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Change in some quality parameters and oxidative stability of olive oils with regard to ultrasound pretreatment, depitting and water addition

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ABSTRACT

Ultrasound pretreatment with different times (0, 4, 8, 10 min) on olive paste previous malaxation of olive oil extraction along with depitting and water application procedures were studied. The effects of these procedures on oil yield, quality parameters and oxidative stability have been studied. Therefore, the olive oil yield increase with increasing time of ultrasound treatment. On the other hand, the application of ultrasound did not adversely affect the quality characteristics, as well as the antioxidant activity, when comparing with untreated paste. Furthermore, the oxidative stability data, we can conclude that ultrasound treatment can affect the olive oil oxidative stability. This study could provide useful information for industry to produce olive oil with high yield and quality.

1. Introduction

Olive oil today represents one of the main part of Mediterranean dietary regimen, especially for fatty acids responsible for the assertive nutritional and health benefits [1]. In Turkey and Tunisia, olive oil market has always played an important role in agriculture and food industry with an annual production of 45,000 t.and 170,000 t, respectively [2].

Extra virgin olive oil is obtained from *Olea europaea* L. fruit using only physical procedures such as crushing of the fruit, kneading of olive paste, solid-liquid separation, and clarification by centrifugation and/or precipitation. The quality of olive oil depends on physical, chemical, and biochemical reactions which occur during the extraction process, its storage, and its bottling [3–5]. Kneading has several effects on the olive oil quality parameters, nutritional and sensorial characteristics. The controlling of kneading time which have great importance for the extraction yield and there are control limits over temperature and time for high quality olive oil. Inadequate management of malaxation leads to quality degradation and a loss of yield [6,7]. Therefore, a recent novel innovation has been introduced to olive oil extraction process in order to develop a more efficient malaxation step [7]. More recently [8,9], studied the effects of novel conditioning of olive paste using microwaves, megasound, and its combination at industrial scale on extra virgin olive oil quality and related chemical and sensory descriptors. This research team concluded that both microwave and megasonic technologies reduced olive paste consistency, not only to enhance oil yield performance but also to the International Olive Oil Council standard parameters which complied with the international specifications for extra olive oil definition for all tested novel conditioning interventions as well as the traditional control. On the other hand, Servili et al. [10] showed that there were a positive impact to the phenolic composition of EVOO but only when the system operated at highest pressure of 3.5 bar, confirming the importance of the regulation of process pressure to improve the performance of the ultrasound treatment in the breakdown of olive cells and the release of intracellular content.

It is possible to design complex technical tools and innovative equipment to increase extraction efficiency, shorten processing time and modulate the biological reaction in olive paste. Therefore, some new technologies such as depitting of fruits before kneading,

Abbreviations: TEAC, trolox equivalent.antioxidant capacity; DPPH, 2,2-diphenyl-1-picrylhydracyl; ABTS, 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid); FA, free acidity; PV, peroxide value; OD, optical density; UAE, ultrasound assisted extraction; US, ultrasound; EVOO, extra virgin olive oil.

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kneading in inert atmosphere and cold kneading may be applied. One of the recently used technologies is the ultrasound pretreatment of olive paste [11,12].

Ultrasounds can be described as the sound waves frequencies from 20 Hz up to 20.kHz that are not audible for human ear [13]. There are two groups of these ultrasonic waves: power ultrasounds (20 kHz and 1 MHz) and diagnostic ultrasounds (higher than 1 MHz). The propagation of high-power ultrasounds through an environment is possible with two basic effects, physical and chemical. Ultrasound has physical effects that can be defined as mechanical movements generated by high and low pressure cycles. Thanks to the mechanical and shear forces obtained, the mass transfer increases and the cell walls break down [14,15]. Relying on these perspectives, over the past few years, high frequency US (>1 MHz) was used in the olive oil extraction process. The results showed that the chemical and physical effects related to cavitation were minimal, while the effect of acoustic flow was predominant; high frequency separation was based on the principle of displacing suspended particles or droplets exposed to an ultrasonic standing wave field [8–10].

