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VALORIZATION OF ROASTED HAZELNUT CUTICLES SUPPORTED BY LABORATORY TECHNIQUES

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ABSTRACT

This paper reports the experimental results of an on-going project running at lab-scale and aimed at the valorization of roasted hazelnut cuticles through both chemical (i.e., solvent extraction) and thermochemical treatment (i.e., torrefaction) routes. In particular, the potential of using water as a green solvent for the extraction of bioactive compounds (i.e., substances of chemical-food-pharmaceutical interest, such as the polyphenols) contained in residues originated by industrial processing of hazelnuts has been investigated, applying the conventional laboratory Soxhlet extraction procedure. A subsequent valorization stage has been explored for the spent post-extraction residues versus the "as collected" ones; they lend themselves to become "renewable" solid fuels thanks to torrefaction, which is a "mild" thermochemical conversion process. The obtained results are first presented in terms of theoretical yields of the bioactive compounds of interest with respect to the original mass of hazelnut residue; in addition, the findings on torrefaction are discussed in terms of performance indexes with respect to the torrefied fuel and quantitatively expressed by correlations as a function of temperature.

1. INTRODUCTION

Hazelnut *(Corylus avellana L.)* is one of the most popular tree nuts consumed for human food worldwide, ranking second in production after almond. Turkey, specifically the Black Sea region, is the world leading producer of hazelnut, contributing over 72% to the global production, although other important producing areas include Georgia, Spain and Italy (Faostat, 2020). In Italy, the Campania region has been the leader in the field production of hazelnut in 2021, with an amount of about 25000 ton and less than half in the province of Salerno (Istat, 2021). The hazelnut skin (*perisperm*), hard shell (*pericarp*), green leafy cover (*floral bracts*) and the hazelnut tree leaf are the byproducts of the roasting, cracking, shelling/hulling, and harvesting processes, respectively.

The present paper is in the framework of a R&D project aimed at valorization of the above non-edible parts. As the public opinion is aware and under the focus of current R&D activities, residues and wastes of biogenic origin are more and more considered as a valuable source of both bioactive substances and biofuels, whatever their original moisture content is.

It is well known that a diet rich in tree nuts adds benefits because of their mono- and polyunsaturated fatty acid content (Ros and Mataix, 2006), their high level of dietary fiber (Salas-Salvadó et al., 2006), and the presence of several bioactive molecules in the kernel and skin ranging from tocopherols to arginine and to polyphenols (Andrés et al., 2002), which might exert positive cardiovascular effects such as low-density lipoprotein (LDL) protection from oxidation or enhanced endothelial function (Andrés et al., 2002). The antioxidant capacity of various nut byproducts has been widely investigated, and several works have acknowledged that nut byproducts are especially rich sources of natural phenolic compounds with potential bioactivity (Shahidi et al., 2007). Phenolic compounds are the primary bioactive components in plants. Consequently, the utilization of natural phenolic antioxidants instead of synthetic ones has recently raised considerable interest

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among food scientists, manufacturers and consumers. In particular, the hazelnut skin (Shahidi et al., 2007) and the green leafy cover (Alasalvar et al., 2006) have been investigated to exploit the content of some phenolic acids.

The bioactive compounds of interest can be separated, in principle, from the source hazelnut matrix by means of a conventional liquid-solid extraction triggered by a conventional solvent, typically an organic one. Nowadays, a switch to "greener" solvents like limonene, bioethanol or even water is being proposed in view of more environmentally sustainable processing. In any case, the extraction process generates solid residues, in a wet or dry state, which are to be disposed of. They are suitable candidates for a further valorization stage in the more general frame of energy transition, i.e., producing solid biofuels.

Therefore, the idea underlying the present paper pursues the following sequential investigation pattern: i. extracting bioactive compounds with a "green" solvent, ii. recovering and drying the solid residues, iii. producing solid biofuels by a mild thermal processing, i.e., torrefaction (Chen et al., 2021). On an experimental basis, the conventional lab based Soxhlet extraction technique and the batch torrefaction in a fluidized bed have been adopted.

For simplicity, just one of the above-mentioned hazelnut residues is taken into consideration here, i.e., roasted hazelnut skins. They are discarded upon the roasting process of the hazelnuts. The amount of hazelnut skin is about 2.5% of the total kernel weight and it is the main by-product after roasting (Bertolino et al., 2015). Based on the previous datum and the current statistics (Istat, 2021), this makes available an amount of about 1000 and 300 ton of hazelnut roasted cuticles in Italy and Campania, respectively, during the production season.

