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Original

Availability:
This version is available http://hdl.handle.net/11390/1172155.11 since 2022-01-28T18:14:41Z

Publisher:

Published
DOI:10.1016/j.ultsonch.2019.104954

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04 February 2025

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PII: S1350-4177(19)31665-7
DOI: https://doi.org/10.1016/j.ultsonch.2019.104954
Reference: ULTSON 104954

To appear in: *Ultrasonics Sonochemistry*

Received Date: 21 October 2019
Revised Date: 20 December 2019
Accepted Date: 29 December 2019

Please cite this article as: S. Plazzotta, R. Ibarz, L. Manzocco, O. Martín-Belloso, Optimizing the antioxidant biocompound recovery from peach waste extraction assisted by ultrasounds or microwaves, *Ultrasonics Sonochemistry* (2019), doi: https://doi.org/10.1016/j.ultsonch.2019.104954

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Optimizing the antioxidant biocompound recovery from peach waste extraction assisted by ultrasounds or microwaves

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Abstract

The possibility to valorize peach juice waste, either frozen or air-dried, through microwave (MAE) and ultrasound assisted extraction (UAE) was evaluated. MAE power, UAE amplitude and time were optimized using a 2^2-factorial design. For frozen waste, optimal MAE (540 W, 50 s) and UAE (23%, 120 s) processes gave extracts presenting analogous content (on 100 g dry matter) of polyphenols (309-317 mg GAE), flavonoids (94-120 mg QE), anthocyanins (8-9 mg CGE), and similar antioxidant activity (2.1-2.2 mg TE). Extracts from dried waste resulted higher in polyphenols (630-670 mg GAE) but lower in flavonoids (75-90 mg QE), anthocyanins and vitamin C (not detectable). Although developing an energy density 2-fold higher than that of UAE, MAE more efficaciously extracted vitamin C (108 mg/100 g dm) and required half extraction time (50 s). MAE would also be less impactful than UAE in terms of greenhouse gas emission and energy requirements on industrial scale. The industrial valorization of peach waste through the application of microwave and ultrasound assisted extraction requires quantitative data, able to encourage company interest and investment. This study not only identifies optimal MAE and UAE parameters to assist the extraction of peach waste bioactive compounds but also provides a preliminary estimation of the potential economic and environmental impact on an industrial scale of these technologies.

Keywords: peach waste; waste valorization; microwave; ultrasound; feasibility

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1. Introduction

More than 15 million metric tons of peaches are annually processed into juices worldwide (FAO, 2010). Peach juice industry produces a huge amount of waste, mainly represented by skin, seeds and some pieces of fruit. Depending on the ripeness of the peaches, approximately 10 % is discarded during processing (Argun & Dao, 2017). Peach waste is rich in bioactive compounds such as vitamin C and polyphenols, which show a prominent antioxidant activity (Adil, Çetin, Yener, & Bayindirli, 2007; Redondo, Arias, Oria, & Venturini, 2017; Redondo, Venturini, Luengo, Raso, & Arias, 2018). Although the extraction of bioactive compounds has been proposed for the valorization of peach waste, the main issue related to its management is the high moisture content, making it quickly prone to microbial spoilage (Ajila, Brar, Verma, & Prasada Rao, 2012). For this reason, peach waste intended for valorization is commonly frozen or dried, possibly modifying the content, composition and extractability of bioactive compounds.

The sustainable valorization of peach waste requires the use of green extraction technologies, which are energy-efficient and environmental-friendly, such as microwave (MAE) and ultrasound assisted extraction (UAE) (Rombaut, Tixier, Bily, & Chemat, 2014).

MAE is based on electromagnetic radiation, usually at the frequency of 2.45 GHz, which favors extraction from plant materials due to ionic conduction and dipole rotation. Ionic conduction refers to the migration of the charge species, such as ions, under the effect of the microwave-induced electric field. Dipole rotation involves dipolar molecules, such as water, attempting to align themselves with the alternating microwave electric field. Both these effects generate a “friction” between the moving molecules and the medium, enhancing the extraction. Moreover, the increase of internal pressure of the cell can disrupt plant structure, favoring the release of target bioactive compounds (Ameer, Shahbaz, & Kwon, 2017; Chen & Spiro, 1994; Vinatoru, Mason, & Calinescu, 2017).

UAE exploits the propagation into the extraction solvent of low frequency (20-24 kHz) high-energy sound waves, which generate alternate phases of compression and rarefaction, leading to acoustic cavitation (Medina-Torres, Ayora-Talavera, Espinosa-Andrews, Sanchez-Contreras, &

Pacheco, 2017). The latter is considered the driving force in sonochemical induced effects. This phenomenon refers to the formation, growing and subsequent collapse of cavitation bubbles (Canselier, Delmas, Wilhelm, & Abismail, 2002). Bubble implosion generates extremely high energies, producing local increase of pressure and temperature, as well as microjets and shockwaves that can disrupt external structure of plant tissues, effectively releasing target bioactive compounds (Chan, See, Yusoff, Ngoh, & Kow, 2017).

In order to boost the industrial application of these green extraction technologies, data relevant to their relative extraction performances, economic and environmental impact are needed. In this regard, different tools can be used. Firstly, extraction performances could be evaluated using response surface methodology (RSM), that offers a large amount of information with a smaller number of experiments, with respect to other traditional experimental designs, and allows observing the interaction effect of the independent parameters on the response, as well as identifying optimal treatment conditions (Baş & Boyaci, 2007). Secondly, energy density (E_V), defined as the total heating energy experienced by a unit volume of extraction, can be used. Being independent on specific parameters such as MAE power and UAE amplitude, it can be used to compare the extraction efficiency of the two technologies (Chan et al., 2017). Finally, an estimation of possible investment costs, energetic requirements and environmental impact of plants required for the industrial application of MAE and UAE would represent the starting point for their scaling-up (Simeoni, Nardin, & Ciotti, 2018).

Based on these considerations, the aim of the present study was to identify optimal MAE and UAE treatment parameters to obtain antioxidant extracts from peach waste, either frozen or dried. Moreover, the effects of MAE and UAE were compared based not only on the energy density involved in the extraction process but also on an estimation of their investment cost and environmental impact on an industrial scale.

2. Materials and Methods

2.1 Reagents

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The used reagents were absolute ethanol (Scharlau, Barcelona, Spain); bidestilled water (Milli-Q system, Millipore, Bedford, USA); Sodium carbonate (Carlo Erba, Milan, Italy); Folin-Ciocalteu reagent, Gallic acid: 3,4,5-Trihydroxybenzoic acid 97%, Sodium acetate anhydrous, Aluminum chloride, Quercetin: 2-(3,4-Dihydroxyphenyl)-3,5,7-trihydroxy-4H-1-benzopyran-4-one, DTT: 1,4-Dithiothreitol, Potassium chloride, Meta-phosphoric acid, L-ascorbic acid, $ABTS^{\cdot+}$: 2,2'azino-bis(3-ethylbenzothiazoline-6-sulphonic acid), Trolox: (±)-6-hydroxy-2,5,7,8-tetramethylchromane-2-carboxylic acid 97%, Potassium persulfate (Sigma Aldrich, St. Louis, U.S.A.).

