biology and life sciences forum

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Proceeding Paper

Effect of Ultrasound-Assisted Extraction with Probe or Bath on Total Phenolics from Tomato and Lemon By-Products †

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† Presented at the 4th International Electronic Conference on Foods, 15–30 October 2023; Available online: https://foods2023.sciforum.net/.

Abstract: The aim of this study was to compare between ultrasound-assisted extraction (USAE) with probe (USAE-P) and USAE with bath (USAE-B), using different solvents (ethanol:water), on the extraction of total phenolic compounds (TPC) from tomato pomace and lemon peels by-products. The TPC after USAE with probe ranged from 1.2- to 3.1-fold and from 1.1- to 2.0-fold more than USAE with bath in tomato and lemon by-products, respectively. The solvent with the highest extraction of TPC from tomato pomace was 100% ethanol (between 1.2- and 2.6-fold more than the other solvents) while the best solvent in lemon peel was 100% water (between 1.1- and 2.0-fold more). USAE with probe demonstrated to be a clean, efficient, and a green technology for the extraction of TPC from tomato and citrus by-products.

Keywords: revalorization; green-technologies; food loss

Citation: Mozafari, L.; Cano-Lamadrid, M.; Martínez-Zamora, L.; Zapata, R.; Artés-Hernández, F. Effect of Ultrasound-Assisted Extraction with Probe or Bath on Total Phenolics from Tomato and Lemon By-Products. *Biol. Life Sci. Forum* **2023**, *26*, x. https://doi.org/10.3390/xxxxx

Academic Editor(s): Name

Published: date

1. Introduction

During the food chain, food by-products/waste are generated which are importer sources of bioactive compounds. Depending on the stage of production, they are referred to "food loss" (generated in a process carried out in the agri-food industry) or "food waste" [1]. Tomato pomace consists of high amounts of tomato peels and seeds which are currently used for animal feed and fertilizers [2]. Lycopene content in tomato peel contains at least 2- and 4-fold more than industrial waste and whole tomato, respectively [3]. On the other hand, after squeezing lemon fruits, depending on the cultivar, between 55 to 72% of the lemon is wasted consisting in peel, albedo, and seeds. Citrus pomace presents 2.5 to 4 times higher Folin–Ciocalteu reducing capacity values (connected with the amounts of phenolic compounds and ascorbic acid) compared to pulps [4]. Functional and techno-functional bioactive compounds extracted from tomato and lemon by-products such as carotenoids, flavonoids, phenolic compounds, and vitamins are of interest for their potential uses for food and pharmaceutical industry.

According to the extraction procedure to obtain such functional ingredients, the selection of solvent should be based on the nature of bioactive compound being focused. Solvents catalogued as green ones should be selected for revalorization fruit and vegetables by-products, although the recovery of the specific bioactive compounds is not the highest one. Minimal or zero usage of organic solvents has acquired great importance [5]. Among green extraction techniques of bioactive compounds, ultrasound-assisted

extraction (USAE) has emerged as a promising method to revalorize food by-products. Among the different parameters influencing the ultrasound (US) technology, we are focused on the type of the equipment: probe and bath. One of the main differences between both types is that an US with probe is submerged directly into the solution, while in an US with bath, the vessel container is immersed. Also, it important to take the intensity into account, being higher in US with probe than US with bath. In a US probe, the maximum power is the nominal power, while in a US bath, the nominal power is the minimum that can be increased due to the modulators [6].

Therefore, the aim of this study was to compare between USAE with probe and USAE with bath, using different solvents (50:50, 100:0, and 0:100 of ethanol:water), on the extraction of total phenolic compounds (TPC) from tomato pomace and lemon peels by-products.

2. Materials and Methods

2.1. Tomato/Lemon By-Products Ultrasound-Assisted Extraction (USAE)

Valkirias tomato by-products after obtaining grated tomato were obtained from Bonnysa Group (Alicante, Spain). Fino lemons were provided by Toñifruit company (Murcia, Spain). Drying and grinding pre-treatments were carried out to obtain a stable and homogeneous raw material. Samples were freeze-dried using a Telstar® LyoBeta (Terrassa, Spain). In this study, USAE with bath (USAE-B) was carried out by a Sonorex® Digiplus DL 514 BH US bath (Berlin, Germany) with a capacity of 18.7 L, using a power of 720 W, at a frequency of 35 kHz at set temperature. As to USAE with probe (USAE-P), the extraction was carried out using a sonicator with probe (387 × 203 × 216 mm) (Fisherbrand™ Q705, Madrid, Spain). During the extraction, the fixed variables were: (i) particle size (<56 μm), (ii) drying method (freeze-drying), (iii) solid:liquid ratio (1:25), (iv) temperature (50 °C), and (v) time (13 min); while the continuous variable was solvent: 50:50 Ethanol:water; 100 Ethanol; and 100 Water (Table 1). Once the samples extraction was completed, they were centrifugated to separate the solid from the extract. The extracts were stored at −80 °C until analysis.