Hazelnut skin, which is the part of the hazelnut with the highest antioxidant activity, is a rich source of phenolic compounds and also dietary fiber (Alasalvar et al., 2009; del Rio et al., 2011). Specifically identified polyphenolic components are known to be linked to several health effects in animals and humans, and the astounding antioxidant capacity of these skins makes them a very interesting and innovative ingredient to increase the daily antioxidant intake with natural ingredients. Hazelnut skins, which are usually considered a byproduct, are probably one of the richest edible sources of polyphenolic compounds (Dinkçi et al., 2021).

2. MATERIALS AND METHODS

2.1 Apparatus, materials and experimental procedure for Soxhlet extraction

A conventional Soxhlet extractor was adopted in the present study, which is equipped with a 250 W electric heater (by Falc), a 500 mL glass flask, a 200 mL extraction chamber, a 43x123 mm cellulose thimble and a 300 mm long Graham condenser for solvent vapor condensation.

The selected solvents were bi-distilled water, ethanol and R-limonene, these latter two being high-purity lab-grade liquids used as surrogates of bioethanol and limonene, which are nowadays made available as "green" solvents through suitable processing of natural renewable feedstocks. For comparison, n-hexane was used in a reference test.

The roasted hazelnut cuticles were provided in a dry state by the partner companies PRODAL and Grimaldi in this R&D project.

The Soxhlet extraction tests (see Table 1) were carried out on the "as collected" skins, having a typical moisture content of 7.25 and 9.20% wt. for the PRODAL and the Grimaldi feedstock, respectively. They were tested without any preparation, e.g., drying or sieving. Just in the case of the PRODAL feedstock, the 2-4 mm size fraction was used in a duplicated test (see #25 and #48 in Table 1), after gentle sieving of the original sample. In another case, the spent Grimaldi cuticles left after extraction with n-hexane and with R-limonene (see tests No.1 and 41 in Table 1, respectively) were subjected to a second extraction stage with ethanol (see tests No.4 and 42 in Table 1, respectively).

The effectiveness of the extraction has always been confirmed by the change in color of the extracted solution (see Table 1). Then, each extracted solution was stored in a glass bottle away from light.

The UV spectrophotometric analysis has been prelimi-

TABLE 1: Summary of the Soxhlet extraction tests with roasted hazelnut cuticles.

Soxhlet test ID	Sample	Sample mass (g)	Extraction solvent	Solvent volume (mL)	Number of cycles	Color of extract
1	Roasted cuticle Grimaldi "as collected"	20.0	n-hexane	300	13	Pale yellow
4	Roasted cuticle Grimaldi after extraction with n-hexane	14.3	ethanol	400	10	Brown
2	Roasted cuticle Grimaldi "as collected"	20.0	ethanol	300	10	Brown
5 (replica of #2)	Roasted cuticle Grimaldi "as collected"	20.0	ethanol	300	10	Brown
41	Roasted cuticle Grimaldi "as collected"	20.05	R-limonene	300	10	Pale yellow
42	Roasted cuticle Grimaldi after extraction with R-limonene	15.45	ethanol	300	10	Dark brown
43	Roasted cuticle Prodal "as collected"	20.0	ethanol	300	10	Dark brown
25	Roasted cuticle Prodal, 2-4 mm	14.3	water	300	10	Dark brown
48 (replica of #25)	Roasted cuticle Prodal, 2-4 mm	14.3	water	300	10	Dark brown

nary used for a qualitative assessment of the actual presence of bioactive compounds in the Soxhlet extracted solutions. To this end, a laboratory UV spectrophotometer has been used and at least one extracted solution has been tested for each reference sample of hazelnut residue. In particular, the presence of polyphenols was expected in the 200-300 nm wavelength window.

The method of Singleton and Rossi (1965) using Folin and Ciocalteu's phenol reagent was followed for the quantitative determination of total polyphenols as mg (gallic acid equivalent)/L. The method of Price et al. (1978) was adopted for the quantitative determination of tannins in Soxhlet extracts. In both cases the analytical determination was carried out in triplicate.