2.2 Peach waste

Ground peach waste, either frozen (-18 °C) or dried (140 °C), was kindly furnished by Indulleida S.A. (Alguaire, Lleida, Spain). Frozen and dried peach wastes were characterized by a moisture amount of 85.0 ± 1.1 and 4.0 ± 1.0 g/100 g, respectively. Moisture content of the wastes were determined by gravimetric method (AOAC, 1997). Frozen peach waste was thawed at 4 °C prior to use. Both frozen and dried peach wastes were equilibrated at 20 °C before use.

2.3 Extract preparation

Peach waste dispersions at 0.70 g/g ethanol concentration were obtained. Peach waste dispersions (120 g) were manually mixed for 1 min, covered with a plastic cap to avoid solvent evaporation during extraction treatments, subjected to extraction treatments and filtered using 1.2 μm filters (Sartorius 17593-100 cellulose acetate 25 mm/1.20 μm, Filtros, Anoia, Spain) to remove the solid residue, obtaining the extracts. The latter were stored in the dark at -18 °C until analysis. Control extracts were prepared by filtering the peach waste dispersions immediately after the manual mixing, without any further assisted extraction procedure.

2.4 Assisted extraction procedures

An ultrasonic processor (Hieschler Ultrasonics GmbH, mod. UP400S, Teltow, Germany), with a titanium probe of 10 mm diameter, operating at constant frequency of 24 kHz and an amplitude of 20 % to 100 % or a domestic microwave (Mig225, Orbegozo, Murcia, Spain) working at frequency

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of 2450 MHz and at power levels of 180 to 900 W, were used. The levels of the ultrasounds (US) amplitude and microwaves (MW) power were selected based on the maximum operative range of the equipment. The treatment times were selected based on preliminary trials. On the one hand, the maximum time was selected when the temperature of the medium does not reach the evaporation temperature of the solvent mixture ethanol:water (70:30). Therefore, the molar percentage for the solvent in this mixture was 47.73 %, and the boiling temperature for this molar concentration is 80 ºC. In this regard, the maximum time was defined as the maximum treatment time at the highest assisted extraction procedure (900 W and 100 % for MW and US, respectively) when temperature did not exceeded the solvent mixture boiling temperature. On the other hand, the minimum treatment time was selected as the extraction time needed to be able to quantify an enhancement of the biocompounds content in the extract compared to those of the untreated extract.

2.4.1 Ultrasounds assisted extraction (UAE)

Peach waste dispersions were introduced into 250 mL capacity beakers (110 mm height, 60 mm internal diameter). The probe was placed in the center of the sample, with a 20 mm immersion depth. In this case, US treatments were performed at different amplitudes (20, 60, 100%, corresponding to 25, 75, 125 μm) and times (20, 70, 120 s).

2.4.2 Microwaves assisted extraction (MAE)

Peach waste dispersions were introduced into 1 L capacity beakers (145 mm height, 110 mm internal diameter). Afterwards, MW treatments were performed at different nominal powers (180, 540, 900 W) and times (10, 30, 50 s).

2.5 Extraction performances of MAE and UAE

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2.5.1 Temperature measurement

Sample temperature was measured just before and immediately after MAE, and during UAE by a copper-constantan thermocouple probe connected to a portable data logger (mod. TP100, XS Instruments, Carpi, Italy).

2.5.2 Total phenolic content (TPC)

Total phenolic content was determined using the Folin-Ciocalteu method (Singleton & Rossi, 1965), adapted to a 96-well microplate. 20 µL of ethanolic extract was mixed with 100 µL of Folin-Ciocalteu reagent (0.1 mL/mL) and 80 µL of sodium carbonate (150 g/L) and stored 90 min at room temperature in the dark. Absorbance was measured at 750 nm using a UV/VIS Thermo Multiskan Spectrum spectrophotometer (Thermo Scientific, Waltham, USA). Ethanol blanks (0.70 g/g) were run in each assay. A calibration curve was built with gallic acid (0–500 mg/L). Results were expressed as mg of gallic acid equivalents (GAE) per 100 g of dry matter (dm).

2.5.3 Total flavonoid content (TF)

Total flavonoids were evaluated using the method of Humadi and Istudor (2008), adapted to a 96-well microplate. 25 µL of ethanolic extract was mixed with 140 µL deionized water, 10 µL sodium acetate (82 g/L) and 10 µL aluminium chloride (100 g/L) and stored 40 min at room temperature in the dark. The absorbance was determined at 405 nm using a UV/VIS Thermo Multiskan Spectrum spectrophotometer (Thermo Scientific, Waltham, USA). Blanks containing water instead of sodium acetate and aluminium chloride were run in each assay. A calibration curve was built with quercetin (0–1000 mg/L). Results were expressed as mg of quercetin equivalents (QE) per 100 g dm.

2.5.4 Total anthocyanin content (TA)

Total anthocyanin content was evaluated by differential pH method (Chaovanalikit & Wrolstad, 2004). 2.5 mL of ethanolic extract was added to 2.5 mL of 45.0 g/L metaphosphoric acid solution containing DTT (7.2 g/L). Two 1 mL aliquots of the obtained mixture were added with 1 mL of

pH 1.0 buffer (1.9 g/L potassium chloride) or pH 4.5 buffer (32.8 g/L sodium acetate). Absorbance of the two mixtures was measured using a CECIL 2021 spectrophotometer (Cecil Instruments Ltd., Cambridge, UK), at both 520 (A_{520}) and 700 nm (A_{700}). TA (mg/L) was calculated using (eq. 1):

$$TA = \frac{A \times W \times DF \times 1000}{\varepsilon \times L} \qquad \textit{(eq. 1)}$$

$$A = [(A_{520} - A_{700})pH1.0] - [(A_{520} - A_{700})pH4.5] \qquad \textit{(eq. 2)}$$

where A is complex absorbance, measured according to (eq. 2), ε is the cyanindin-3-glucoside molar absorption coefficient (26,900 $L \cdot mol^{-1} \cdot cm^{-1}$), L is the cell path length (1 cm), W is the molecular weight of cyanindin-3-glucoside (449.2 Da), DF is the dilution factor. Data were expressed as mg of cyanindin-3-glucoside equivalents (CGE) per 100 g dm.

