Enrichment of fresh pasta with antioxidant extracts obtained from by-products of the artichoke canning by using ultrasound-assisted technology

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Running title: Pasta containing artichoke by-product extracts

Summary

This work is aimed at: (i) analyzing the extracts obtained from canning by-products of three artichoke cultivars (Opal, Capriccio and Catanese); (ii) comparing color, textural properties, and cooking performance of fresh pasta enriched of the most antioxidant extract, with control pasta. UHPLC-ESI-MS/MS profile of concentrated Catanese cv. extracts showed the highest antioxidant activity (1662 µmol Trolox equivalents L^{-1}) and the highest levels of luteolin-7-*O*-rutinoside (55.9 mg L^{-1}), luteolin-7-*O*-glucoside (14.2 mg L^{-1}), and apigenin-7-*O*-rutinoside (4.7 mg L^{-1}), compared to the other cultivars. Fresh pasta enriched of Catanese cv. extract showed significantly higher (p<0.05) total phenolic compounds and antioxidant activity (500 mg gallic acid kg^{-1} and 1324 µmol Trolox kg^{-1}, respectively) than control pasta (306 mg gallic acid kg^{-1} and 886 µmol Trolox kg^{-1}, respectively). The extract significantly (p<0.05) increased pasta brownness (from 19.93 to 23.34), and decreased yellowness (from 27.11 to 23.09), but did not alter textural parameters and cooking performance.

Key words: pasta, functional foods, phenols, texture, antioxidant activity

Introduction

About 13.5 million tons of pasta are yearly produced worldwide. Italy produces 3.32 million tons and is the country with the highest per capita consumption (26 kg per year) although United States, with 2.7 million tons per year, rank first in world pasta consumption (International Pasta Organization, 2013). According to moisture content, 12.5% at most or 24% at least, pasta is classified in “dry” and “fresh”, respectively (Italian Republic, 2001). Fresh pasta, made of durum wheat semolina, common wheat flour, or mixtures of them (Italian Republic, 2001), is produced both at industrial and artisan level. In the latter case, fresh pasta is hand-shaped to give traditional niche products (Alexander, 2000). Valued for the nutritional features and environmentally sustainable, both dry and fresh pasta are “mature” products, that in recent years have been innovated with the release of new “functionalized” types, to meet the increasing expectative for healthier foods (Pasqualone et al., 2015; 2016).

Globe artichoke is a good dietary source of phenolic compounds with high bioavailability, in particular hydroxycinnamates and flavonoids (Shutz et al., 2004). Due to its hepatoprotective, choleretic, hypocholesterolemic, and antioxidant properties (Curadi et al., 2005), globe artichoke is considered a nutraceutical food and is exploited for the extraction of health-promoting substances. The leaves contain high levels of total phenolics and are widely used in phytopharmaceutical applications. Artichoke leaf extracts are marketed for the treatment of liver diseases and show hypocholesterolemic activity due to two parallel mechanisms: reduction of cholesterol biosynthesis and inhibition of LDL oxidation (Bundy et al., 2008).

Industrial by-products, such as outer bracts, leaves, and stems, are the residue of artichoke heart canning and represent about the 80% of the biomass (Ceccarelli et al., 2010). The processing yields recorded for the most cultivated European varieties are largely lower than those gathered from the best hybrids (Lahoz et al., 2004). Some cultivated varietal types, especially Catanese, show particularly high waste to total head ratio, with processing yield as

low as 300 g kg^{-1} raw head (Bonasia et al., 2010). The discarded outer bracts, leaves, and stems are valuable sources for the production of functional extracts, potentially suitable to be incorporated into pasta.

The application of ultrasound-based technologies for assisting the extraction of nutraceuticals from an array of plant and animal sources has been recently reviewed (Chemat and Khan, 2011). Advantages include reduction of solvent usage, decrease of extraction time, and yield increase. Ultrasound can enhance the extraction of valuable functional components such as phenolics, anthocyanins, and aromatic compounds (Chemat and Khan, 2011). Recent papers have evaluated the application of ultrasound technology to the extraction of phenolic compounds from artichoke (Punzi et al., 2014; Rabelo et al., 2016). However, there are no reports about the actual use of these extracts as food additives and their effect on quality of end-product.

The aim of this study has been to explore the feasibility of employing artichoke by-product extracts obtained by ultrasound-assisted technologies in the production of potentially functional fresh pasta. The research was developed in two steps: 1) production and characterization of the extracts obtained from three artichoke cultivars (Opal, Capriccio and Catanese); 2) production and characterization of fresh pasta enriched of the best extract, and comparison with control pasta in terms of color, textural properties, and cooking performance. A traditional hand-made semolina-based fresh pasta type was chosen for the trials.