A Waters 1525 HPLC equipment with PDA 2996 detector, Waters symmetry C18e 5 µm column, 4.6 x 150 mm, located in the Chemical-Analytical Laboratory of Prodal Scarl, in Fisciano (SA), was used for the quantitative determination of the content of selected bio-active phenolic compounds.

2.2 Equipment, materials and experimental procedure for torrefaction

A laboratory scale fluidized bed reactor (38 mm ID, 350 mm height) was adopted for the torrefaction tests. More details about the experimental apparatus and procedures can be found in Brachi et al. (2019). The granular solid chosen as "inert" bed for the torrefaction tests was a fine quartz sand of nominal cut 250-125 µm, having a minimum fluidization velocity of 1.99 cm/s at room temperature (Brachi et al., 2019).

In a typical experimental run, the reactor was charged by a mass of 140 g of sand, which corresponds to a bed aspect ratio (i.e., the bed height to diameter ratio) of 2.1. Nitrogen was used as the fluidizing gas during torrefaction tests with a flow rate of 100 NL/h (corresponding to a gas superficial velocity of 2.5 cm/s at room temperature). This choice ensures a good mixing of the solids within the bed, while maintaining the fluidization in the "bubbling" regime and away from the "slugging" condition.

Torrefaction tests (see Table 3) were conducted in a batch mode with respect to cuticles with a biomass particle residence time t=5 min. The biomass feedstock was first the residual cuticles coming from the Soxhlet extraction of either Grimaldi or PRODAL roasted cuticle with the solvent ethanol (see tests T14 and T19 in Table 3), downstream of subsidiary operations of drying and sieving. In more details, the wet residues were conditioned down to a moisture content of about 6% wt., which represents the equilibrium value they achieved when left under a fume hood at room temperature for 2 days. After drying, the collected samples were sieved, and the 2-4 mm size fraction was retained for the subsequent torrefaction. Two further reference tests (see tests T15 and T11 in Table 3) were carried out with an "as collected" feedstock at the same temperature (i.e., 200°C), always with the same 2-4 mm sieve fraction.

The sample mass loaded into the bed of inert solids was prefixed to be far below the critical value of the biomass fraction (X_B = 4.18% wt.) beyond which the quality of fluidization and mixing of solids deteriorates (Brachi et al., 2017). Specifically, the adopted biomass-to-inert ratio was between 1 and 3% wt.

In order to investigate the effect of temperature on the torrefaction of the "as collected" feedstock, two other temperatures, i.e., T = 250°C and T = 300°C, were explored while keeping the same residence time (see Table 3).

After the completion of each test, the torrefied solids were accurately separated from the inert solids by gentle sieving, weighed and in some case further sieved for the determination of the particle size distribution. The initial and final weights of the samples allowed determining the mass yield MY on a dry basis (db) with the equation (Brachi et al., 2019):

$$MY\ (\%, db) = (mass\ of\ torrefied\ solids/mass\ of\ dry\ sample) \bullet 100 \quad (1)$$

The proximate and ultimate analyses of both the original and the torrefied samples allowed the calculation of the Lower Heating Value (LHV) of the torrefied solids thanks to the empirical correlation by (Channiwala and Parikh, 2002), hence determining the energy densification index IED and the energy yield EY with the equations (Brachi et al., 2019):

$$I_{ED}\ (-, db) = \frac{LHV_{torrefied\ solid}}{LHV_{raw\ feedstock}}|_{db} \quad (2)$$

$$EY\ (\%, db) = MY\ (\%, db) \bullet I_{ED}(-, db) \quad (3)$$

3. RESULTS AND DISCUSSION

3.1 UV spectrophotometric qualitative results

Representative results of the UV spectrophotometric analysis of Soxhlet liquid extracts are shown in Figure 1. They have been obtained by taking the absorbance profile of each pure solvent, respectively, as a "baseline" in the investigated wavelength range.

All of the Soxhlet extracted solutions display absorbance near and across 270 nm, which is the wavelength corresponding to polyphenols. The peak exhibited by the solution extracted by R-limonene in Figure 1B is likely attributed to the non-transparent color of the solution itself.

The absorbance profiles of solutions extracted by water (see Figure 1A) and ethanol (see Figure 1C-E) exhibit a similar shape, but the extension up to 600 nm and a slightly increased level of absorbance in Figure 1A seems to demonstrate a better extraction capability of water.