2.5.5 Vitamin C content (VIT C)

Vitamin C content was determined based on the procedure proposed by Odriozola-Serrano, Hernández-Jover and Martín-Belloso (2007). 2.5 mL of ethanolic extract was added to 2.5 mL of 45 g/L metaphosphoric acid solution containing DTT (7.2 g/L). The mixture was homogenized, passed through a Millipore 0.45 μm membrane and injected in the HPLC system. The latter was equipped with a 600 Controller and a 486 Absorbance Detector (Waters, Milford, MA) working at 245 nm. Samples were introduced onto the column through a manual injector equipped with a sample loop (20 ll). The flow rate was fixed at 1.0 mL/min at room temperature. A reverse-phase C18 Spherisorb® ODS2 (5 μm) stainless steel column (4.6 mm 250 mm) was used as stationary phase. The mobile phase was a 0.1 g/L solution of sulphuric acid adjusted to pH 2.6. A calibration curve was built with L-ascorbic acid (0-50 mg/L). Results were expressed as mg of vitamin C (VIT C) per 100 g dm.

2.5.6 Antioxidant activity (AA)

TEAC assay was performed according to Al-Duais, Müller, Böhm and Jetschke (2009) with some modifications. 25 mL of $ABTS^{\cdot+}$ radical cation aqueous solution (3.6 g/L) was added to 25 mL of

potassium persulfate aqueous solution (0.7 g/L) and stored in the dark for 16 h at room temperature. The solution was diluted with ethanol until reaching an absorbance at 750 nm of 0.700 (± 10%) and maintained at 30 °C. 10 µL of ethanolic extract was mixed with the prepared solution and absorbance was read at 750 nm after 5 min using a UV/VIS Thermo Multiskan Spectrum spectrophotometer (Thermo Scientific, Waltham, USA). Ethanol blanks (0.70 g/g) were run in each assay. A calibration curve was built with Trolox (0.005–0.250 mg/L). Results were expressed as mg of trolox equivalents (TE) per 100 g dm.

2.6 Experimental design

A response surface methodology (RSM) was used to evaluate the performances of MAE and UAE in the extraction of TPC, TF, TA, VIT C and their effect on the AA of frozen and dried peach waste extracts. A 2^2-factorial design was used for both MAE and UAE, considering as independent variables MAE power and treatment time, UAE amplitude and treatment time, respectively. For each factor, extreme lower and upper values were identified and combined to form the factorial part of the design (4 factorial points). MAE power was set at 180 and 900 W, MAE time at 10 and 50 s, UAE amplitude at 20% (25 µm) and 100% (125 µm), UAE time at 20 and 120 s. To complete the design, 1 central point was defined. For MAE and UAE, the central point conditions were: 540 W power and 30 s time; 60% (75 µm) amplitude and 70 s time, respectively. All the factorial points were replicated twice, and the two replicates were assigned to two different blocks. Every analytical analysis was carried out in triplicate. The central point was replicated 3 times in each block. The full set of sampling points is reported in Supplementary **Tab** S1, S2, S3 and S4. Experimental data were fitted to a polynomial response surface. The response function was predicted by the following equation (eq. 3):

$$Y_i = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k-1} \sum_{j>1}^{k} \beta_{ij} X_i X_j \qquad \textit{(eq. 3)}$$

where Y is the response, B_0, B_i and B_{ij} are the constant, linear and interaction regression coefficients, respectively, and X represents the independent variables. RSM was employed for the experimental

design, data analysis, model building and contour plot generation using the Design Expert 7.01 software (Stat Ease Inc., Minneapolis, MN, USA).

2.7 Comparison of MAE and UAE

2.7.1 Optimization and validation

An optimization in the range of the studied parameters was carried out according to the method described by Derringer and Suich (1980). The highest desirability represented the most adequate condition to concomitantly reach the highest levels of TPC, TF, TA, VIT C and AA in the extracts. A set of 8 experiments was carried out to validate the predictive models of the experimental design in the optimized conditions. The determination coefficients between the predicted and the experimental data were taken as indicator of prediction accuracy.

Extracts obtained by MAE and UAE under the optimized conditions were then compared to determine differences in extraction performances of the two procedures, using Students' *t*-test for independent samples ($p<0.05$), performed using R (The R foundation for statistical computing, v.3.1.1).

2.7.2 Energy density

Optimal MAE and UAE treatments were compared based on developed energy density (E_V, J/mL), which corresponds to the energy dissipated per volume unit of the product, measured by calorimetric analysis (eq. 4) (Chan et al., 2017):

$$E_V = P_V \cdot t \qquad \textit{(eq. 4)}$$

where P_V is the power density (W/mL) and t is the treatment time (s). P_V indicates the heating power experienced by a unit volume of extraction solvent (eq. 5).

$$P_V = \frac{m\, C_P \left(\frac{\partial T}{\partial t}\right)}{V} \qquad \textit{(eq. 5)}$$

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where m is the sample mass (g), C_p is the solvent specific heat (2.964 $Jg^{-1}°C^{-1}$), V is the sample volume (mL) and $\delta T/\delta t$ (°C/s) is the heating rate during treatment.

2.7.3 Economic, environmental and scaling-up investigation

A survey was conducted on companies producing industrial MAE and UAE extractors, on both laboratory and industrial scale. Extractor working capacity, cost and power supply requirements were used to estimate investment cost (€), energetic demand (kWh/kg of extract) and environmental impact (kg CO_2/kg of extract) of the optimised MAE and UAE procedures, under the assumption that the same extraction yields would be obtained on an industrial scale, given the same processing conditions (Albarelli, Santos, Cocero, & Meireles, 2016). The environmental impact was measured through emitted carbon dioxide, using the proper emission conversion factors of electricity (0.422) for the Italian electricity production system (Simeoni et al., 2018).

3. Results and discussion

3.1 Peach waste control extracts

The waste material considered in this study is the residue obtained by the peach industrial juicing process. Due to its high moisture content, such material is quickly prone to spoilage and should be thus frozen or dried immediately after production, in order to allow further processing and valorisation. For this reason, the peach waste considered in the present work was either frozen or air-dried.

In order to compare the two matrices, untreated (control) extracts were obtained from frozen and dried peach wastes and characterized for bioactive composition and antioxidant activity (**Tab** 1). The total phenolic content (TPC) of the dried waste (416 ±7 mg GAE/100 g dm) was significantly higher ($p<0.05$) than that of frozen one (204 ± 4 mg GAE/100 g dm), probably due to the effect of air-drying. The latter, in fact, turned peach waste into porous flour with high extractive surface (Londoño-Londoño et al, 2010). Air-drying also causes the formation of novel compounds such as Maillard reaction derivatives, which are known to react with Folin reagent used for TPC determination

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(Echavarria, Pagan, & Ibarz, 2013; Mrkìc, Cocci, Dalla Rosa, & Sacchetti, 2006). On the contrary, total flavonoids (TF) resulted higher ($p<0.05$) in extracts frozen waste (32 ± 3 mg QE/100 g dm) as compared to dried waste (12.1 ± 0.8 mg QE/100 g dm) (**Tab** 1), probably due thermal degradation induced by air-drying (140 ºC), as also observed in *Centella asiatica* and onions (Zainol, Abdul-Hamid, Bakar, Dek, 2009; Sharma et al., 2015). The anthocyanins (TA), and vitamin C (VIT C) determination confirmed the detrimental effect of air-drying on peach waste: these compounds, in fact, were not detectable in the dried waste. Indeed, Kara and Erçelebi (2013) found a 90% anthocyanin reduction in mulberry juice upon thermal treatment at 80 °C. Similarly, Vikram, Ramesh and Prapulla (2005) reported a 50% VIT C degradation in orange juice upon only 3 min heating at 90 °C. As a result, the AA of the dried waste (97 ± 9 mg TE/100 g dm) resulted significantly lower ($p<0.05$) than that of the frozen matrix (131.0 ± 0.3 mg TE/100 g dm) (**Tab** 1), since air-drying induced the bioactive degradation.