Materials and methods

Materials

Opal and Capriccio recently released F1 hybrids, and Catanese cultivated varietal type of globe artichokes [*Cynara cardunculus* L. subsp. *scolymus* (L.) Hayek], were harvested in Mola di Bari

(Italy). The flower heads were collected with part of stem and leaves. Artichoke scraps consisting of outer bracts, leaves, and stems, representing the by-products of artichoke canning, were manually separated from heads according to the usual industrial procedure. Durum wheat semolina (12.7 g 100 g^{-1} moisture, 0.83 g 100 g^{-1} ash, 11.8 g 100 g^{-1} dry gluten, 88 gluten index) was purchased at a local retailer.

Ultrasound-assisted extraction of phenolic compounds from artichoke scraps

Artichoke scraps were immediately used, fresh, for subsequent extractions of phenolic compounds. At this purpose, the scraps were manually shredded with a knife to about 3-mm size pieces and carefully mixed up. An aliquot of 150 g was added of 450 mL ethanol:water (30:70, v/v) into a 1-L polyethylene terephthalate bottle, and sonicated for 45 min in ultrasonic bath (CEIA S.p.A., model CP 104 Digit, Arezzo, Italy), at room temperature, 40 kHz frequency, 240 W power, and 0.5 W cm^{-2} ultrasound intensity. Time and temperature of ultrasound treatment were set according to a previous paper (Punzi et al., 2014) and verified by preliminary trials as those able to recover higher levels of phenolic substances. The bottles were manually shaken every 5 min in order to allow a homogeneous sonication. Then, the samples were filtered on n. 1 Whatman paper and centrifuged at 9000 *g* for 10 min to recover a clear solution (US extract). Extraction trials were also performed in the same conditions but without ultrasound treatment (NO-US extract), for verifying the efficacy of US treatments. In order to remove ethanol for subsequent pasta-making trials, the US extract was then concentrated up to 40% of the initial volume (C-US extract) by means of R-300 Rotavapor (Büchi, Flawil, Switzerland), at 30 °C.

Fresh pasta-making procedure

A small-scale standardized laboratory procedure was used for the production of fresh pasta. According to farinograph water absorption of durum wheat semolina used, either 55 mL of tap water or the same amount of C-US extract of Catanese cultivar were added to 100 g of semolina to obtain control and artichoke pasta, respectively. After 20 min of manual kneading, the dough was left to rest 15 min, then was hand-shaped into a traditional short-cut pasta type produced in Apulia region (Southern Italy), called '*orecchiette*' (literally "little ears"). This kind of pasta, described by Alexander (2000) and by Delvecchio and Pasqualone (2013), has a concave circular shape, diameter of 2.0-2.5 cm, and thickness of 2-3 mm. The concavity is smooth and the convex side is rough (Fig. 1). *Orecchiette* were obtained by preparing cylindrical dough strips of 1 cm diameter, followed by cutting the strips into 1-cm long pieces and stretching each piece using a special knife with rounded edge. After shaping, the single pasta pieces were put on a wooden vessel, covered with a cotton cloth and left to dry 3 h at room temperature. Values of 26±1 g 100 g^{-1} moisture and 0.93±0.01 a_w were reached, accomplishing the legal requirements for fresh pasta (Italian Republic, 2001).

Determination of total phenolic compounds (TPC)

TPC were determined in NO-US, US, and C-US extracts and in pasta as follows. Control and artichoke pasta samples were finely ground under liquid nitrogen, then 10 g of sample were added of 25 mL ethanol:water (70:30, v/v). The suspension was stirred for 1 h in the dark at room temperature, then centrifuged at 9000 *g* for 5 min. The supernatant was recovered, whereas the solid residue was submitted to a second extraction in the same conditions. The obtained pasta extracts, as well as NO-US, US, and C-US extracts were then filtered on 0.22 µm regenerated cellulose filters (LLG Labware, Meckenheim, Germany), before being submitted to Folin-Ciocalteu reaction as in Gambacorta et al. (2012). Briefly, in a test tube, 100 µL of extract were added of 500 µL water, 100 µL Folin-Ciocalteu reagent (Carlo Erba, Milano, Italy) and,

after 5 min, 500 µL of 10% (w/v) Na_2CO_3. The absorbance was measured at 700 nm after 90 min, by means of a Beckman Coulter DU 800 spectrophotometer (Beckman Instruments Inc., Fullerton, CA, USA). Gallic acid standard solutions at a concentration ranging from 0.08 to 0.002 mg mL^{-1} (R^2 = 0.9989) were used for the calibration. The results were expressed as gallic acid equivalents.