It is already known that ultrasound allows the olive paste to heat up quickly and improve the olive oil extractability without any change in the extra virgin olive oil quality parameters (free acidity value, peroxide value, K_{270}, and K_{232}) [12]. In addition, Clodoveo et al. [16], applied ultrasound to the olive paste before malaxation and to the olives before crushing. Likewise observed by Bejaoui et al. [12], they reported a quick heating of olive paste, improving the oil extractability and no negative effects on the olive oil quality, too.

As an industrial relevance, Leone and his co-workers have mentioned in two publications on 2017 and 2018, that it twas possible to introduce microwave and megasonic prototypes in an industrial line in order to overcome the batch nature of the malaxation process, thereby producing a continuous process. The combination of microwave and megasonic equipment into a modular unit could represent a new frontier for olive paste conditioning in olive oil extraction plants [8,9]. More recently, Servili et al. [10] have studied the effect of ultrasound technology to test its action on the extraction of olive oil at the industrial scale. They have found that when ultrasound was subjected to olive paste with a pressure of about 3.5 bar, there was a significant increase of extractability compared to the traditional process. On the other hand, they have concluded that there was no significant effect between ultrasound treatment and traditional technology on extractability when ultrasound at a pressure level of 1.7 bar was used.

Therefore, the purpose of this research was to investigate the ultrasound application to olive paste, previous to malaxation, at laboratory scale and its effect on oil extraction process yield and some olive oil quality parameters.

2. Materials and methods

2.1. Olive fruit samples and maturity index

In this work, olives from the variety *Chemlali* (Sfax, Tunisia) and *Memecik* (were Aydın, Turkey) studied. The olives were harvested during the crop season 2015-2016. The maturity index was determined according to the method developed by the Agronomic Station of Jaén [17] as a function of fruit color (epicarp and mesocarp). For this study, a maturity index, equal to 6, was determined on 100 randomly selected olives trees. After harvesting, the olives were transported on the same day to the laboratory.

2.2. Oil extraction system

After the removal of leaves the olives were washed. The olives of each cultivar were divided into twelve equal batches of 2.5 kg. The method described previously by Yahyaoui et al. [18] was used for the simulation of industrial scale extra virgin olive oil production. Therefore, a hammer crusher (a stainless-steel hammer mill operating at 3000 rpm provided with a 5 mm sieve), a malaxer (Hobart Corporation, USA) and a centrifuge (Centrifuge MF 20, Awel Industries, France) were used. The ultrasound treatment was applied on olive paste after crushing at different times (0, 4, 8, and 10 min) using an Elmasonic S60H ultrasonic bath (Elma Hans Schmldbauer Gmbh & Co., Singen, Germany) with the following characteristics: frequency, 35 kHz; tank volume, 4.25 L; tank internal dimensions, W × D × H 240 × 137 × 150 mm; ultrasonic power effective, 150 W. After the ultrasound treatment, the olive paste of each batch was malaxed for 15 min using a stainless-steel container (4.5 L) equipped with a helical blade immersed in a water bath at 35 °C (rotational speed, 24 rpm). Olive oil was filled in dark coloured glass bottles without headspace filled under nitrogen flux. The samples were kept in the refrigerator at +4 °C. Experiments were performed in triplicate. In addition, the ultrasound energy (E) was calculated according to the equation E = Wt/m (1), where, W is microwave oven power, t is the time of ultrasound exposure, and m = quantity of sample [19]. Furthermore, the energy given to olive paste for the studied samples were: 14.4 kJ/kg, 28.8 kJ/kg and 36 kJ/kg for 4 min, 8 min and 10 min of ultrasound treatments, respectively.

The oil extraction yield (%, w/w) was calculated as follows:

$$\text{Olive oil extraction yield, } \% = \frac{\text{Mass of extracted oil (g)}}{\text{Mass of olive paste (g)}} \times 100 \qquad (2)$$

2.3. Quality indices

Free acidity (% oleic acid) and peroxide values (meq O_2/kg VOO) were determined by using the methods described in European Regulation EEC 2568/91 [20].

2.4. Extraction of phenolic fraction from olive oil

The phenol extracts were obtained as described before by Rigane et al. [2]. Briefly, 2 mL of *n*-hexane were added to 4 g of oil and 4 mL of a methanol-water (80:20; v/v). After, the mixture was centrifuged for 3 min. The hydroalcoholic phase was then collected and the hexanic phase was extracted twice more with 4 mL of methanol-water (80:20; v/v) solution. Finally, the hydroalcoholic fractions were gathered together, washed with 4 mL of *n*-hexane to get rid of the residual oil, and then dried by evaporative centrifugation under vacuum at 35 °C.