3.2 Extraction performances of MAE and UAE

The efficacy of MAE power (W) and UAE amplitude (%), and of extraction time (s) on bioactive content and antioxidant activity of frozen and dried peach waste extracts was evaluated through RSM, using a 2^2-factorial design. Obtained data are summarized in **Fig** 1 and 2, and comprehensively reported in the supplementary material (**Tab** S1, S2, S3 and S4).

As expected, the applied treatments resulted in extract heating, due to the thermal effect of MAE and UAE, which increased with power, amplitude and treatment time (**Fig** 1 and 2). Such thermal effect was expected, since an increase in these parameters is known to increase the energy amount delivered to the treated sample (Chan et al., 2017). In fact, in both cases, energy delivered to the sample is proportional to treatment time. In addition, MAE-induced heating increases with microwave power, which is converted into heat energy into the sample (Ameer et al., 2017). Similarly, in the case of UAE, the thermal effect increases with amplitude, since at high amplitudes the number of compression and rarefaction cycles of ultrasonic waves increases, leading to an

intensification of cavitation-induced phenomena (Al-Dhabi, Ponmurugan, & Maran, 2017). An increase in extract temperature is expected to promote extraction efficacy, due to tissue softening and a concomitant increase in diffusion rate (Zheng et al., 2013).

In fact, the TPC of extracts from both frozen and dried peach waste was maximized by increasing MAE power, UAE amplitude and treatment time (**Fig** 1 and 2), reaching values about 1.5 higher than those of control extracts (**Tab** 1). A similar increase in the TPC with MAE power and time was observed in extracts from *Orthosiphon stamineus* and citrus mandarin peel (Chen et al., 2018; Hayat et al., 2009). Similarly, UAE amplitude and treatment time were reported to favor TPC extraction from *Orthosiphon stamineus* (Chen et al., 2018), orange peel (Khan, Abert-Vian, Fabiano-Tixier, Dangles, & Chemat, 2010) and acerola fruit (Le & Le, 2012).

Similar to TPC, the highest TF values were obtained at the highest MAE power and UAE amplitude, combined with the longest treatment time (**Fig** 1 and 2). For both assisted procedures, those extraction conditions allowed obtaining TF values about 3 and 6 times higher than those obtained in the control extract (**Tab** 1), for frozen and dried peach waste, respectively. These results were not expected, since these treatments resulted in temperatures higher than 50 °C (**Fig** 1 and 2), which have been reported to promote flavonoid degradation (Zainol et al., 2009; Sharma et al., 2015). Nevertheless, the obtained results can be explained considering that both MAE and UAE can generate heating in a reduced time, leading to a so-defined high temperature-short time treatment, which has been reported to increase extraction rate without damaging flavonoids in different fruit derivatives, despite the high temperature reached during treatment (Saikia, Mahnot, & Mahanta, 2015). To this regard, Raner, Strauss, Vyskoc and Mokbel (1993) reported that MAE power variation from 500 to 1000 W on a model system had no significant effect on the flavonoids if the applied treatment time allowed maintaing temperature at values lower than 110 °C. Similarly, TF degradation in hawthorn seed extracts obtained by UAE was observed only at temperatures higher than 90 °C (Pan, Yu, Zhu, & Qiao, 2012).

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Regarding TA and VIT C, these biocompounds resulted not detectable in all the extracts obtained from dried peach waste, independently on MAE and UAE intensity, confirming that the drying process was probably responsible for the thermal degradation of these compounds. In frozen peach waste extracts, the highest TA content was obtained upon the application of low MAE power or UAE amplitude, applied for long times (**Fig** 1 and 2), which allowed obtaining concentrations 26 and 38 times higher than those of control extract (**Tab** 1), respectively. A similar effect of MAE power and time were also observed in the case of VIT C, which reached values about double than the control extract (**Tab** 1) upon 50 s MAE treatment at 180 W (**Fig** 1). As also reported for blueberry and orange (Vikram et al., 2005; Zheng et al., 2013), these results can be attributed to the reduced temperature reached during such treatments (**Fig** 1 and 2), limiting thermal damage of these thermolabile bioactive compounds (Chan et al., 2017; Le & Le, 2012; Sang et al., 2017). By contrast, our results indicated a low UAE efficacy in VIT C extraction (**Fig** 2). None of the applied UAE treatments, in fact, resulted in a significant increase of VIT C as compared to the control extract (**Tab** 1). This can be explained by the counterbalancing effect of UAE-induced extraction and rapid VIT C oxidation during the treatment. VIT C, in fact, is well known for its preferential reaction with ·OH radicals generated during sonication, favoring its oxidation (Sprinz, Beckert, & Brede, 1998). In this regard, similar results were also observed in orange and strawberry juice (Tiwari, O'Donnell, Patras, & Cullen, 2008; Tiwari, O'Donnell, Muthukumarappan, & Cullen, 2009).

With regards to AA, all extracts presented the highes values upon the application of high MAE power and UAE amplitude, associated to long extraction times (**Fig** 1 and 2), reaching values that double those of the control extract (**Tab** 1). Being efficiently extracted in these conditions, TPC and TF should be probably mainly accounted for extract AA, as also reported in other studies (Li et al., 2012). Interestingly, extracts from frozen and dried peach waste showed analogous AA values. Naturally occurring phenolic compounds are expected to be responsible for the AA detected in frozen peach waste extracts. On the contrary, the AA of dried peach waste extracts should be mainly

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attributed to thermal-induced compounds, formed during drying, such as partially oxidized polyphenols and Maillard reaction products (Nicoli, Anese, & Parpinel, 1999).

3.3 Comparison of MAE and UAE

3.2.1 Optimization and validation

The obtained results were elaborated in order to identify treatment parameters more significantly affecting the extraction, as well as the equations describing bioactive concentration and antioxidant activity of the obtained extracts as a function of MAE and UAE parameters (**Tab** 2 and 3). In the case of MAE, all models resulted significant, indicating a strong dependence of bioactive concentration and antioxidant activity on MAE power and treatment time. Similarly, UAE models describing the effect of UAE amplitude and time on the extraction of TPC, TF, TA and on AA resulted significant. By contrast, UAE model for VIT C extraction resulted not significant, confirming the scarce efficacy of sonication in extracting this compound.