Determination of *in vitro* antioxidant activity (AA)

The extracts used to determine the total phenolic compounds were analyzed also for their *in vitro* antioxidant activity by the assay based on 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid) (ABTS) (Sigma-Aldrich Co., St. Louis, USA), in the conditions reported by Ferrara et al. (2014). Briefly, to produce $ABTS^{\bullet+}$, 7 mmol L^{-1} ABTS solution were allowed to react with 2.45 mmol L^{-1} potassium persulfate aqueous solution for 16 h in the dark at room temperature. The solution containing $ABTS^{\bullet+}$ was diluted with water to obtain an absorbance of 0.80 ± 0.1 at 734 nm. One hundred µL of extract were added to 3.9 mL of diluted $ABTS^{\bullet+}$ solution, and absorbance was measured after 5 min. Solutions of 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) (Sigma-Aldrich Co., St. Louis, USA) were prepared at a concentration ranging from 20 to 1000 µM (R^2 = 0.9965) for the calibration. The results were expressed as Trolox equivalents.

UHPLC-ESI-MS/MS determination of phenolic profile

The ultra-high performance liquid chromatography electrospray ionization tandem mass spectrometry (UHPLC-ESI-MS/MS) analysis of phenolic profile of the same extracts used to determine the TPC and AA was performed by using the UHPLC Dionex Ultimate 3000 system (LPG-3400 RS quaternary pump, WPS-3000 TRS autosampler, and TCC-3000 RS column oven), coupled with the HESI-II probe and the LTQ Velos Pro ion trap mass spectrometer (Thermo

159 Fischer Scientific, Waltham, MA, USA). The separation of phenolic compounds was performed
160 by Luna C18 column (150 × 3.0 mm id, 3 µm particle size, Phenomenex, Torrance, CA, USA)
161 maintained at 30 °C. A mobile phase consisting of (A) water/formic acid (99.9:0.1, v/v) and (B)
162 acetonitrile/formic acid (99.9:0.1, v/v), at the constant flow rate of 0.55 mL min^{-1} was used.
163 The gradient program of solvent A was as follows : 0-1 min isocratic 94%; 1-26 min increase to
164 55%; 26-33 min decrease to 30%; 33-36 min isocratic 30%; 36-55 min increase to 94%. The
165 total flow was split using a T connection at the outlet of column and 220 µL min^{-1} was the
166 measured flow at the ESI interface. The MS conditions were: capillary temperature 320 °C;
167 source heater temperature 280 °C; nebulizer gas N_2; sheath gas flow 30 arbitrary units;
168 auxiliary gas flow 7 arbitrary units; capillary voltage -2800 V, S-Lens RF Level 60%. Data were
169 acquired in negative ionization mode. Samples were analyzed with two methods. A full scan
170 method from 150 to 1200 *m*/*z* was used to quantify the phenolic compounds by the extraction
171 of molecular ion signals in post-acquisition. A data-dependent experiment was used to collect
172 ms^2 data in order to identify the eluted compounds. The data-dependent settings were: full
173 scan from 150 to 1200 *m*/*z*, activation level 65000 counts, isolation width 2 Da, default charge
174 state 2, CID energy 35. The auto-tuning was performed on 5-*O*-caffeoylquinic acid. A
175 calibration curve was obtained by the injection of 5-*O*-caffeoylquinic acid standard solutions at
176 a concentration ranging from 50 to 0.05 mg L^{-1} (slope = 317738.76; LOD = 0.28; LOQ = 2.76; R^2
177 = 0.9999). All data were acquired and processed using Xcalibur v.2 (Thermo Fischer Scientific,
178 Waltham, MA, USA). The tentative identification of phenolic compounds was achieved by
179 combining elution times, molecular ions, MS/MS fragmentation patterns and literature data
180 (Negro et al., 2012). Since some discrepancies were observed about the identification of
181 isomers of caffeoylquinic and dicaffeoylquinic acids, a green coffee methanol extract, that
182 contains all isomers of these compounds, was prepared and analyzed in the same conditions,
183 according to Alonso-Salces et al. (2009). Semi-quantitative data for each identified compound

were obtained by using the calibration curve of 5-*O*-caffeoylquinic acid and assuming equal response.

Color determinations

Color indices of fresh pasta (yellow index, corresponding to *b**; red index, corresponding to *a**; brown index, corresponding to 100-*L*) were determined by means of the reflectance colorimeter Chroma Meter CR-300 (Konica Minolta Sensing, Osaka, Japan), with 10° Standard Observer and D65 illuminant.