Table 1. Experimental design and variables used in the USAE of tomato and lemon by-products.

By-Product	Drying method	% EtOH	% H_2O	Codification Solvent	Ratio Solid:Liquid	US Type	Time (min)	T (°C)
Tomato	FD	50	50	50E:50H	1:25	Probe: USAE-P	13	50
	FD	100	0	100E	1:25	Probe: USAE-P	13	50
	FD	0	100	100H	1:25	Probe: USAE-P	13	50
	FD	50	50	50E:50H	1:25	Bath: USAE-B	13	50
	FD	100	0	100E	1:25	Bath: USAE-B	13	50
	FD	0	100	100H	1:25	Bath: USAE-B	13	50
Lemon	FD	50	50	50E:50H	1:25	Probe: USAE-P	13	50
	FD	100	0	100E	1:25	Probe: USAE-P	13	50
	FD	0	100	100H	1:25	Probe: USAE-P	13	50
	FD	50	50	50E:50H	1:25	Bath: USAE-B	13	50
	FD	100	0	100E	1:25	Bath: USAE-B	13	50
	FD	0	100	100H	1:25	Bath: USAE-B	13	50

FD: Freeze-drying.

2.2. Total Polyphenolic Content

The determination of TPC was carried out according to the method described by Singleton & Rossi [7] with some modifications by Martínez-Zamora et al. [8]. The TPC was calculated using a gallic acid standard and expressed as g of gallic acid equivalent per kg of dried weigh (g GAE/kg dw).

2.3. Statistics

Box plot using XLSTAT Premium 2016 (Addingsoft, Barcelona, Spain) was conducted. Figure 2 shows the one–way ANOVA test using "sample" (n = 6 samples: 3 solvents * 2 USAE type:) for each by-product. Tukey test was used for means comparison (95% confidence level).

3. Result and Discussion

3.1. Total Polyphenolic Content of Tomato and Lemon By-Products

The TPC extraction by USAE in tomato by-products and lemon by-products is shown in Figure 1. In descriptive statistics, box plot method present Minimum/Maximum value (blue dots), 1st quartile (upper limit of the box), 3rd quartile (lower limit of the box), median (paralel line between 1st and 3rd quartile), and average (red cross). In the case of tomato by-products (Figure 1, left), minimum and maximum values were 332.6 mg TPC/kg dw and 1248.6 mg TPC/kg dw, respectively, 1st quartile was 642.4 mg TPC/kg dw, 3rd quartile was 1000.6 mg TPC/kg dw, median was 810.1 mg TPC/kg dw, and average was 808.9 mg TPC/kg dw. In the other hand, for lemon peels by-products (Figure 1, right), minimum and maximum values were 2398.8 mg TPC/kg dw and 5485.4 mg TPC/kg dw, respectively, 1st quartile was 3564.5 mg TPC/kg dw, 3rd quartile was 4836.4 mg TPC/kg dw, median was 4475.4 mg TPC/kg dw, and average was 4175.4 mg TPC/kg dw.

