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## **Valorization of tomato wastes: influence of ohmic heating process on polyphenols extraction time**

COELHO, Marta<sup>(1,2)</sup>; PEREIRA, Ricardo<sup>(1)</sup>; TEIXEIRA, JA, PINTADO, Manuela<sup>(1)</sup>

<sup>1</sup> *Universidade Católica Portuguesa, CBQF - Centro de Biotecnologia e Química Fina – Laboratório Associado, Escola Superior de Biotecnologia, Porto, Portugal*

<sup>2</sup> *CEB - Centre of Biological Engineering, University of Minho, Portugal.*

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### *Corresponding autor:*

Manuela Pintado

Universidade Católica Portuguesa, CBQF - Centro de Biotecnologia e Química Fina

Laboratório Associado, Escola Superior de Biotecnologia, Porto, Portugal

mpintado@porto.ucp.pt

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### **Abstract**

Current extraction treatments may cause degradation of biocompounds hampering their added value. Phenolic compounds extraction of *Lycopersicon esculentum* (tomato) by-products was optimized using ohmic heating (OH) as an alternative extraction technology.

Design of experiments was applied to evaluate the effect of extraction time, temperature and ethanol concentration and further optimized by a desirability function. The antioxidant activity and characterization of phenolic compounds was performed.

A significant increase of phenolics content and antioxidant activity were obtained at 70°C and 40°C ( $p < 0.05$ ). The best extraction conditions were 70 °C, 15 min and 70% of ethanol with a total phenolic content of  $2.550 \pm 0.072$  mg gallic acid equivalents/g(fw). The individual phenolic compounds were identified by HPLC-DAD analysis.

In conclusion, OH shows to have a high potential as an environmental-friendly, economical and fast process for the recovery of polyphenols from industrial tomato by-products.

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### **Introduction**

Tomato production generates substantial amounts of organic wastes with high economic and environmental costs (Bergougnoux, 2014). These agro-industrial by-products are composed of skins and pulp remnants still rich in bioactive compounds such as polyphenols and carotenoids. If properly recovered these bioactive compounds can be further incorporated into the food chain acting as functional additives for assorted products and applications. The current extraction

treatments still represent an environmental hazard due to high cost and excessive use of solvents, which can increase toxicity of the recovered compounds, thus reducing their biological value. For this there is a growing interest in alternative extraction technologies, such as Ohmic Heating (OH). OH is considered an environmentally-friendly technique (i.e. use of electrical energy) and presents several advantages when compared to conventional thermal extraction procedures, such as an uniform and faster direct

heating with a high energetic efficiency (> 90%) (R. N. Pereira & Vicente, 2010).

This study aims to optimize the extraction of phenolics, antioxidants and carotenoids from tomato wastes through the application of ohmic heating as an alternative extraction technology.

## Materials and Methods

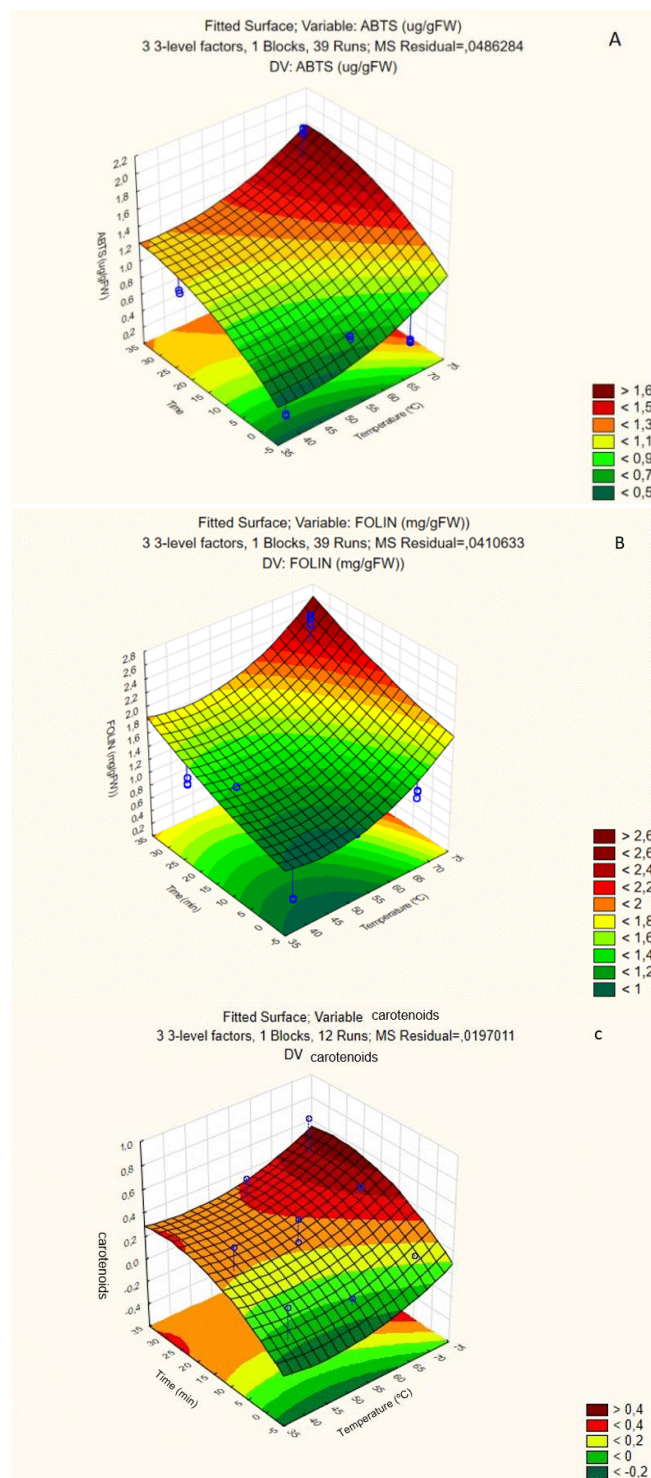
Fresh samples of industrial tomato wastes (pulp, seeds and skins) were immediately stored at -80 °C until further analysis. OH was used as a thermal assisted treatment (1:5 v/w) for the extraction of biocompounds from tomato samples as described elsewhere (Pereira, Souza, Cerqueira, Teixeira, & Vicente, 2010; Pereira, Teixeira, & Vicente, 2011; Rodrigues et al., 2015). In order to establish the optimal extraction conditions under OH a design of experiments (DOE) was applied according to a custom design of Box–Behnken  $n=2^3$ , with three levels (-1, 0, +1) and twelve experimental combinations. These experiments combined holding times of 0, 15 and 30 min, heating temperatures (40, 55 and 70 °C) and percentages of ethanol in water (0, 35 and 70 %). The holding time of 0 min, corresponds to the moment that the treatment temperature in the sample is achieved.