Fresh pasta cooking test

Fresh pasta was put in boiling distilled water at 1:10 (w/v) pasta to water ratio, without the addition of salt, until optimum cooking time (OCT) was reached. Based on the empirical convention for this kind of product, OCT of fresh pasta (that, in contrast to dry pasta, does not show a well-perceivable white and opaque core gradually disappearing during cooking, commonly used to assess the OCT), was preliminary determined as the time when pasta pieces raised at the surface of boiling water, and accounted for 4 min and 20 s, and 4 min and 29 s for artichoke enriched and control pasta, respectively. After cooking and draining, the samples were rinsed with distilled water and allowed to rest for 5 min. Cooking loss and water absorption of pasta were determined as in Pasqualone et al. (2016).

Fresh pasta texture profile and viscoelastic parameters

A Z1.0 TN texture analyzer (Zwick Roell, Ulm, Germany) equipped with a stainless steel square probe (4 cm side) and a 1 kN load cell was used for performing the texture profile analysis (TPA) and for determining the viscoelastic parameters. Due to irregular geometry of *orecchiette*, for these analyses the dough was 3-mm sheeted (Atlas 150 Wellness sheeting

209 machine, Marcato, Campodarsego, Italy) and cut as 10-cm sided squares. The squared dough
210 sheets were then left to dry at room temperature to reach 26±1 g 100 g^{-1} of moisture, cooked
211 at OCT, drained, rinsed with distilled water, and allowed to rest for 5 min before analysis. Data
212 were acquired by means of the TestXPertII v. 3.41 software (Zwick Roell, Ulm, Germany) at the
213 frequency of 400 Hz. The TPA conditions involved a cyclic compression test: 1 mm/s probe
214 compression rate; 25% sample deformation in both the compressions; 10 s pause before
215 second compression. The analysis of viscoelastic parameters was set up as in D'Egidio et al.
216 (1993): load, 500 g; time of loading, 40 s; time of recovery after loading off, 20 s. The following
217 parameters were taken from the strain-time curve: initial pasta thickness (E, mm); thickness
218 before loading off (e_1, mm); final thickness (e_2, mm). Consistency (C), elastic recovery (ER), and
219 viscoelasticity index (VI) were defined and calculated as in D'Egidio et al. (1993).
220
221 **Statistical analysis**
222 Three extraction trials from artichoke by-products and three pasta-making trials were carried
223 out. All analytical determinations were performed in triplicate. Statistical analysis was carried
224 out using IBM SPSS software v. 19 (IBM Corp., Armonk, NY, USA). Significant differences were
225 determined by one-way ANOVA followed by Tukey HSD test.
226
227 **Results and discussion**
228
229 **TPC, AA and phenolic profile of artichoke by-product extracts**
230 The aqueous-ethanolic extracts of artichoke by-products obtained by ultrasound-assisted
231 technologies (US) showed significantly ($p < 0.05$) higher TPC contents than NO-US, irrespective
232 of the cultivar considered (Table 1), confirming the results of Punzi et al. (2014) and Rabelo et
233 al. (2016). US are known to act on plant tissues by the cavitational phenomenon induced at the

234 solid/liquid interface. This effect facilitates the release of extractable compounds and
235 enhances the mass transport by disrupting the plant cell walls (Chemat and Khan, 2011). After
236 removing ethanol from the US extracts, the concentrated aqueous solution (C-US) showed a
237 TPC content in the range 422-545 mg L^{-1}.
238 Capriccio cv. showed the highest TPC level both in NO-US and US extraction trials, followed by
239 Opal and Catanese cvs. However, the TPC content of C-US obtained from Opal cv. overcome
240 that of Capriccio cv., probably due to condensation and/or polymerization phenomena that
241 occurred during the concentration phase. The existence of a positive correlation could be
242 supposed between the initial TPC value and the extent of this polymerization process.
243 The combination of aqueous ethanol and US in recovering TPC from artichoke scraps was more
244 effective than water alone and US, proposed in a previous research (Punzi et al., 2014).
245 However, the TPC contents of our extracts were lower than those obtained, without US, by
246 using methanol (Bonasia et al., 2010), or mixtures of acetone, methanol, and ethanol (Negro et
247 al., 2012). The choice of avoiding the use of these toxic solvents and the removal of ethanol
248 (that could be subsequently recovered for containing costs at industrial level) after the
249 extraction, allowed us to obtain a healthy extract, suitable for subsequent food production.
250 The AA confirmed the results observed for TPC: the US extracts showed AA values significantly
251 higher ($p < 0.05$) than NO-US, irrespective of the cultivar. The subsequent concentration raised
252 the AA to the range 313-1662 mg L^{-1} in C-US (Table 1).
253 About the effect of cultivar on the AA, an unexpected result was obtained. In spite of the
254 lowest TPC content, Catanese cv. showed the highest AA level, irrespective of the type of
255 extraction, followed by Capriccio and Opal cvs. (Table 1). This behavior could be explained by
256 examining the profile of the phenolic compounds determined by UHPLC-ESI-MS/MS (Table 2).
257 In fact, Catanese cv. extracts showed significantly ($p < 0.05$) higher content of luteolin and