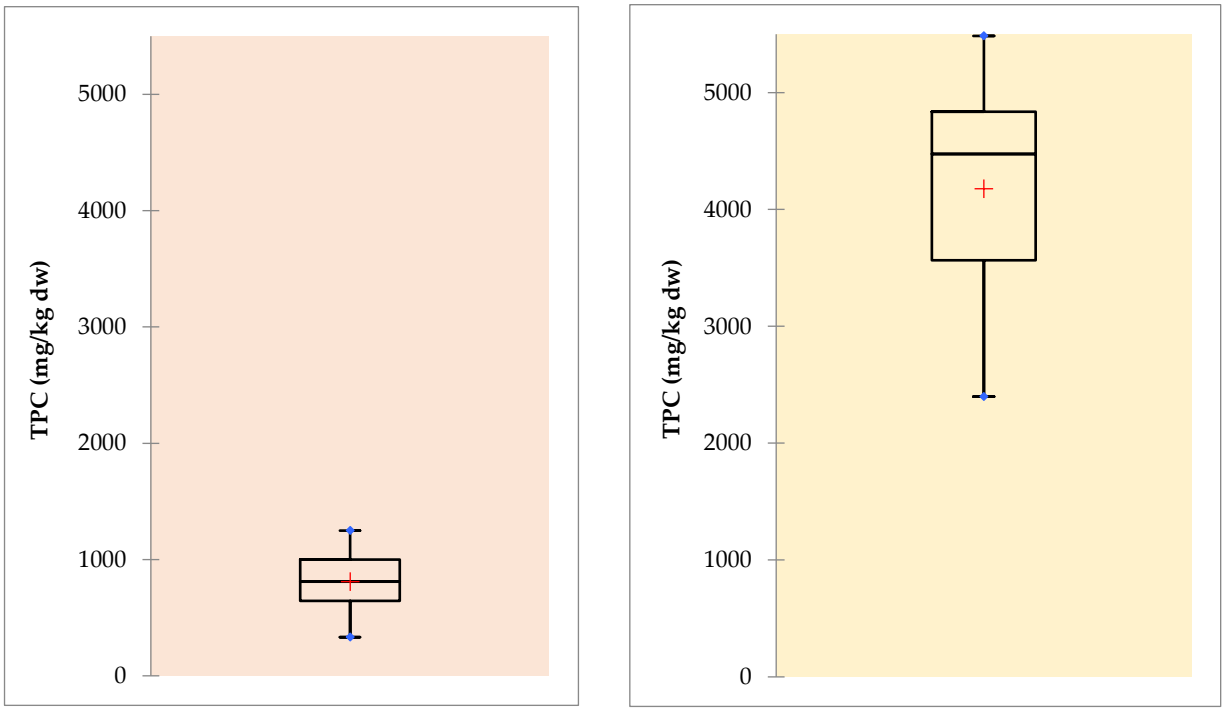

Figure 1. Box plot of the total polyphenolic content of tomato (**left**) and lemon (**right**) extracts before USAE.

Therefore, it can be observed that lemon by-products extracts presented between 2- and 5-fold more TPC than tomate by-products. Apart from a source of phenolic compounds in tomato pomace, others non-phenolic compounds such as saturated and unsaturated fatty acids, and carotenoids were previously detected and those compounds have excellent redox properties in tomato [9] and lemon [10].

3.2. Ultrasound-Assisted Extraction with Probe vs. with Bath

The effect of the solvent and type of US equipment on the TPC extraction in tomato and lemon by-products is shown in Figure 1. Focusing on the effect of solvent, significant

differences were observed among the studied solvents in both tomato and lemon by-products. The solvent with the highest extraction of TPC from tomato pomace was 100% ethanol (between 1.2- and 2.6-fold more than the other solvents) while the best solvent in lemon peel was 100% water (between 1.1- and 2.0-fold more), as also shown by previous authors [11,12]. While in the case of lemon the best results were shown with water as solvent. It is essential to mention that there are other solvents with higher capacity to extract key bioactive compounds but there are not catalogued as green solvents.

Comparing USAE-P and USAE-B, TPC after USAE-P ranged from 1.2- to 3.1-fold and from 1.1- to 2.0-fold more than USAE-B in tomato and lemon by-products, respectively. The novelty of this research is that for the first time the extraction of polyphenolic content by USAE-B is compared with the extraction by USAE-P under the effect of the same variables.