Subsequently, the optimal treatment conditions, concomitantly exhibiting the maximum TPC, TF, TA, VIT C and AA were identified using the desirability function (**Tab** 4). In the case of frozen waste, MAE treatment at 540 W and 50 s and UAE treatment at 23% amplitude and 120 s were identified. In the case of dried waste, the desirability function identified the optimal MAE treatment at 900 W and 50 s and the optimal UAE treatment at 100% amplitude and 120 s. Further extracts were thus obtained by applying the identified optimal conditions. The observed extraction performances of optimal MAE and UAE treatments are reported in **Tab** 4. These data were used to validate the obtained models. In particular, the correlation between the observed and predicted values resulted always higher than 0.72, indicating that the equations obtained for each assay (**Tab** 2 and 3) adequately fitted experimental results.

Within both frozen and dried peach waste extracts, no significant differences ($p \geq 0.05$) were found between the extraction performances of optimized MAE and UAE treatments. The only exception

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was represented by VIT C, which was successfully extracted only by MAE (**Tab** 4). As already anticipated, this is possibly due to the oxidative degradation of this compound induced by sonication.

By contrast, the comparison of results obtained in frozen and dried peach waste extracts confirmed that drying degraded most of the peach waste bioactive compounds (TF, TA, VIT C), suggesting frozen peach waste as a better raw material for the extraction of antioxidant molecules (**Tab** 4).

3.2.2 Energy density

The identified optimal MAE and UAE treatments cannot be properly compared, being based on completely different extraction principles and thus parameters, i.e. MAE power and UAE amplitude. Energy density (E_v), defined as the amount of heating energy delivered to the system (energy dissipated) by MAE and UAE, has been widely recognized as a tool to compare the efficacy of these treatments (Chan et al., 2017). Therefore, to identify the most efficient extraction treatment, energy density (E_v) developed by MAE and UAE optimized procedures was calculated. The E_v delivered by MAE optimized treatment resulted 4 and 2 times higher than that developed by UAE optimal treatment in frozen and dried waste extracts, respectively (**Tab** 4). Although this suggests UAE to be more efficient than MAE, since a lower energy was required to reach not significantly different extraction performances, it must be underlined that UAE optimal treatment was characterized by an extraction time more than double than that required by MAE optimal treatment (**Tab** 4). These results agree with those obtained by Hayat et al. (2009) and Chan et al. (2017) on citrus mandarin peel and *Orthosiphon stamineus* fruit. These Authors found no significant differences in bioactive extraction performances of optimized MAE and UAE treatments, but highlighted MAE treatment time to be much shorter (4-50 times) than that of UAE. This can be attributed to the fact that microwave process produces a volumetric heating, so that the entire sample volume is evenly heated (Desai, Parikh, & Parikh, 2010). By contrast, ultrasound propagation does not allow a homogeneous energy distribution into the treated sample, since energy is located around the sonication probe, leading to the need for longer treatments (Ameer et al., 2017; Canselier, et al., 2002).

By comparing the results obtained in frozen and dried peach waste extracts, it is evident that drying not only degraded most of the peach waste bioactive compounds but was also associated to MAE and UAE procedures developing values of E_v much higher than those associated to frozen peach waste. This is surely due to the fact that the value of optimal MAE power and UAE amplitude identified for dried peach waste resulted much higher than those identified for the frozen waste (**Tab** 4).

3.2.3 Economic, environmental and scaling-up investigation

Results acquired up to now clearly indicate that frozen peach waste should be chosen over dried one for bioactive extraction, based on both bioactive content and composition, and developed energy density. It can also be inferred that UAE should be preferred over MAE, since giving similar extraction performances at lower energy density. At the same time, however, MAE would offer the advantage of reducing treatment time as compared to UAE (**Tab** 4).

Further data are thus required to characterise the identified MAE and UAE processes. In particular, the availability of data relevant to economic and environmental impact as well as on scaling-up potential could represent a chance to increase industry confidence in these technologies. In fact, although the promising results obtained by the application of these procedures in producing antioxidant extracts from peach waste, the lack of knowledge would keep industry from their implementation in real industrial process (Chemat et al., 2017). To get a first insight in these aspects, the working capacity, investment cost, energy consumption and greenhouse gas emission of MAE and UAE plants applying the identified optimal extraction procedures to frozen peach waste were compared on both laboratory and industrial scale (**Tab** 5). To this aim, a multi-objective study approach was applied (Simeoni et al., 2018). The latter was based on the collection of data on the energy consumption (kWh), investment cost (€) and working capacity (kg of extract) of MAE and UAE plants from laboratory up to large industrial scale. A simulation of the optimal MAE and UAE process identified in **Tab** 4 using laboratory and industrial-scale plants was thus carried out. Obtained data revealed that the scaling-up of microwave and ultrasound extractors to a large industrial scale

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would respectively allow dropping to about one quarter and one third both the energy consumed, and the carbon dioxide emitted for producing 1 kg of bioactive extract from peach waste (**Tab** 5). Although the comparable working capacity of equipment, UAE plants would require a higher investment cost than MAE extractors at both laboratory and small industrial scale. By contrast, at large industrial scale, both technologies would require a similar capital investment. In such a case, however, MAE would guarantee lower electrical energy consumption and greenhouse gas emission (**Tab** 5). This is probably mainly attributable to the fact that MAE optimised treatment was shown to require a shorter processing time as compared to UAE (**Tab** 4). It must be noted that for this feasibility analysis, laboratory results were scaled up under the assumption that the same yields and performances would be obtained on an industrial scale, given the same processing conditions (Albarelli et al., 2016). For this reason, obtained data are to be considered as preliminary and should be accurately validated on a real industry context. Nevertheless, they can represent a useful starting point to develop a decision support system for industries, able to rationally support the choice of investment in the most sustainable procedure for extracting bioactive compounds from peach waste.

4. Conclusions

The MAE and UAE of bioactive compounds from frozen and dried peach waste was successfully optimized by identifying optimal microwave power, ultrasound amplitude and treatment time. Frozen peach waste resulted richer than dried matrix in specific compounds such as flavonoids, anthocyanins and vitamin C, due to thermal damage induced by drying.

Both assisted extraction processes can be optimized to give comparable extraction performances in terms of total phenolic compounds, flavonoids, anthocyanins and antioxidant activity. The only exception was vitamin C, which was successfully extracted only by MAE, due to oxidative vitamin C degradation occurring during UAE.

The obtained results on process efficiency, in terms of developed energy and required time, are relevant to select the most adequate extraction technology to be used in the valorization of peach

wastes. In fact, even if energy density developed by UAE treatment resulted lower than that developed by MAE, the latter required a much lower time than UAE to give analogous extraction performances. In addition, preliminary scaling-up considerations, seem to suggest a lower environmental impact of MAE as compared to UAE. Thus, MAE can be considered a more feasible and sustainable technology for the valorization of peach waste into antioxidant extracts.