2.5. Antioxidant activity

1. DPPH radical scavenging activity. The radical scavenging effect was tested by measuring of oil methanolic extracts towards the 2,2-diphenyl-1-picrylhydracyl (DPPH$^{\bullet}$) [21]. Radical scavenging activity, expressed as percent inhibition, was calculated by the following formula:

Percent radical scavenging activity = (control OD - sample OD/control OD) × 100

2. Trolox equivalent antioxidant capacity (TEAC) assay. The method previously described by Rigane et al. [22] was used which was based on measuring the reduction of the ABTS radical cation by antioxidants. The Trolox equivalent antioxidant capacity (TEAC) of the sample was calculated as inhibition percentage of ABTS$^{\bullet+}$. The results were calculated using a Trolox standard curve prepared with five concentrations (1.5–30 μmol/l). The results were given as mmol of Trolox equivalents.

2.6. Oxidative stability test

Oxidative stability was evaluated by the Rancimat apparatus, model 743 (Metrohm Co., Herisau, Switzerland). Rancimat vessels containing 2.5 g of oil sample were covered with the heads, placed into the Rancimat apparatus at room temperature, and then heated under an air flow rate of 20 L/h. The instrument was run at 110 °C. The time (h) taken until there is a sharp increase of conductivity is called the induction time (IT), and it is expressed in hours. All determinations were carried out in triplicate [23].

2.7. Statistical analysis

Results of the 3 measurements were given as mean ± standard deviation (SD). One-way analysis of variance (ANOVA) was used to calculate the statistical differences with the help of Student's t-test. Differences were considered significant at $p < 0.05$.

3. Results and discussion

3.1. Effect of ultrasound treatment on oil yield

It is well known that the one of the economical parameters that growers give most importance is the oil extraction yield. In order to improve this parameter, it is possible to encounter ultrasound applications for various treatment times. From Table 1, we show a high positive correlation between ultrasound treatment time and oil yield for *Chemlali* cultivar obtained without pits ($r^2 = 0.814$ and $r^2 = 0.890$, using water or not, respectively), furthermore, and for the *Memecik* cultivar, the highest correlation between these two parameters were obtained pits ($r^2 = 0.749$ and $r^2 = 0.729$ using water or not, respectively). Ultrasound treatment of olive paste at 35 kHz ended up with the highest oil yields after 10 min of treatment for both studied varieties. It is worth noting that *Chemlali* depitted olive paste have the highest oil yield (20.40 and 20.53 %, respectively, with and without water addition), while the highest value (Table 1), for *Memecik* cv., were obtained when whole olive paste were treated with ultrasound (~18 % and 19.55 %, respectively, with water and not). It should be noted here, the percentage yield values were calculated based on the actual mass in the beginning of the extraction; meanly the "weight of olive paste with pits" for the yield of whole olive paste and; the "weight of olive paste without pits" for the yield of depitted olives were used. The yield values for depitted olive extraction were higher as expected before, because the amount of oil increased proportionally to the weight pits removed.

These results were in accordance with those reported previously in review by Koubaa et al. [24] and scientific results by Koubaa et al. [25] as well as Rosello-Soto et al. [26]. These authors mentioned that the application of UAE has many advantages including reducing the amount of solvent used for extraction, which has more economic and environmental benefits and characteristics, higher extraction efficiency of the targeted molecule, reduced extraction time and energy consumption and higher process throughput. A recent study [8] has evaluated as industrial scale the above-mentioned technologies, separately and in combination: (i) the conditioning of the olive paste with microwave without malaxation, (ii) the introduction of a megasound treatment post-malaxation step in the traditional process, and (iii) the combined treatment of non-malaxed pastes with microwave followed by megasound. This study demonstrated a new technological opportunity for the development of a continuous olive oil process that consists of microwave + megasound conditioning, which provided additional olive oil yield.