When comparing the absorbance profiles of solutions extracted by ethanol (see Figure 1C-E), no particular effect of the first extraction stage (i.e., tests #1 and #41 in Table 1) appears evident.

3.2 Soxhlet Extraction Tests

The results can be summarized as follows:

- No polyphenols or phenolic acids are extracted by a non-polar solvent like n-hexane and R-limonene (see tests #1 and #41 in Table 1), as documented by the results of the Folin and Ciocalteu's tests in Table 2.
- Vice versa, polyphenols and/or tannins are extracted by a polar solvent even on spent solids after extraction with a non-polar solvent. In fact, polyphenols and

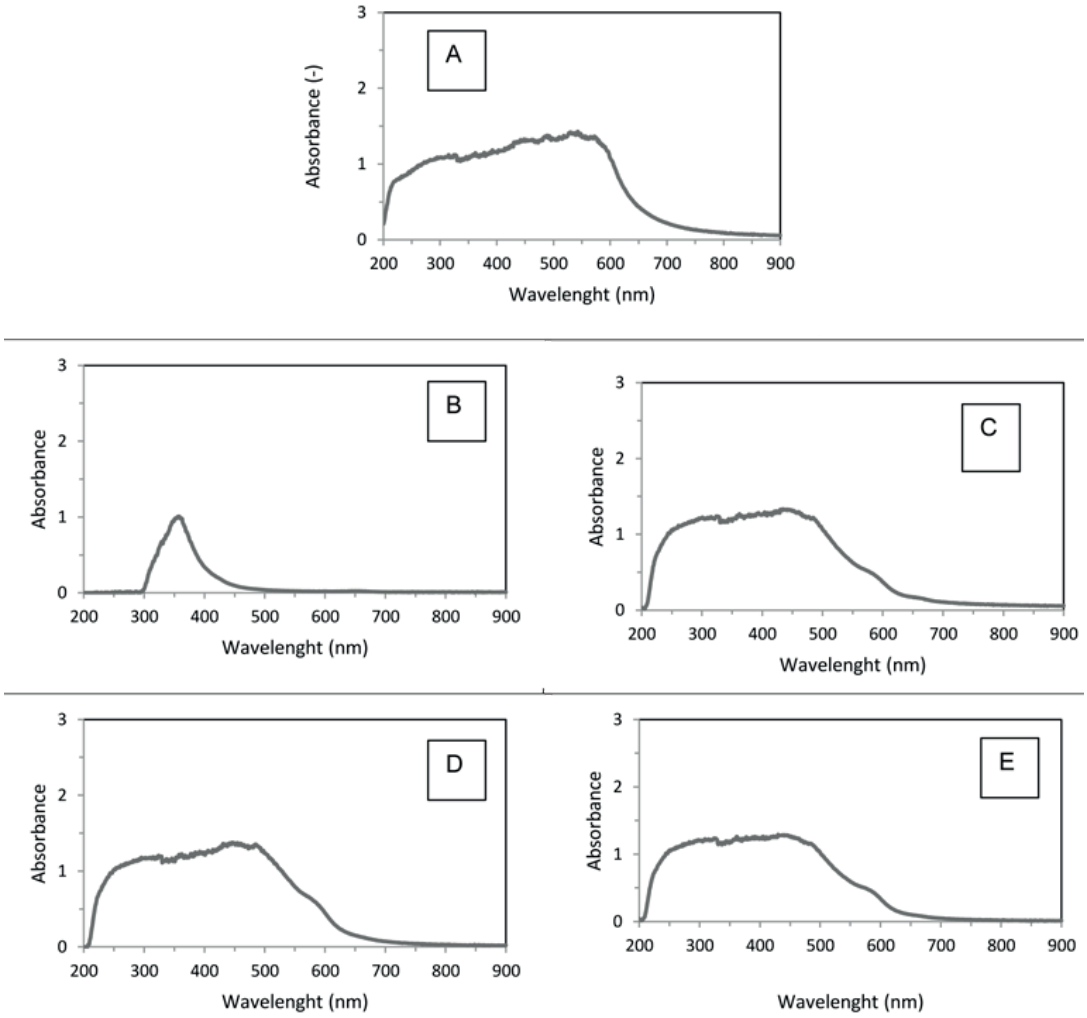

FIGURE 1: Representative UV spectrophotometric profiles. PRODAL roasted cuticles: A. test #25 extract in water. Grimaldi roasted cuticles: B. test #41 extract in R-limonene; C. test #2 extract in ethanol; D. test #42 extract in ethanol after R-limonene; E. test #4 extract in ethanol after n-hexane.