5. Acknowledgments

This work has been performed within the project SOE1/P1/E0123 Interreg Sudoe REDVALUE, Technological Alliance to Complete the Forest and Agroindustrial Production Cycle. R. Ibarz thanks the University of Lleida for her postdoctoral contract (UdL-Impuls).

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Figure captions

Fig 1. Effect of microwave power (W) and time (s) on temperature (*T*, °C), total phenolic content (*TPC*, mg GAE/100 g dm), total flavonoids (*TF*, mg QE/100 g dm), total anthocyanins (*TA*, mg CGE/100 g dm), vitamin C (*VIT C*, mg/100 g dm) and antioxidant activity (*AA*, mg TE/100 g dm) of frozen and dried peach waste extracts. *MAE* microwave assisted extraction, *ND* not detected, *dm* dry matter.

Fig 2. Effect of ultrasound amplitude (%) and time (s) on temperature (*T*, °C), total phenolic content (*TPC*, mg GAE/100 g dm), total flavonoids (*TF*, mg QE/100 g dm), total anthocyanins (*TA*, mg CGE/100 g dm), vitamin C (*VIT C*, mg/100 g dm) and antioxidant activity (*AA*, mg TE/100 g dm) of frozen and dried peach waste extracts. *UAE* ultrasound assisted extraction, *ND* not detected, *dm* dry matter.

Table headings

Tab 1. Bioactive compound concentration and antioxidant activity of control extracts obtained from frozen and dried peach waste. Data shown are a mean ± standard deviation. *TPC* Total phenolic content (mg GAE/100 g dm), *TF* Total flavonoid content (mg QE/100 g dm), *TA* Total anthocyanin content (mg CGE/100 g dm), *VIT C* Vitamin C (mg VIT C/100 g dm), **AA** Antioxidant scavenging activity (mg TE/100 g dm), ND not detectable, *dm* dry matter.

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Tab 2. ANOVA of the first-order polynomial models for extracts obtained by microwave assisted extraction (*MAE*) from frozen and dried peach waste. Adjusted determination coefficient (R^2 adj) for evaluating model goodness-of-fit and model equations are also reported. *P* power (W), *t* time (s), *TPC* total phenolic content (mg GAE/100 g dm), *TF* total flavonoids (mg QE/100 g dm), *TA* total anthocyanins (mg CGE/100 g dm), *VIT C* vitamin C (mg/100 g dm), *AA* antioxidant activity (mg TE/100 g dm), *dm* dry matter.

Tab 3. ANOVA of the first-order polynomial models for extracts obtained by ultrasound assisted extraction (*UAE*) from frozen and dried peach waste. Adjusted determination coefficient (R^2 *adj*) for evaluating model goodness-of-fit and model equations are also reported. *A* amplitude (%), *t* time (s), *TPC* total phenolic content (mg GAE/100 g dm), *TF* total flavonoids (mg QE/100 g dm), *TA* total anthocyanins (mg CGE/100 g dm), *VIT C* vitamin C (mg/100 g dm), *AA* antioxidant activity (mg TE/100 g dm), *dm* dry matter.

Tab 4. Process parameters, desirability, extraction performances and energy of optimized microwave and ultrasound treatments applied to frozen and dried peach waste extracts. *MAE* microwave assisted extraction, *UAE* ultrasound assisted extraction, *P* power (W), *A* amplitude (%), *t* time (s), *TPC* total phenolic content (mg GAE/100 g dm), *TF* total flavonoids (mg QE/100 g dm), *TA* total anthocyanins (mg CGE/100 g dm), *VIT C* vitamin C (mg/100 g dm), *AA* antioxidant activity (mg TE/100 g dm), E_v energy density (J/mL), *dm* dry matter.

Tab 5. Working capacity, investment cost, energy consumption and carbon emissions of equipment required for optimal microwave (*MAE*) and ultrasound (*UAE*) assisted extraction of bioactive compounds from frozen peach waste, at laboratory and small/large industrial scale. *P* power, *A* amplitude, *t* time.

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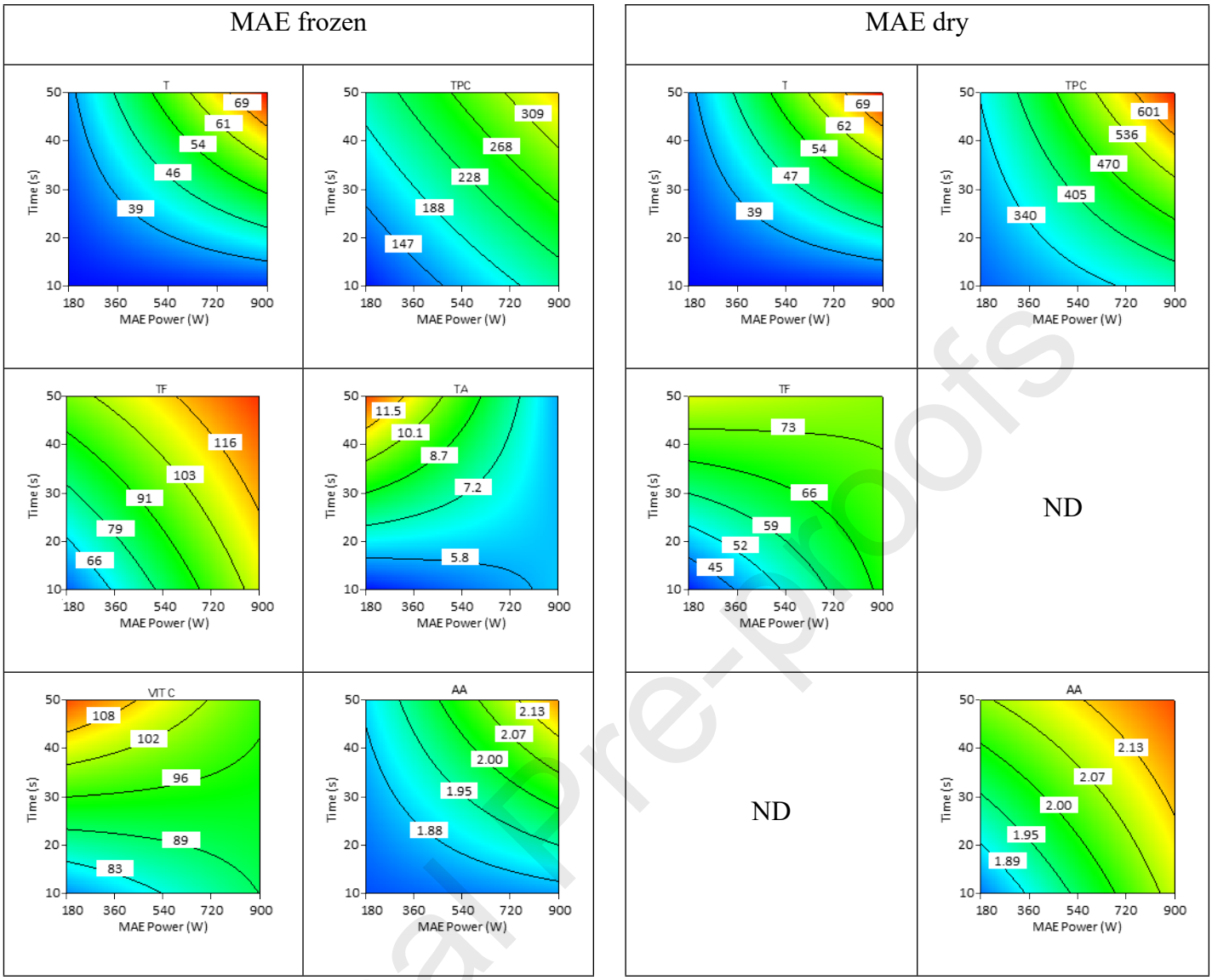