apigenin glycosides than the other two cultivars. Luteolin glycosides, in particular, have higher antioxidant activity compared to chlorogenic acid (Rice-Evans et al., 1997; Kim et al., 2000). Quantitative data evidenced other varietal differences in the phenolic profiles. In detail, irrespective of the type of extraction, the luteolin-7-*O*-rutinoside, followed by 5-*O*-caffeoylquinic acid, luteolin-7-*O*-glucoside, and apigenin-7-*O*-glucuronide, were the predominant compounds in Catanese cv. extracts. In Capriccio and Opal cvs., instead, 5-*O*-caffeoylquinic acid was the most abundant, followed by 3,5- and 3-4-di-*O*-caffeoylquinic acids in Capriccio cv., and by luteolin-7-*O*-glucoside and 3-4-di-*O*-caffeoylquinic acid in Opal cv. The phenolic profile of Opal cv. was particularly poor in luteolin-7-*O*-rutinoside. Overall, the observed results confirmed that 5-*O*-caffeoylquinic and 3,4-di-*O*-caffeoylquinic acids are the most abundant hydroxycinnamates in artichoke, whereas the main flavonoids are apigenin and luteolin, together with their glycosides (Pandino et al., 2010). In Figure 2 is reported the total ion chromatogram of the Catanese cv. US extract, as well as the single ion chromatograms of the phenolic compounds identified and quantified.

US extracts showed significantly ($p < 0.05$) higher concentrations of single phenolics than NO-US extracts, for all the cultivars considered (Table 2). However, comparing US with C-US, a non-univocal behavior was observed, with significant differences among cultivars. In particular, the concentration of some phenolic compounds increased in C-US, as expected, but other phenolics remained constant or even decreased, probably due to the above hypothesized condensation and polymerization phenomena. These decreases, minimal in Catanese cv. and moderate in Opal cv., were more marked in Capriccio cv. The findings were in good agreement with the TPC results reported in Table 1.

TPC, AA, phenolic profile and technological characteristics of fresh pasta

282 The high value of AA observed in C-US extract from Catanese cv. induced to choose this extract
283 as a functional ingredient for fresh pasta-making trials, with the purpose of innovating the
284 hand-made "*orecchiette*" traditional pasta type. The increase of AA, indeed, is one of the main
285 aims of pasta functionalization (Pasqualone et al., 2015; 2016). Moreover, Catanese cv. is
286 reported to have higher phenol content in waste than in the heart, as well as high waste to
287 total head ratio (Bonasia et al., 2010). Pasta enriched of Catanese C-US extracts showed
288 significantly ($p < 0.05$) higher TPC and AA than control pasta without extract (Table 3).
289 Increases of 63% and 49% were observed in TPC and AA respectively, demonstrating the
290 efficacy of the enrichment strategy adopted. The phenolic compounds detected in the
291 enriched pasta reflected the varietal profile of the extract used, with a prevalence of luteolin-
292 7-*O*-rutinoside (Table 4).
293 Functional food production may involve quality drawbacks. To take into account this
294 possibility, the main quality features of enriched pasta have been checked. Table 5 reports the
295 cooking performances, textural parameters, and color characteristics of control and enriched
296 pasta. Cooking loss of pasta depends on the degree of resistance to physical disruption upon
297 boiling (Pagani et al., 2007). Minimal solid loss into the cooking water denotes high pasta
298 quality (Lucisano et al., 2012). No significant differences were observed in cooking
299 performances of the two pasta types, evidencing that the extract from artichoke by-products
300 did not interfere with a proper gluten network formation. Both cooking loss, due to gelatinized
301 starch leaching to cooking water, and water absorption, related to the hydration degree and
302 tolerance of pasta to cooking, were in the ranges for good quality pasta (Cleary and Brennan
303 2006; Pasqualone et al., 2016; Piwińska et al., 2016).
304 Regarding the textural properties, good quality pasta is characterized by high firmness and
305 springiness. These two parameters were lower in artichoke pasta than in control, but without
306 statistically significant differences. Cohesiveness, related to the force of internal bonds holding