tannins are extracted by ethanol (see Table 2 with the test #4 on spent solids resulting from the test #1 with n-hexane), yielding a content of 2.01 g (gallic acid equivalent)/L and 16.85 g/L, respectively, in the extracted solution. Similarly, polyphenols are extracted by ethanol (see Table 2 with the test #42 on spent solids resulting from the test #41 with R-limonene), yielding a concentration of 2.19 g (gallic acid equivalent)/L in the extracted solution.

A "theoretical" yield from the investigated feedstock is proposed. It is calculated by the Eq. 4, respectively in total polyphenols and tannins:

$$Theoretical\ yield = \frac{volume\ of\ extracted\ solution \cdot measured\ concentration\ of\ the\ compound(s)\ of\ interest}{sample\ mass\ (Dry\ Solids)\ used\ in\ the\ Soxhlet\ test} \quad (4)$$

and the results are reported in Table 2.

- Based on a comparison on the same feedstock (i.e., the PRODAL roasted cuticle), water seems more effective than ethanol in the Soxhlet extraction of total polyphenols as water yields 0.070 g/g (DS) (see test #25 in Table 2), whereas ethanol extracts 0.032 g/g (DS) (see test #43 in Table 2). Actually, the test #25 was carried out with a 2-4 mm narrow cut solid sample, but such a difference is offset by the Soxhlet extraction technique.

Some bioactive phenolic compounds could be measured individually, whereas some others turned out below the minimum threshold of the HPLC apparatus. The available results are reported once more in terms of "theoretical" yield of the individual phenolic compounds as follows:

- With regard to catechin, a yield of 1.32, 0.62 and 0.51 mg/g (DS) was found for the test #2, #4 and #25 (Table 1), respectively;
- With regard to p-coumaric acid, similar values of yield, i.e., 0.02 and 0.03 mg/g (DS), were found for the test #4 and #25 (Table 1), respectively, whereas it was not detectable in the extract from the test #2;
- Finally, the gallic acid was detected in the extract of the test #25 only for which the calculated yield is 0.22 mg/g (DS); as expected, this value is by far lower than

TABLE 2: Key results of the Soxhlet extraction tests with roasted hazelnut cuticles.

Soxhlet test ID	Sample	Liquid-to-dry solids ratio (mL/g)	Average polyphenols g (GAeq)/L	RSD Polyphenols (%)	Tannins (g/L)	Average theoretical yield in total Polyphenols (g/g DS)	RSD Polyphenols yield (%)	Theoretical yield in Tannins (g/g DS)
1	Roasted cuticle Grimaldi "as collected"	16.52	ND	= = =	ND	= = =	= = =	= = =
4	Roasted cuticle Grimaldi after extraction with n-hexane	27.97	2.01	= = =	16.85	0.030	= = =	0.249
2-5 (average of duplicated tests)	Roasted cuticle Grimaldi "as collected"	16.52	2.20	6,41	7.10	0.031	6,47	0.100
41	Roasted cuticle Grimaldi "as collected"	16.48	ND	= = =	ND	===	= = =	===
42	Roasted cuticle Grimaldi after extraction with R-limonene	19.42	2.19	= = =	NV	0.025	= = =	NV
43	Roasted cuticle Prodal "as collected"	16.17	3.24	= = =	NV	0.032	= = =	NV
25-48 (average of duplicated tests)	Roasted cuticle Prodal, 2-4 mm	22.62	3,78	17,57	7.28	0.070	7,98	0.151

ND = Not Detected; NV = Value not evaluated; DS = Dry Solids

the theoretical yield in total polyphenols for the same test #25 in Table 2, which was expressed in terms of grams of gallic acid equivalent, but was actually comprising all polyphenols.

For an assessment of the second stage, in the case of ethanol-based extraction, the "theoretical" yield in total polyphenols from the same feedstock (i.e., the Grimaldi cuticle) is reported in Figure 2.