The characterization of phenolic compounds and carotenoids in the extracts were evaluated. A spectrometric methods were used to assess the total antioxidant activity, by the 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid (ABTS) method (Gião et al., 2007), total phenolics by Folin Ciocalteu's method (Gião et al., 2007), and total carotenoids (Kimura, Rodriguez-Amaya, & Godoy, 1990). Individual phenolic acids and carotenoids were identified by HPLC-DAD analysis as described elsewhere (C. Oliveira, Ferreira, Costa, Guerra, & De Pinho, 2004; C. M. Oliveira, Barros, Silva Ferreira, & Silva, 2015).

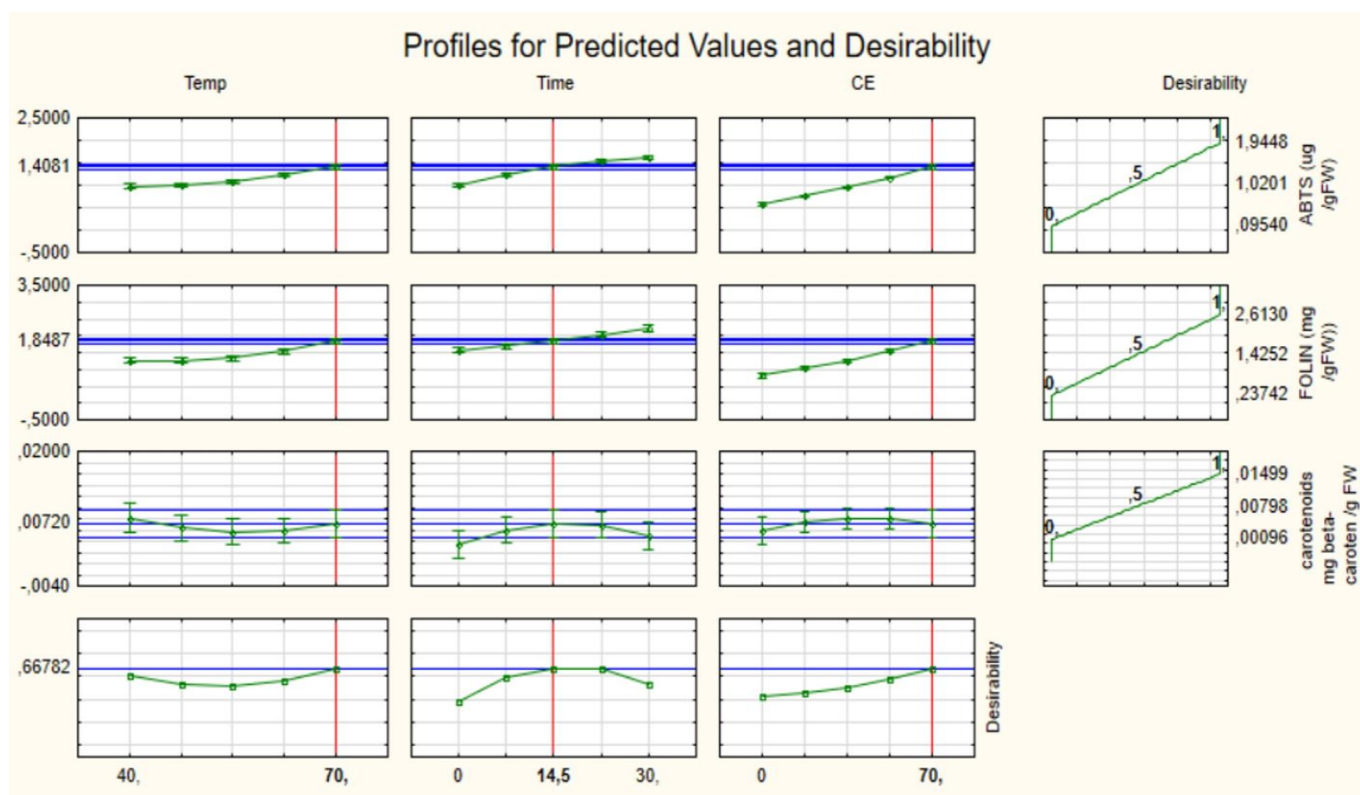
## Results and Discussion

The extracts with the highest antioxidant activity -  $1.920 \pm 0.030$  mg ascorbic acid equivalents/g fresh weight (fw) - were obtained at 70 °C for 30 min and with 70% of ethanol compared to 0.106 µg ascorbic acid equivalents/g fw obtained at 40 °C, 0 min and 0% ethanol. Results are in accordance with the literature, whereas the recovery efficiency is affected by different extraction conditions, such as, solvent, polarity, temperature, time (Kim & Chin, 2016). In addition,

with the increased of temperature a thermodynamic effect may be observed improving the solubilization (Kim & Chin, 2016; Ricardo N. Pereira et al., 2016; Shao et al., 2012).



**Figure 1- 3D Surface Plot of (A) antioxidant, (B) polyphenols and (C) carotenoids of tomato by-products.**



**Figure 2 - Profiles to predict values and desirability.**

Total phenolics also increased with temperature and percentage of ethanol - at 70 °C with 70% of ethanol and after 30 min.  $2,550 \pm 0.072$  mg gallic acid equivalents/g fw were obtained against  $0.244 \pm 0.007$  mg gallic acid equivalents/ g fw observed in the control (40 °C, 0 min, 0% ethanol). In addition, the response surface analysis indicates that the percentage of ethanol influences the antioxidant activity and recovery of phenolic compounds, while the treatment time was only significant for the extraction of carotenoids (Figure 1). Through HPLC-DAD it was possible to identify compounds such as rutin, kaempferol, naringenin, quercetin and chlorogenic acid (results not shown).