On the other hand, our research team studied the effect of water addition as a co-adjuvant on the oil yield. Results show that water addition caused a slight but significant increase on the yield for both the Tunisian and Turkish olive cultivars. These results were in accordance with those reported by Bejaoui and co-workers in 2017 who mentioned that a series of fast compressions and expansions in the medium created by the propagation of ultrasonic wave in olive paste increases oil yield observed for high power ultrasound treatment without malaxation. This movement may resemble a sponge that is repeatedly squeezed and released. This is called the

Table 1
Some quality parameters and oxidative stability of olive oil (*cv.* Memecik) with regard to different ultrasound pretreatments.

		with pits		*without pits*	
		with water	without water	with water	without water
Yield (%)	M	15.86 ± 1.32b	12.01 ± 1.36b	18.18 ± 1.27a	16.63 ± 1.53
	M + 4 m US	16.08 ± 1.48b	18.49 ± 1.12a	17.77 ± 1.59b	16.27 ± 1.50
	M + 8 m US	18.80 ± 1.14a	17.62 ± 1.67a	17.60 ± 1.02b	16.91 ± 1.52
	M + 10 m US	17.99 ± 1.25ab	19.55 ± 1.49a	16.96 ± 1.64b	16.94 ± 1.37
Free acidity (% oleic acid)	M	2.25 ± 0.072a	1.70 ± 0.069a	2.60 ± 0.038a	1.85 ± 0.045a
	M + 4 m US	2.07 ± 0.082a	1.46 ± 0.106b	1.38 ± 0.42c	1.73 ± 0.121ab
	M + 8 m US	1.50 ± 0.079b	1.32 ± 0.070b	1.19 ± 0.20c	1.31 ± 1.52b
	M + 10 m US	1.42 ± 0.057c	1.44 ± 0.127b	2.10 ± 0.275b	1.50 ± 0.049bc
Peroxide value meq O2/kg oil	M	1.84 ± 0.20 b	2.13 ± 0.25a	4.32 ± 0.20a	4.14 ± 0.086a
	M + 4 m US	1.93 ± 0.20 ab	2.36 ± 0.25b	4.35 ± 0.20a	3.57 ± 0.16b
	M + 8 m US	2.16 ± 0.18 a	2.45 ± 0.20 c	4.08 ± 0.22a	3.78 ± 0.20b
	M + 10 m US	2.20 ± 0.20 a	2.43 ± 0.30 d	3.50 ± 0.06b	3.96 ± 0.16 ab
DPPH (% inhibition)	M	59.44 ± 0.29c	80.78 ± 1.93a	58.74 ± 2.16b	68.78 ± 1.37c
	M + 4 m US	80.82 ± 0.61a	81.59 ± 3.11a	56.85 ± 1.51b	77.63 ± 1.43a
	M + 8 m US	61.18 ± 1.25c	75.56 ± 1.06b	62.45 ± 1.56a	80.45 ± 1.60b
	M + 10 m US	68.00 ± 2.60b	71.41 ± 2.06b	66.85 ± 2.45a	84.93 ± 3.83a
TEAC (mmol TE/kg extract)	M	0.38 ± 0.075b	0.53 ± 0.049a	0.37 ± 0.047b	0.41 ± 0.031
	M + 4 m US	0.49 ± 0.005a	0.40 ± 0.060b	0.43 ± 0.003a	0.45 ± 0.064
	M + 8 m US	0.52 ± 0.005a	0.41 ± 0.008b	0.42 ± 0.011a	0.45 ± 0.012
	M + 10 m US	0.53 ± 0.007a	0.42 ± 0.029b	0.45 ± 0.042a	0.45 ± 0.054
Induction time (h)	M	11.32 ± 0.20b	13.14 ± 0.58c	10.44 ± 0.20c	10.71 ± 0.63c
	M + 4 m US	13.35 ± 0.20a	15.57 ± 0.16b	11.93 ± 0.20b	12.63 ± 0.25b
	M + 8 m US	13.42 ± 0.37a	15.14 ± 0.37b	12.15 ± 0.43ab	13.71 ± 0.80a
	M + 10 m US	13.50 ± 0.86a	16.96 ± 0.16a	12.71 ± 0.20a	13.26 ± 0.30ab

[†]Mean value ± standard deviation.
[‡]Differences.in the same row shown by different lower case letters are significant ($p \leq 0.05$).

“sponge effect” [27]. While this phenomenon facilitates the flow of intracellular fluid outward, it may form micro channels that aid the movement of fluid within a medium as a result of compression and expansion of the medium.