The addition of a second extraction stage, after the first one with a non-polar solvent, does not contribute any benefit as the polyphenol extraction yield decreases. This is further confirmed by the comparison of the theoretical yields in catechin between the tests #2 and #4 (Table 1): the yield in catechin for the test #2, i.e., 1.32 mg/g (DS), is larger than that obtained for the test #4, i.e., 0.62 mg/g (DS), which was carried out as a second stage after the first extraction with a non-polar solvent. This finding would imply a simplification in an actual process implementation that would not need any pre-extraction stage.

3.3 Torrefaction results

Table 3 reports the test conditions and the key results of torrefaction for the investigated feedstocks, i.e., the Grimaldi and PRODAL cuticles.

As an example of the visual changes introduced by torrefaction on the investigated feedstock, Figure 3 shows the torrefied particles (Test T15 in Table 3) in comparison with the initial raw material (Grimaldi cuticle).

A first analysis was directed at pinpointing a possible difference and, hopefully, an advantage when torrefying the

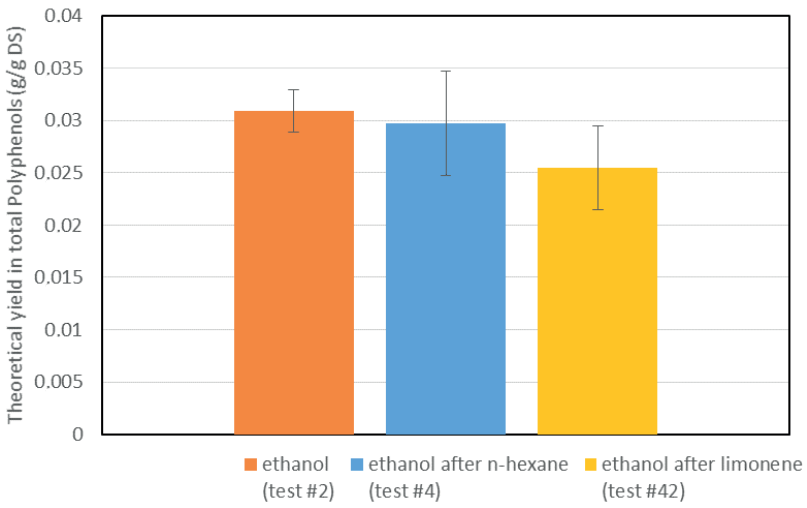

FIGURE 2: The effect on the theoretical yield in polyphenols induced by a non-polar solvent extraction stage preceding the ethanol extraction test.

TABLE 3: Summary of the torrefaction tests with roasted hazelnut cuticles.

Test ID	Sample	Sample particle size (mm)	Sample mass (g)	Bio-mass-to-inert ratio (%)	T (°C)	MY (%,db)	I_{ED} (-,db)	EY (%,db)
T14	Roasted cuticle Grimaldi after Soxhlet extraction with ethanol	2-4	1.5	1.07	200	66.81	0.93	62.20
T15	Roasted cuticle Grimaldi "as collected"	2-4	1.5	1.07	200	80.75	0.83	67.34
T16-T37 (average of duplicated tests)	Roasted cuticle Grimaldi "as collected"	2-4	4.3	3.07	250	52.50 RSD = 2.44%	0.83 RSD = 0.25%	43.68 RSD = 14.69%
T17	Roasted cuticle Grimaldi "as collected"	2-4	2.9	2.07	300	37.97	0.83	31.58
T19	Roasted cuticle PRODAL after Soxhlet extraction with ethanol	2-4	2.9	2.07	200	59.66	0.93	55.60
T11	Roasted cuticle PRODAL "as collected"	2-4	1.5	1.07	200	78.92	0.94	74.34
T21-T22-T38 (average of triplicated tests)	Roasted cuticle PRODAL "as collected"	2-4	4.3	3.07	250	62.68 RSD = 0.00%	1.07 RSD = 0.09%	67.08 RSD = 6.07%
T24	Roasted cuticle PRODAL "as collected"	2-4	4.3	3.07	300	50.15	1.04	51.98

cuticles coming as a solid residue after the solvent extraction. Actually, only the energy densification index IED seems to be benefited by an extraction step prior to torrefaction, as the increase in IED from 0.83 to 0.93 would indicate (see test T15 and T14 in Table 3) for the Grimaldi cuticle. However, the same increase in IED is not confirmed for the other investigated feedstock, that is the PRODAL cuticle (see 0.94 vs. 0.93 for the tests T11 and T19 in Table 3). On the other side, the mass yield MY and the energy yield EY decrease in solids that undergo torrefaction after a preceding extraction step (see test T15 as compared to T14, test T11 as compared to T19 in Table 3). This finding can be explained in terms of an "enhancing" effect that the removal of organic constituents from the solid matrix effected by the preceding extraction step (see the previous section) has on the pyrolytic reactions occurring in the subsequent torrefaction step.

