **Event:** CyTA-CESIA 2017
   **Kind of event:** Congress
   **Participation:** Poster
   **City:** Madrid, Spain
   **Date:** 17-19 May 2017

2. **Title:** Propiedades funcionales de higos (ficus carica, Var. Mission) recubiertos con una película comestible preparada a partir de Aleo Vera, alginato y aceite de oliva
   **Authors:** Francesca Comas, Rafael Minjares-Fuentes, Carmen Molina, Mónica Umaña y Antoni Femenia
   **Event:** CyTA-CESIA 2017
   **Kind of event:** Congress
   **Participation:** Poster
   **City:** Madrid, Spain
   **Date:** 17-19 May 2017

3. **Title:** Influence of the Acoustically Assisted Low Temperature Drying of Lemon Peel on the Pectin Extraction Yield
   **Authors:** Mónica Umaña, Beatriz Rayo, Henry Vaquiro, Jose Bon, Oscar Rodríguez
   **Event:** 21st International Drying Symposium
   **Kind of event:** Congress
   **Participation:** Poster
   **City:** Valencia, Spain
   **Date:** 11-14 September 2018

4. **Title**: Revalorisation of agrofood by-products according to their potential uses: Extraction of compounds of interest by application of power ultrasound
   **Authors**: Mónica Umaña
   **Event** PhD. Students training
   **Kind of event**: 6th The Baltic University Programme Students Conference 2018
   **Participation**: Oral communication
   **City**: Rogów, Poland
   **Date**: 25-29 November 2018

5. **Title**: Efecto de la aplicación de ultrasonidos y del solvente en la extracción de ergosterol a partir de subproductos de champiñón
   **Authors**: Mónica Umaña, Susana Simal, Remedios González, Esperanza Dalmau, Cristina Reche
   **Event**: X Congreso Nacional Cyta CESIA
   **Kind of event**: Congress
   **Participation**: Poster
   **City**: Leon, Spain
   **Date**: 15-17 Mayo 2019

6. **Title:** Modelización de la extracción acústica de compuestos antioxidantes de residuo de naranja
   **Authors:** Esperanza Dalmau, Valeria Eim, Mónica Umaña, Juan Cárcel, Antoni Femenia
   **Event:** X Congreso Nacional Cyta CESIA
   **Kind of event:** Congress
   **Participation:** Poster
   **City:** Leon, Spain
   **Date:** 15-17 May 2019

7. **Title**: Use of mushroom fibre concentrate to stabilize oil-in-water emulsion by spray drying
   **Authors**: Mónica Umaña, Christelle Turchiuli, Cristina Reche, Carmen Rosselló, Susana Simal
   **Event:** 7th European Drying Conference Eurodrying 2019
   **Kind of event:** Congress
   **Participation:** Oral communication
   **City:** Turin, Italia
   **Date:** 10-12 July 2019

*Doctoral thesis Mónica María Umaña Zamora*

8. **Title**: Artichoke by-product as wall material in the microencapsulation of sunflower oil

   **Authors**: Mónica Umaña, Susana Simal, Artur Lewandowski, Carmen Rosselló, Ireneus Zbiciński

   **Event:** 7th European Drying Conference Eurodrying 2019

   **Kind of event:** Congress

   **Participation**: Poster

   **City**: Turin, Italia

   **Date**: 10-12 July 2019

9. **Title**: Revalorización integral de subproductos mediante la extracción acústica de compuestos de interés

   **Authors**: Mónica Umaña

   **Event:** VIII Jornadas Doctorales y las III Jornadas de Divulgación Científica del Grupo 9 de Universidades

   **Kind of event:** Doctoral conferences

   **Participation**: Poster

   **City**: Saragossa, Spain (online)

   **Date**: 23-25 November 2020

10. **Title:** Revalorización de subproductos de la industria agroalimentaria

    **Authors**: Mónica Umaña

    **Kind of event:** Congress

    **Event:** I Congreso Internacional de Investigación-USO 2020

    **Participation**: Guest conference

    **City**: Sonsonate, El Salvador (online)

    **Date**: 23-27 November 2020