3.2. Effect of ultrasound treatment on FA and PV parameters

According to the results showed in Tables 1 and 2, our research team found a significant decrease in all FA values for all the studied olive oils. These values were more than 0.8 % according to EEC [28]. This high values of acidity could be explained by triglycerides hydrolyses reactions [29]. In general, for FA parameter *Chemlali* oils showed the lowest values if compared with *Memecik oils*.

Because of the cavitation occurs during ultrasound application, a special interest was focused on peroxide values (PV) which allows us to make an estimation of oxygen quantity necessary for the degradation of olive oils. In general, the oil PV parameter analysed showed significant differences during ultrasound treatment time (Tables 1 and 2). Furthermore, the peroxide values behaviour of virgin olive oils could be explained by changes during oxidation process, by the formation of hydroperoxides while decrease in values were due to the appearance of secondary products. These results were lower than that found by Leone et al. [9] who used single microwave and megasonic treatments, or their combinations in an industrial olive oil extraction plant.

These results were not in accordance with those reported previously by Jimenez et al. [11] and Clodoveo et al. [16] who mentioned that no significant differences were found attributable to the ultrasonic treatment for these chemical parameters.

3.3. Antioxidant activity

Concerning the antioxidant profile of the two oil varieties considered herein, the results are summarized in Tables 1 and 2. Two different antioxidant tests (DPPH and ABTS radicals) have been conducted in order to evaluate the free radical scavenging capability of the obtained olive oil extracted after ultrasound treatment. In general, the antioxidant activity of the studied olive oils showed important differences between treatments, even if no differences were observed. For both studied olive oil cultivars (Tunisian and Turkish), the antioxidant activity of olive oil obtained from olive paste without pits reached its maximum after 10 min of ultrasounds pretreatment. On the other hand, when *Chemlali* and *Memecik* olive pastes with pits were treated with ultrasound at 35 kHz, we can note that the inhibition of DPPH radicals showed its maximum after treatment during 4 min (Tables 1 and 2). In addition, inhibition percentage of $ABTS^{•+}$ was not affected by ultrasound pretreatment only a significant increase on TEAC activity for *Chemlali* olive oil obtained from whole olive fruit without addition water. These results could be explained by the antioxidant mechanisms and actions regarding DPPH and ABTS radicals.

3.4. Oxidative stability

The oxidative stability of olive oil obtained in the different experiments was summarized in Tables 1 and 2. From these data, we can conclude that ultrasound treatment can affect the olive oil oxidative stability. The increased ultrasound treatment time had a greater impact on induction time for the oils of depitted olives of both cultivars. The increase on the induction time could be explained by the obtained antioxidant activities (DPPH and ABTS tests) which attempt its maximum activities after 10 min of treatment. From Table 2, the induction time of *Chemlali* olive oil obtained from whole olive fruits reached its maximum after 4 min of ultrasound pretreatment. Therefore, a linear relationship (data not shown) exists between the antioxidant activities and the oxidative stability of the two olive oil samples.

Compared to European oils [30,31], our samples seem to have low stability values (<70 h). Our results were not in accordance with data found by Bejaoui et al. [30] and Mateos et al. [31] who confirmed that ultrasound treatment did not affect olive oil

Table 2
Some quality parameters and oxidative stability of olive oil (*cv.* Chemlali) with regard to different ultrasound pretreatments.