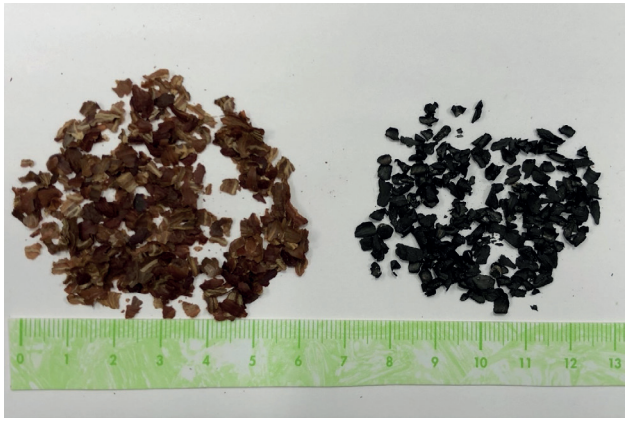

FIGURE 3: Picture of the roasted hazelnut cuticles (Grimaldi feedstock) and torrefied particles (test T15, at a temperature of 200°C and a residence time of 5 min).

It is worth noticing that the results in Table 3 confirm the well-known trends from literature (Brachi et al., 2019; Negi et al., 2020; Chen et al., 2021) by which both the mass yield MY and the energy yield EY decrease with the torrefaction temperature. All in all, the MY values in Table 3 are in line with the typical findings for non-wood biomass in literature (Brachi et al., 2019; Negi et al., 2020; Chen et al., 2021), whereas the EY values in Table 3 appear in the mid-to-bottom part of the typical range in literature (Brachi et al., 2019; Negi et al., 2020; Chen et al., 2021).

The set of torrefaction tests in Table 3 on the investigated feedstocks, i.e., the Grimaldi and PRODAL cuticles, allows a first quantitative analysis of the influence of temperature. Based on the set of results in Table 3, only a linear correlation analysis appeared reasonable. They are graphically reported for MY, IED and EY as a function of temperature in Figure 4 for the two feedstocks, more precisely in Figure 4A-C for Grimaldi cuticle and in Figure 4D-F for PRODAL cuticle.

Based on the set of actual results in Table 3, the goodness of fit is generally variable, e.g., the correlation coefficient R^2 is very high for MY (see Figure 4A and 4D), whereas it gets the lowest values for IED (see Figure 4B and 4E), becoming non-significant for the Grimaldi cuticle (see Figure 4B). All in all, these correlations represent a first tool for a quantitative description and a black-box modeling of the fluidized bed torrefaction process.