Fig 1. Effect of microwave power (W) and time (s) on temperature (T, °C), total phenolic content (TPC, mg GAE/100 g dm), total flavonoids (TF, mg QE/100 g dm), total anthocyanins (TA, mg CGE/100 g dm), vitamin C (VIT C, mg/100 g dm) and antioxidant activity (AA, mg TE/100 g dm) of frozen and dry peach waste extracts. MAE microwave assisted extraction, ND not detected, dm dry matter.

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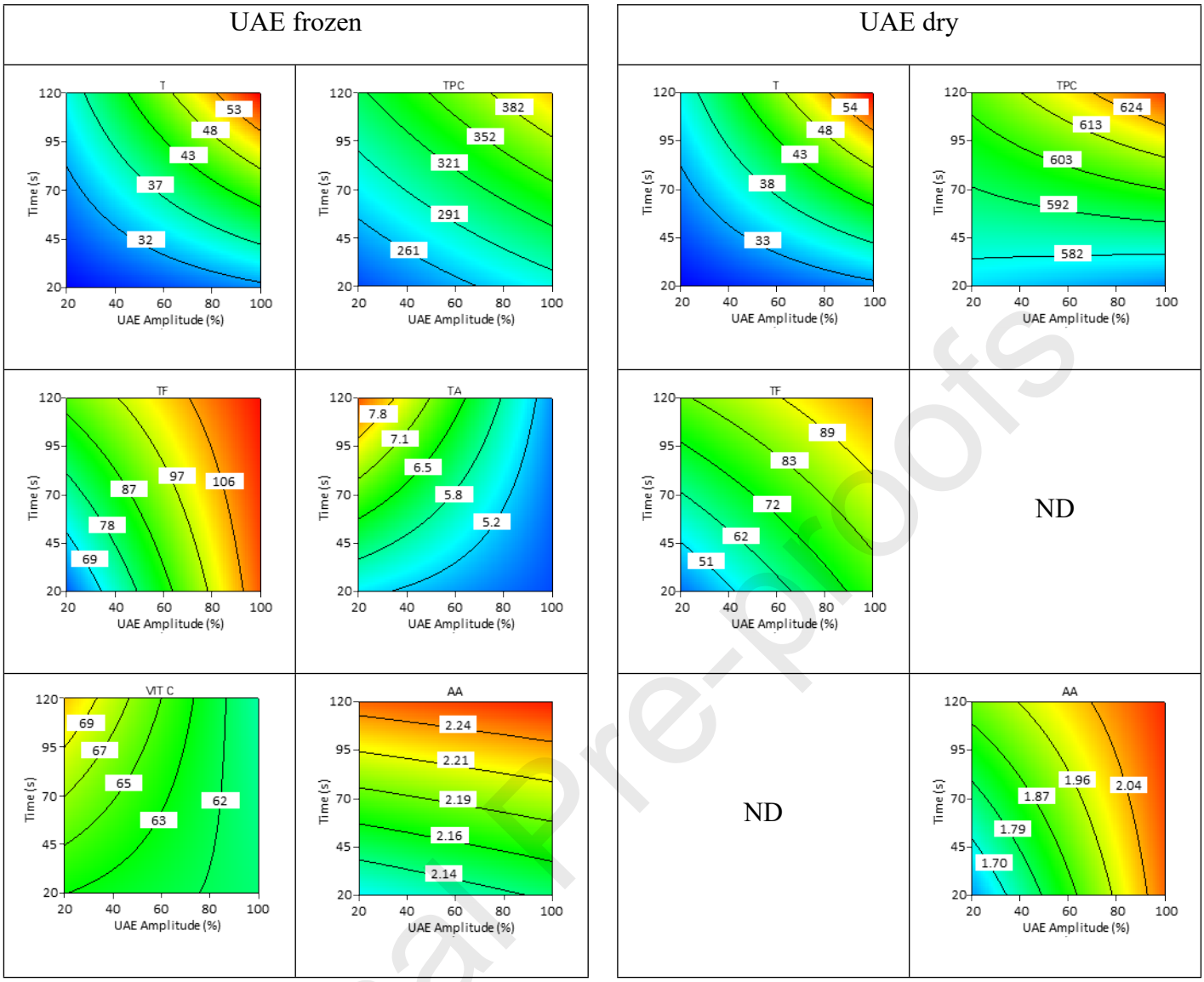

Fig 2. Effect of ultrasound amplitude (%) and time (s) on temperature (T, °C), total phenolic content (TPC, mg GAE/100 g dm), total flavonoids (TF, mg QE/100 g dm), total anthocyanins (TA, mg CGE/100 g dm), vitamin C (VIT C, mg/100 g dm) and antioxidant activity (AA, mg TE/100 g dm) of frozen and dry peach waste extracts. UAE ultrasound assisted extraction, *ND not detected, dm* dry matter.

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***Tab** 1. Bioactive compound concentration and antioxidant activity of control extracts obtained from frozen and dried peach waste. Data shown are a mean ± standard deviation. TPC Total phenolic content (mg GAE/100 g dm), TF Total flavonoid content (mg QE/100 g dm), TA Total anthocyanin content (mg CGE/100 g dm), VIT C Vitamin C (mg VIT C/100 g dm), AA Antioxidant scavenging activity (mg TE/100 g dm), ND not detectable, dm dry matter.*

Peach waste	TPC	TF	TA	VIT C	AA
Frozen	204 ± 4	32 ± 3	0.30 ± 0.04	61 ± 5	1.01 ± 0.06
Dried	416 ± 7	12.1 ± 0.8	ND	ND	0.87 ± 0.08

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Tab 2. ANOVA of the first-order polynomial models for extracts obtained by microwave assisted extraction (*MAE*) from frozen and dried peach waste. Adjusted determination coefficient (R^2 adj) for evaluating model goodness-of-fit and model equations are also reported. *P* power (W), *t* time (s), *TPC* total phenolic content (mg GAE/100 g dm), *TF* total flavonoids (mg QE/100 g dm), *TA* total anthocyanins (mg CGE/100 g dm), *VIT C* vitamin C (mg/100 g dm), *AA* antioxidant activity (mg TE/100 g dm), *dm* dry matter.