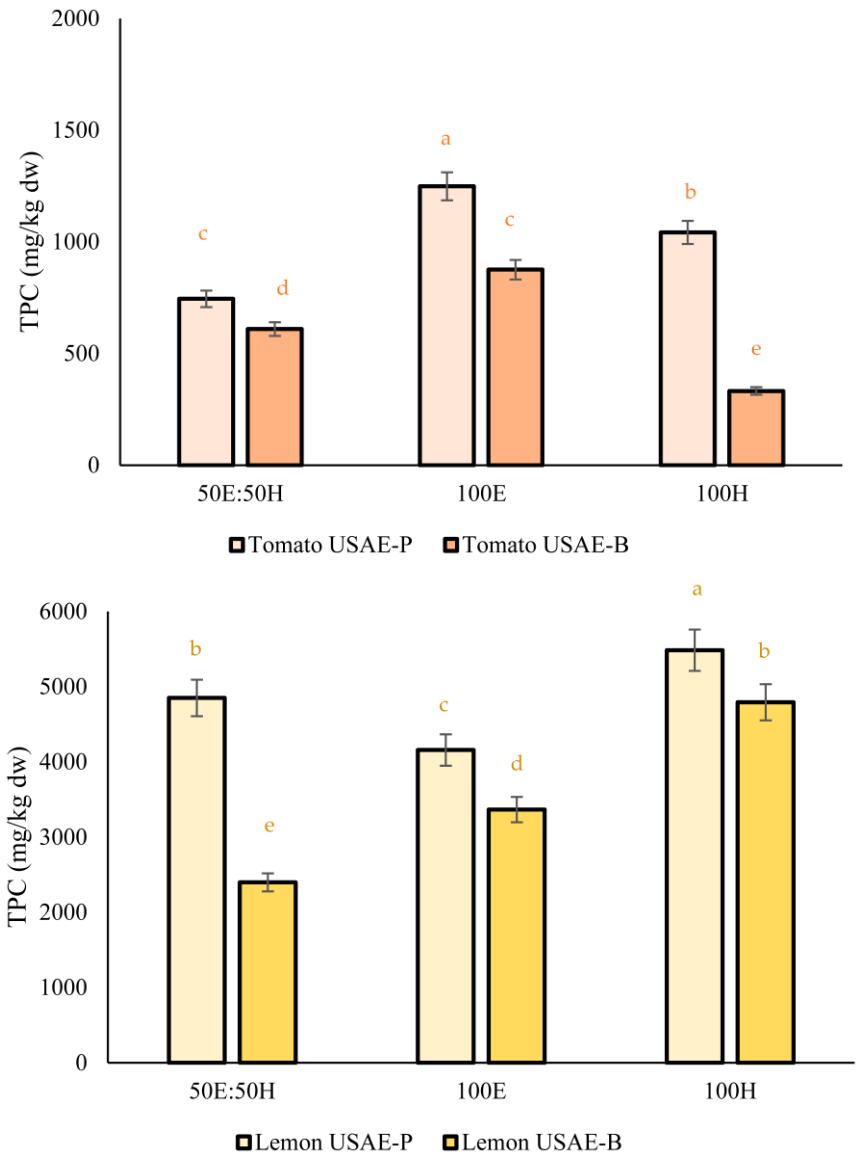

Figure 2. Total polyphenolic content of tomato (**upper graph**) and lemon (**graph below**) extracts after ultrasound-assisted extraction with probe (USAE-P) and with bath (USAE-B).

These results can be justified by the polarity, the nature, and the origin of the bio compounds extracted, as well as the way of application of the USAE. In this sense, previous authors have shown as the effectiveness of US when is applied directly to the extract

using a probe instead of in a bath in which a bottle with the extract is sited, is exponentially increased. In fact, the intensity distribution is heterogeneous in the bath; hence using probe this intensity can be applied homogenously throughout the extract for the recovery and purification of bioactive compounds. Specifically in tomato, the USAE-P has shown to be more effective for the extraction of lycopene [11,12] as well as using lemon by-products for the extraction of phenolics [10,13].

4. Conclusions

In general, it can be concluded that the type of solvent and food by-product influenced the extraction of TPC, being higher in lemon by-product than in tomato by-products. Ultrasound-assisted extraction demonstrated to be a clean, efficient, and a green alternative for the extraction of TPC and bioactive compounds with total antioxidant capacity. USAE-P was a greater tool for the solid-liquid extraction of bioactive compounds than USAE-B. Aqueous and ethanolic US extraction are green alternatives to recover TPC for lemon and tomato by-products, respectively. This research is framed within the number 12 goal (Responsible consumption and production) of the 2030 Agenda for Sustainable Development.

Author Contributions: Conceptualization, F.A.-H., M.C.-L. and L.M.-Z.; methodology, formal analysis, investigation, L.M., M.C.-L., R.Z. and L.M.-Z.; resources, F.A.-H.; data curation, L.M., R.Z., M.C.-L. and L.M.-Z.; writing—original draft preparation, M.C.-L. and L.M.-Z.; writing—review and editing, F.A.-H.; visualization, L.M., M.C.-L., L.M.-Z., R.Z. and F.A.-H.; supervision, F.A.-H.; project administration F.A.-H.; funding acquisition, F.A.-H. All authors have read and agreed to the published version of the manuscript.

Funding: Project PID2021-123857OB-I00 financed by the Spanish Ministry of Science and Innovation, the Spanish State Research Agency/10.13039/501100011033/and FEDER. This work has also been financed by the Autonomous Community of the Region of Murcia through the Seneca Foundation and the European program NextGenerationEU.

Institutional Review Board Statement:

Informed Consent Statement:

Data Availability Statement: Not applicable.

Acknowledgments: L.M.-Z. contract has been financed by the Program for the Re-qualification of the Spanish University System, Margarita Salas modality, by the University of Murcia. The M.C.-L. contract has been co-financed by Juan de la Cierva-Formación (FJC2020-043764-I) from the Spanish Ministry of Education.

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

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