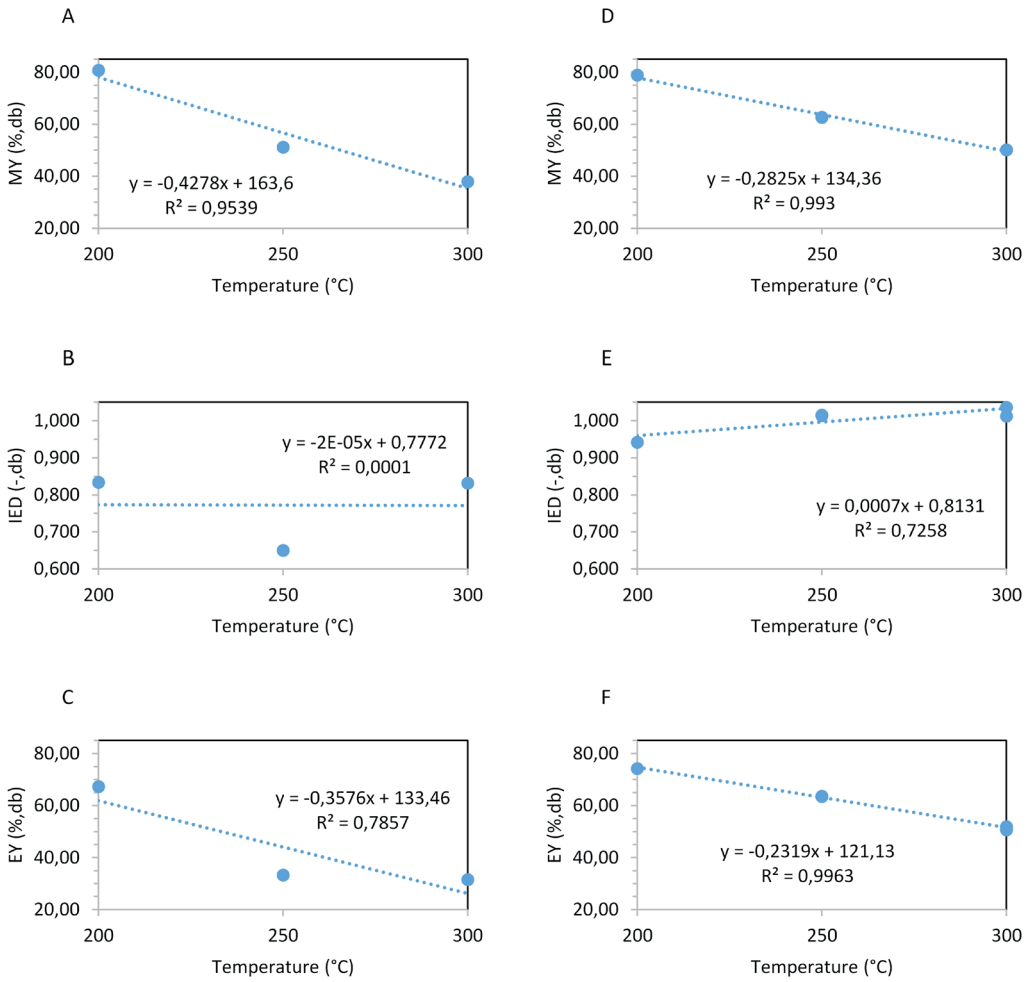

FIGURE 4: Torrefaction results. Simple correlations showing the influence of temperature at constant batch residence time (t=5 min): Grimaldi cuticle (A, B, C); PRODAL cuticle (D, E, F).

4. CONCLUSIONS

The findings of the present work can lead to the following conclusions:

- The Soxhlet technique shows that handy and "green" solvents like water and (bio)ethanol work well with residues of industrial hazelnut processing like roasted cuticles, and are leading to a thorough extraction of compounds of interest like polyphenols.
- The lab procedure and the analytical determinations enable the prediction of a theoretical yield of the compound(s) of interest with respect to the original feedstock (on a dry basis). This is something that will turn out useful in process design calculations aimed at an actual industrial implementation of extraction from hazelnut residues.
- The batch fluidized bed torrefaction of such a highly fragile material like roasted cuticles is feasible and works smoothly. However, such a feedstock is to be reduced from an original wide-cut size (e.g., 1 to 8 mm) to a more processable size cut (i.e., 2 to 4 mm) for operation of a binary mixture (i.e., biomass and sand) in a fluidized bed.
- A biomass-to-inert feed ratio up to 3% wt. is required to allow the proper mixing of cuticle particles in the torrefaction reactor as induced by the fluidization of a binary solid mixture.
- Based on the results of this work, simple linear correlations have been proposed for the dependence of the key performance indicators of torrefaction, i.e., the mass yield MY, the energy densification index IED and the energy yield EY, as a function of the temperature. They exhibit a variable goodness of fit, but they represent in any case a first tool for a quantitative description and future modeling of the fluidized bed torrefaction process.
- The present work lends itself to be a step along the route to a biorefinery implementation, in view of pursuing circular economy goals. This encompasses various processing steps, relying mainly on solvent extraction and torrefaction of solids, but considering all residues of the hazelnut value chain and additionally useful processing steps like pelletization. Actually, not only roasted cuticles, but also dry leafy husks are very light and difficult to handle solids; hence, they might be upgraded to a better-quality solid biofuel by means of

pelletization, either before or after torrefaction. Finally, the biorefinery might benefit of feedstock differentiation and integration (e.g., by opening to similar residues of other nut processing routes), and state-of-art habilitating technologies (like heat integration, energy efficiency, real time optimization, advanced process control).

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