307 the pasta structure (Sissons et al., 2006), was not different between the two pasta types. As a
308 consequence, the texture profile analysis did not ascertain significant differences in the
309 derived parameter chewiness, defined as the effort required to masticate pasta to a state of
310 swallowing (Sissons et al., 2006).
311 Viscoelastic parameters (consistency, elastic recovery, and viscoelasticity index) agreed with
312 cooking loss data: in spite of the absence of a high-temperature (HT) drying step, known to
313 harden pasta consistency, the observed values were high and appeared to be similar to those
314 reported for HT-dried pasta (D'Egidio et al., 1993). This result was probably due to good quality
315 level of semolina used (11.8 g 100 g^{-1} dry gluten, 88 gluten index). Artichoke pasta samples
316 showed similar consistency but lower elastic recovery than control ($p < 0.05$). The latter
317 negatively influenced the viscoelasticity index, but without statistical significance.
318 If cooking performances and textural parameters of artichoke-enriched and control pasta were
319 similar, on the contrary significant differences were observed in the color indices. In particular,
320 artichoke pasta showed significantly higher brown index, and lower yellow and red indices
321 than control samples ($p < 0.05$), due to the brown-greenish color of the artichoke by-product
322 extract added. Pasta color was considered an essential attribute that strongly influences
323 consumer choice, being bright yellow pasta more appealing than discolored products.
324 However, undefined is today the actual response of consumer to the browning of pasta. The
325 recent introduction of new pasta types, perceived as more healthy, such as pasta from whole
326 wheat flour or from pseudocereals and cereals other than wheat, has been changing the
327 consumer behavior.
328
329 **Conclusions**
330 In conclusion, it was demonstrated the feasibility of functionalizing with extracts from
331 artichoke by-products the fresh pasta type chosen for the trials, namely the hand-made

semolina-based "*orecchiette*" pasta, achieving the purpose of increasing the healthy value and innovating a traditional food product.

Irrespective of the artichoke cultivar considered, it was confirmed the effectiveness of US in helping the extraction of phenolic compounds from scraps. Varietal differences were observed in the phenolic profiles: Catanese cv. extracts had higher contents of luteolin and apigenin glycosides than the other two artichoke cultivars examined, and showed higher AA. The use of the extract from Catanese cv. artichoke scraps in fresh pasta-making significantly affected the end-product color, but did not alter the main textural parameters and cooking performance of pasta.

The proposed strategy adds value to by-products of artichoke industrial processing, particularly abundant in cultivars with high waste to total head ratio, such as Catanese, and helps to increase the antioxidant dietary intake. In addition, the application of US allows an eco-friendly management of the extractive process because does not need toxic solvents and produces only biodegradable exhausted artichoke by-products.

Acknowledgements

This research was financially supported by the MIUR in the framework of the Scientific Research Programs PRIN 2009 "Innovative technologies for recovery of nutraceuticals from artichoke by-products" and PON/01_01145/1/8 "Sviluppo tecnologico e innovazione per la sostenibilità e competitività della cerealicoltura meridionale - ISCOCEM".

Conflict of interest

The authors declare no conflict of interest.

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445

Figure captions

Figure 1 '*Orecchiette*'-shaped fresh pasta, uncooked.

Figure 2 UHPLC-ESI-MS chromatograms of aqueous-ethanolic extract of Catanese cv. artichoke industrial by-products obtained by means of ultrasound-assisted technologies (top, total ions current; bottom, single ion extracts of identified compounds).

454 **Table 1** Content of total phenolic compounds (TPC) and antioxidant activity (AA) of aqueous-
455 ethanolic extracts of artichoke industrial by-products from three different cultivars. NO-US,
456 extract obtained without ultrasound; US, extract obtained with ultrasound; C-US, extract
457 obtained with ultrasound and concentrated. In the same column and within each cultivar,
458 means with different small letters differ significantly ($p < 0.05$). In the same column and for the
459 same treatment, means with different capital letters differ significantly ($p < 0.05$). TE =
460 equivalents of 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox).

Sample	TPC		AA (µmol TE L^{-1} extract)
	Yield (mg gallic acid kg^{-1} scraps f.w.)	Concentration (mg gallic acid L^{-1} extract)	
Catanese			
NO-US	370 ± 9^{bC}	123 ± 3^{cC}	416 ± 8^{cA}
US	519 ± 17^{aC}	173 ± 6^{bC}	554 ± 18^{bA}
C-US	519 ± 17^{aC}	422 ± 16^{aC}	1662 ± 54^{aA}
Opal			
NO-US	546 ± 5^{bB}	182 ± 3^{cB}	97 ± 2^{cC}
US	657 ± 5^{aB}	219 ± 2^{bB}	126 ± 3^{bC}
C-US	$657^{*}\pm5^{aB}$	545 ± 11^{aA}	313 ± 22^{aC}
Capriccio			
NO-US	617 ± 1^{bA}	206 ± 4^{cA}	167 ± 8^{cB}
US	720 ± 16^{aA}	240 ± 5^{bA}	354 ± 18^{bB}
C-US	$720^{*}\pm16^{aA}$	485 ± 11^{aB}	580 ± 9^{aB}

461 *The value of yield was the same of US extract because C-US derived from it.