Pellegrini et. al. (2007) refers that the polyphenols content on tomato extracts depends of the type of solvent extraction and its polarization (Pellegrini et al., 2007). Furthermore the heating promotes the cells breakdown and the bioaccessibility of polyphenolic compounds (Kim & Chin, 2016; Pellegrini et al., 2007).

The results obtained indicate that, within the defined experimental boundaries, temperatures of 70 °C combined with extraction times between 15 and 30

min using ethanol solutions at 70% favour the extraction of phenolic compounds, as well as give rise to extracts with higher antioxidant capacity and distinctive phenolic composition ( $p < 0.05$ ).

OH becomes more effective at higher temperatures. Furthermore, a synergistic effect of temperature and electric fields may lead to changes on polyphenols extraction (Ricardo N. Pereira et al., 2016).

As we could verify, the optimum points reached individually for each factor did not coincide in all cases and it can be solved with a desirability function. This consists in transforming the original each response to a dimensionless desirability scale, assigns numbers between 0, representing a completely undesirable value, and 1, representing a completely desirable or ideal response value (Figure 2) (Pizarro, González-Sáiz, & Pérez-del-Notario, 2006). Optimum conditions were defined as the ones of treatment temperature, holding time and ethanol percentage, to get simultaneously the maximum concentration of antioxidant activity, phenolic compounds and carotenoids.

Through a desirability function the optimum extraction conditions were established as 70 °C, 15 min and 70 % of ethanol (Figure 2).

### Conclusion:

Results suggest that OH can be an efficient tool to be used on polyphenols recovery from tomato by-products.

The best extraction conditions for both polyphenols and carotenoids were found to be 70 °C for an extraction time of 15 min with 70 % of ethanol.

OH is a recognized environmental-friendly and economical thermal technology (energetic efficiency > 90 %) allowing for a fast and volumetric internal heating (no hot surfaces) that can enhance thermal permeabilization of vegetable tissues and thus enhance extraction of value-added compounds from tomato waste. The role of electric field and electrical frequency on extraction mechanisms should be further investigated.

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