		with pits		*without pits*	
		with water	without water	with water	without water
Yield (%)	M	15.45 ± 0.40 b*	16.89 ± 0.52 ab	11.36 ± 0.60c	14.65 ± 0.70b
	M + 4 m US	16.37 ± 0.1 ab	16.50 ± 0.46 b	18.29 ± 0.36 b b	14.96 ± 0.33b
	M + 8 m US	16.10 ± 0.76 b	16.38 ± 0.43 b	18.11 ± 0.11b	19.40 ± 0.71a
	M + 10 m US	16.99 ± 0.15 a	17.61 ± 0.02 a	20.40 ± 0.66a	20.53 ± 0.70a
Free acidity (% oleic acid)	M	1.83 ± 0.13 a	1.81 ± 0.01 b	2.21 ± 0.03a	1.74 ± 0.04a
	M + 4 m US	1.81 ± 0.17a	1.31 ± 0.03 c	2.07 ± 0.09a	1.44 ± 0.04bc
	M + 8 m US	1.37 ± 0.06b	1.72 ± 0.23b	1.41 ± 0.08b	1.32 ± 0.07c
	M + 10 m US	1.75 ± 0.00 a	2.14 ± 0.12a	1.42 ± 0.00b	1.51 ± 0.02b
Peroxide value meq O2/kg oil	M	5.28 ± 0.05 a	5.65 ± 0.09 a	2.43 ± 0.02 d	3.05 ± 0.07 c
	M + 4 m US	5.36 ± 0.05 a	4.63 ± 0.10 d	2.81 ± 0.06 c	3.13 ± 0.03 c
	M + 8 m US	4.95 ± 0.08 b	4.82 ± 0.05 c	3.12 ± 0.05 b	3.62 ± 0.16 b
	M + 10 m US	3.51 ± 0.01 c	5.04 ± 0.06 b	3.23 ± 0.04 a	3.90 ± 0.08 a
DPPH (% inhibition)	M	61.30 ± 0.79a c	78.32 ± 0.27a b	65.45 ± 0.19a d	58.74 ± 1.10 c
	M + 4 m US	72.19 ± 0.17b a	80.04 ± 0.07a a	77.21 ± 0.59b c	50.99 ± 0.57 d
	M + 8 m US	59.97 ± 0.50c d	74.26 ± 0.60b c	79.99 ± 0.49c b	61.34 ± 0.54 b
	M + 10 m US	63.01 ± 0.55a b	69.63 ± 0.57c d	85.52 ± 1.03d a	64.99 ± 0.03 a
TEAC (mmol TE/kg extract)	M	0.54 ± 0.01a aa 0	0.39 ± 0.010a	0.12 ± 0.026a ab	0.40 ± 0.006bc
	M + 4 m US	0.28 ± 0.026b b	0.62 ± 0.020b c	0.11 ± 0.026a b	0.36 ± 0.067 c
	M + 8 m US	0.51 ± 0.010a a	0.66 ± 0.015c b	0.16 ± 0.026b b	0.49 ± 0.030 a
	M + 10 m US	0.53 ± 0.023a a	0.78 ± 0.026d a	0.35 ± 0.038c a	0.46 ± 0.011 ab
Induction time (h)	M	11.32 ± 0.20b	14.17 ± 0.58b	10.53 ± 0.20b	9.31 ± 0.63d
	M + 4 m US	12.89 ± 0.20a	15.23 ± 0.16a	11.34 ± 0.20a	10.55 ± 0.25c
	M + 8 m US	10.37 ± 0.36 b	14.69 ± 0.37ab	11.68 ± 0.43a	12.08 ± 0.80b
	M + 10 m US	11.15 ± 0.86 b	12.35 ± 0.16 c	11.26 ± 0.20a	14.21 ± 0.30a

†Mean value ± standard deviation.
‡Differences.in the same row shown by different lower case letters is significant ($p \leq 0.05$).

stability. The induction time have been proposed as applicable to the characterization of olive oils, since the minor compounds differs from one variety to another.

4. Conclusions

In this paper, the use of new technologies such as ultrasound to increase oil extraction efficiency as well as some quality parameters is described. Regarding to the virgin olive oil quality no deterioration or alteration on its antioxidant state were observed with the ultrasound treatments (35 kHz) and for the three different time tested (0, 4, 8, 10 min), on the other hand, treatment with ultrasound could ameliorate the oxidative stability of the obtained oil. Furthermore, further studies can be conducted to assess the effects of ultrasound technology on industrial extra virgin olive oil extraction.

Data statement

All Authors state the availability of our data in our submission.

Author contributions

Ghayth Rigane: done practical experiences, write, follow and check the obtained results.

Amira Yahyaoui and Ayşenur Acar: done some practical experiences.

Sami Mnif: done ultrasound treatment.

Ridha Ben Salem and Derya Arslan: supervised the scientific paper. (As the director of the project I collected the samples, coordinated all the analysis, calculated the results, statistics etc . . .).

Declaration of Competing Interest

The authors declare that there are no conflicts of interest.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.btre.2020.e00442.

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