Table 2 Phenolic compounds (mg L^{-1}, mean ± SD) determined by UHPLC-ESI-MS/MS in the aqueous-ethanolic extracts of artichoke industrial by-products from three different cultivars. NO-US, extract obtained without ultrasound; US, extract obtained with ultrasound; C-US, extract obtained with ultrasound and concentrated; nd, not detected. On the same row and within each cultivar, means with different small letters differ significantly ($p < 0.05$). In the same row and for the same treatment, means with different capital letters differ significantly ($p < 0.05$).

Phenolic compound	Catanese			Opal			Capriccio		
	NO-US	US	C-US	NO-US	US	C-US	NO-US	US	C-US
5-*O*-caffeoylquinic acid	16.1 ± 0.4^{cC}	32.1 ± 0.7^{aC}	22.5 ± 0.6^{bB}	33.7 ± 0.8^{bB}	41.1 ± 0.3^{aB}	38.5 ± 0.4^{aA}	53.8 ± 1.5^{bA}	59.8 ± 1.0^{aA}	5.9 ± 0.2^{cC}
Luteolin-7-*O*-rutinoside	31.2 ± 1.2^{bA}	54.5 ± 0.1^{aA}	55.9 ± 1.1^{aA}	0.3 ± 0.1^{bC}	0.3 ± 0.1^{bC}	0.5 ± 0.1^{aC}	6.3 ± 0.2^{bB}	7.8 ± 0.1^{aB}	4.0 ± 0.1^{cC}
Luteolin-7-*O*-glucoside	12.7 ± 0.1^{cA}	20.0 ± 0.1^{aA}	14.2 ± 0.3^{bA}	8.0 ± 0.1^{cB}	9.6 ± 0.1^{bB}	11.2 ± 0.1^{aB}	3.1 ± 0.1^{bC}	4.0 ± 1.0^{aC}	nd
Luteolin-7-*O*-glucuronide	1.3 ± 0.1^{c}	2.4 ± 0.1^{a}	1.8 ± 0.1^{b}	nd	nd	nd	nd	nd	nd
Apigenin-7-*O*-rutinoside	4.1 ± 0.2^{b}	4.7 ± 0.1^{a}	4.7 ± 0.1^{a}	nd	nd	nd	nd	nd	nd
3,5-di-*O*-caffeoylquinic acid	1.5 ± 0.1^{bC}	3.1 ± 0.1^{aC}	0.9 ± 0.1^{cB}	4.5 ± 0.1^{bB}	7.4 ± 0.2^{aB}	1.7 ± 0.1^{cA}	9.8 ± 0.1^{aA}	9.6 ± 0.2^{aA}	nd
3,4-di-*O*-caffeoylquinic acid	0.8 ± 0.1^{bC}	1.4 ± 0.1^{aC}	0.4 ± 0.1^{cB}	6.7 ± 0.2^{bB}	11.0 ± 0.2^{aA}	1.8 ± 0.1^{cA}	9.6 ± 0.7^{bA}	10.2 ± 0.3^{aB}	nd
Apigenin-7-*O*-glucuronide	7.5 ± 0.1^{cC}	8.3 ± 0.4^{bC}	9.5 ± 0.4^{aB}	1.3 ± 0.1^{bB}	1.4 ± 0.1^{bB}	3.4 ± 0.1^{aC}	6.4 ± 0.1^{cA}	7.4 ± 0.1^{bA}	17.6 ± 0.1^{aA}

Table 3 Total phenolic compounds (TPC) and antioxidant activity (AA) of fresh pasta enriched with concentrated ultrasonic extracts of artichoke by-products (cv. Catanese), compared to control pasta. Different letters in column indicate significant differences at $p < 0.05$. TE = equivalents of 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox).

	TPC (mg gallic acid kg^{-1} d.m.)	**AA (µmol TE kg^{-1} d.m.)**
Control pasta	306±23[b]	886±64[b]
Artichoke-enriched pasta	500±15[a]	1324±64[a]

Table 4 Phenolic compounds (mean ± SD) detected in fresh pasta enriched of concentrated ultrasonic extracts of artichoke industrial by-products (cv. Catanese).