	F-value				
	TPC	TF	TA	VIT C	AA
MAE frozen					
Model	9.40 **	18.14 ***	242.81 ***	6.8 *	11.4 **
P	14.20 **	31.36 ***	123.76 ***	0.15	11.47 **
t	13.53 **	20.29 **	288.84 ***	14.13 **	16.05 **
*P * t*	0.49	2.77	315.82 ***	6.11 *	6.67 *
R^2 *adj*	0.70	0.82	0.99	0.61	0.74
Eq.	$TPC = 60.49 + 0.12$ P $+2.14$ t	$TF = 28.17 + 0.08$ P $+1.28$ t	$TA = 1.31 - 0.005$ P $+0.27\ t - 0.0003\ Pt$	$VITC = 61.71 + 1.16$t -0.001 Pt	$AA = 1.81 + 3.30 \times 10^{-5}$P $+0.0002$ t $+ 8.76 \times 10^{-6}$Pt
MAE dried					
Model	41.51 ***	6.79 *	ND	ND	6.98 *
P	59.2 ***	2.88 *			11.28 *
t	46.98 ***	11.87 **			8.37 *
*P * t*	18.36 **	5.64 *			1.29
R^2 *adj*	0.92	0.61			0.62
Eq.	$TPC = 249.15 + 0.05$ P $+0.29$ t $+ 0.008$ Pt	$TF = 17.47 + 0.05$ P $+1.27$t $- 0.001$ Pt			*AA*=1.69+0.0004 A +0.007 t

*p<0.05, **p<0.01, ***p<0.001, *ND* not detected

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Tab 3. ANOVA of the first-order polynomial models for extracts obtained by ultrasound assisted extraction (*UAE*) from frozen and dried peach waste. Adjusted determination coefficient (R^2 *adj*) for evaluating model goodness-of-fit and model equations are also reported. *A* amplitude (%), *t* time (s), *TPC* total phenolic content (mg GAE/100 g dm), *TF* total flavonoids (mg QE/100 g dm), *TA* total anthocyanins (mg CGE/100 g dm), *VIT C* vitamin C (mg/100 g dm), *AA* antioxidant activity (mg TE/100 g dm), *dm* dry matter.

	F-value				
	TPC	TF	TA	VIT C	AA
UAE frozen					
Model	8.88 **	25.44 ***	89.96 ***	NS	5.35 *
A	7.81 *	57.6 ***	130.54 ***		0.46
t	18.03 **	12.26 **	85.44 ***		15.55 **
A * *t*	0.79	6.46 *	53.89 ***		0.04
R^2 *adj*	0.68	0.87	0.96		0.54
Eq.	$TPC = 204.02 + 0.50\ \mathrm{A} + 0.75\ \mathrm{t}$	$TF = 38.91 + 0.71\ \mathrm{A} + 0.37\ \mathrm{t} - 0.003\ \mathrm{A\,t}$	$TA = 4.73 - 0.003\ \mathrm{A} + 0.04\ \mathrm{t} - 0.0003\ \mathrm{At}$		$AA = 2.08 + 0.0014\ \mathrm{t}$
UAE dried					
Model	9.30 **	4.39 *	ND	ND	12.26 **
A	1.32	5.54 *			27.18 ***
t	23.24 **	7.33 *			6.21 *
A * *t*	3.35	0.31			3.40 *
R^2 *adj*	0.69	0.48			0.75
Eq.	$TPC = 575.38 + 0.197\ \mathrm{t}$	$TF = 22.57 + 0.49\ \mathrm{A} + 0.44\ \mathrm{t}$			$AA = 1.42 + 0.007\ \mathrm{A} + 0.004\ \mathrm{t} - 0.000031\ \mathrm{At}$

*p<0.05, **p<0.01, ***p<0.001, *NS* model not significant, *ND* not detected

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Tab 4. Process parameters, desirability, extraction performances and energy of optimized microwave and ultrasound treatments applied to frozen and dried peach waste extracts. *MAE* microwave assisted extraction, *UAE* ultrasound assisted extraction, *P* power (W), *A* amplitude (%), *t* time (s), *TPC* total phenolic content (mg GAE/100 g dm), *TF* total flavonoids (mg QE/100 g dm), *TA* total anthocyanins (mg CGE/100 g dm), *VIT C* vitamin C (mg/100 g dm), *AA* antioxidant activity (mg TE/100 g dm), E_v energy density (J/mL), *dm* dry matter.

	Frozen		Dried	
	MAE	UAE	MAE	UAE
Process parameters				
P	540	-	900	-
A	-	23	-	100
t	50	120	50	120
Desirability	0.630	0.658	0.829	0.919
Extraction performances				
TPC	309.14 ± 3.22	317.33 ± 2.91	666.41 ± 20.62	636.77 ± 3.24
TF	120.47 ± 4.34	93.93 ± 2.69	74.75 ± 7.19	89.75 ± 8.37
TA	8.95 ± 0.32	8.39 ± 0.56	ND	ND
VIT C	108.04 ± 1.30	68.40 ± 3.31	ND	ND
AA	2.14 ± 0.03	2.24 ± 0.02	2.19 ± 0.05	2.12 ± 0.02
Process energy				
E_v	211.4	45.24	378.85	140.16

ND not detected; No significantly different means were identified by t-test comparing extraction performances of MAE and UAE applied to frozen or dried peach waste ($p \geq 0.05$)

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Tab 5. Working capacity, investment cost, energy consumption and carbon emissions of equipment required for optimal microwave (*MAE*) and ultrasound (*UAE*) assisted extraction of bioactive compounds from frozen peach waste, at laboratory and small/large industrial scale. *P* power, *A* amplitude, *t* time.

Extraction technique	Operative conditions	Scale	Working capacity (kg)	Cost (€)	Energy consumption (kWh/kg extract)	Carbon emission (kg CO_2/kg extract)
MAE	P = 540 W t = 50 s	Laboratory	0.5	500	0.025	0.0106
		Industrial (small)	1.5	3000	0.011	0.0047
		Industrial (large)	10	50000	0.006	0.0023
UAE	A = 23% t = 120 s	Laboratory	0.5	3000	0.033	0.0141
		Industrial (small)	1.5	10000	0.027	0.0113
		Industrial (large)	10	50000	0.010	0.0042

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Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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Peach juice waste is a source of antioxidant bioactive compounds

Peach waste drying causes flavonoid, anthocyanin and vitamin C loss

Ultrasounds and microwave can assist the extraction of peach waste bioactive compounds

Ultrasounds develop higher energy in a larger time than microwaves

Microwaves could have lower environmental impact than ultrasounds on industrial scale

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