Phenolic compound	**Amount (mg kg^{-1} d.m.)**
5-*O*-caffeoylquinic acid	1.62 ± 0.04
Luteolin-7-*O*-rutinoside	22.97 ± 0.41
Luteolin-7-*O*-glucoside	1.95 ± 0.02
Luteolin-7-*O*-glucuronide	0.34 ± 0.01
Apigenin-7-*O*-rutinoside	1.42 ± 0.01
3,5-di-*O*-caffeoylquinic acid	0.16 ± 0.01
3,4-di-*O*-caffeoylquinic acid	0.15 ± 0.02
Apigenin-7-*O*-glucuronide	1.49 ± 0.07

Table 5 Cooking performances, textural and viscoelastic parameters, and color characteristics of fresh pasta enriched of concentrated ultrasonic extracts of artichoke industrial by-products (cv. Catanese), compared to control pasta. Different letters in row indicate significant differences at $p < 0.05$.

	Control pasta	**Artichoke-enriched pasta**
Cooking performances		
Cooking loss (g 100 g^{-1})	3.3^{a}±0.1	3.5^{a}±0.1
Water absorption (g 100 g^{-1})	103^{a}±14	110^{a}±11
Textural parameters		
Hardness (N)	21.4^{a}±0.5	20.6^{a}±0.3
Springiness (-)	0.71^{a}±0.04	0.68^{a}±0.04
Cohesiveness (-)	0.61^{a}±0.03	0.57^{a}±0.02
Chewiness (N)	9.3^{a}±0.4	8.0^{a}±0.3
Viscoelastic parameters		
Consistency (%)	70.13^{a}±0.09	69.95^{a}±0.41
Elastic recovery (%)	72.21^{a}±1.16	67.13^{b}±1.42
Viscoelasticity index	241.75^{a}±11.06	234.95^{a}±13.01
Color characteristics		
Yellow index (*b**)	27.11^{a}±1.10	23.09^{b}±1.01
Red index (*a**)	1.31^{a}±0.12	-1.04^{b}±0.05
Brown index (100 – *L**)	19.93^{b}±1.12	23.34^{a}±1.26

Figure 1

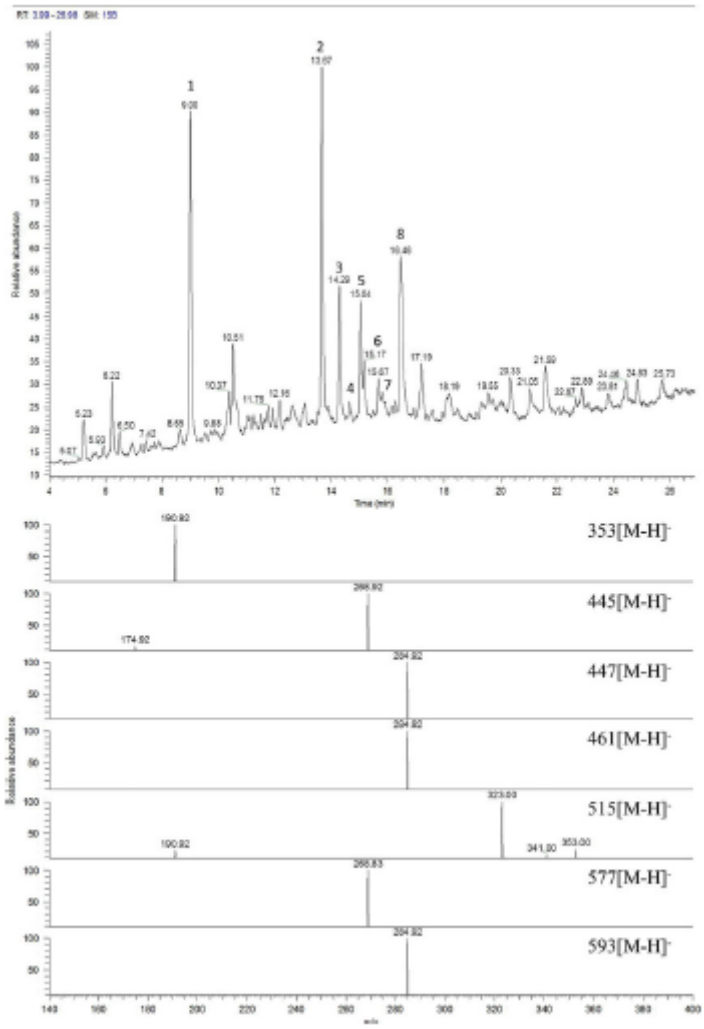

Figure 2