Influence of Temperature and Drying Time on Extraction Yield of Phenolic Compounds from Grape Pomace Variety "Portogizac"

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The influence of drying temperature (60 °C, 70 °C, 80 °C) and fluid-bed drying time (90 min, 135 min, 180 min) on the extraction yield of phenolic compounds and antioxidant activity of extracts were investigated. The content of phenolic compounds and antioxidant activity of extracts obtained from wet grape pomace (WGP) were 73.83 $mg_{GAE}\ g_{db}^{-1}$, 42.24 $mg_{CE}\ g_{db}^{-1}$, 30.53 mg g_{db}^{-1}, and 0.35 $g_{inhDPPH}\ g_{db}^{-1}$ for total phenolic compounds (TPC), total flavonoids (TF), total extractible proanthocyanidins (TPA), and antioxidant activity (AA), respectively. The applied drying conditions caused the reduction of content of all phenolic compounds down to 13.2 %, 43.1 %, 15.3 % and 21.0 % for TPC, TPA, TF and AA, respectively. The most abundant individual phenolic compound in grape pomace extracts was catechin (5.14 – 8.52 mg g_{db}^{-1}). The highest content of observed compounds was retained when applying drying temperature below 70 °C for 90 minutes.

Key words:

grape pomace, fluid-bed drying, phenolic compounds, antioxidant activity

Introduction

Sustainable food production is one of the main goals of the modern world. Nowadays, there is a growing interest in the exploitation of the by-products generated from agriculture and food production as raw materials for obtaining high-value products, which can be used for various purposes in the pharmaceutical, cosmetics and food industries.

During the winemaking process, about 20 % of the grapes[1–3] remains as a solid waste by-product (grape pulp, seeds, skin and stems), representing a rich source of various value-added products, such as ethanol, sugars, proteins, fruit acids, oils, hydrocolloids, dietary fibres and phenolic compounds. Phenolic compounds are particularly important due to their beneficial effects on human health, and their application in improvement of flavour, colour and stability of food[4–8]. Besides polyphenols, grape waste contains proteins, polysaccharides and lignin, which are associated with functional groups responsible for metal ion adsorption, thus making this agricultural waste a good alternative to expensive synthetic adsorbents. Chand *et al.*[9] tested cross-linked grape waste gel for the adsorption and separation of Cr(VI) ions from synthetic aqueous solution.

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Grape pomace has so far been investigated as a fertilizer or soil conditioner[10–13], as biomass for biofuel production[14–16] and as animal feed[17–20].

Wet grape pomace is a highly perishable material subject to uncontrolled microbial spoilage owing to its high moisture content (~60 $\%_{wb}$) and water activity. Therefore, the dehydration process is an important step in extending the shelf life of raw material and keeping its native properties before utilization for different purposes. The selection of the dehydration method depends on raw material properties, the desired characteristics of the dried product, the restriction on the operating conditions and costs. Sun drying is still a favourable method for drying some plant materials (fruits, vegetables, herbs, *etc*.) due to lower drying temperature and costs. However, its main drawbacks are dependence on weather conditions, often-poor product quality, long duration of process, possibility of material contamination with dust, soils, insects, *etc*. Otherwise, convection drying based on heated air in different designed drying devices (cabinet driers, tunnel driers, fluid-bed driers), vacuum drying, freeze-drying, and alternative methods, such as infrared drying and microwave drying, ensure better process control, shelf-stability and microbiologically safe dried product, with minimal degradation of nutrients and sensory quality[21,22].

Numerous researchers have used freeze-drying[2,23–27] or oven-drying[28–31] as a step in preparing/pre-treatment of grape pomace samples during investigation of the effect of different extraction regimes on the recovery of phenolic compounds and their antioxidant activities.

Several studies have investigated the effects of different drying methods on the recovery of phenolic compounds from grape pomace[32–37]. These studies have compared conventional hot air or vacuum drying with freeze-drying as a more effective drying method for by-products where material is not exposed to high temperatures resulting with preservation of organoleptic and nutritive properties of the products. Despite the advantages offered by freeze-drying, the main disadvantage is the long drying time and operating costs, thus the use of such drying method is justified only in obtaining value-added products.

Fluid-bed drying offers a higher rate of heat and mass transfer, and more homogeneous drying than convection-oven or vacuum drying in thin-layer. Furthermore, fluid-bed drying generates lower costs than freeze-drying[38]. This method is suitable for drying grains and other particulate materials. Application of fluid-bed drying for drying seeds[39], lactic acid starter culture[38], olive pomace[40] has been investigated previously. However, reports on fluid-bed drying of grape pomace in the literature are scarce concerning its effects on the phenolic content and antioxidant activity of extracts.

The aim of this study was to investigate the influence of drying air temperature and drying duration on the extraction yield of phenolic compounds from grape pomace and antioxidant activity of grape pomace extracts. The results were compared with undried samples.

Materials and methods

Chemicals

Ethanol (analytical grade), methanol (HPLC grade), acetonitrile (HPLC grade), Folin-Ciocalteu reagent and authentic HPLC standards of phenolic compounds ((+)-catechin, Ca; (–)-epicatechin, ECa; gallic acid, GA; syringic acid, SA; *p*-coumaric acid, *p*-CuA; t-resveratrol, *t*-Re) were purchased from Sigma Chemical Co. (St. Louis, MO). Standard stock solutions of phenolic compounds were prepared with methanol, wrapped in aluminum foil and stored at –20 °C.

Materials

Red grape (*Vitis vinifera* L. cv. Portogizac) pomace (harvest 2013) was provided by local winery from eastern Croatia. It consisted of skin, seeds and stems. Wet grape pomace (WGP) with 66.26 $\%_{wb}$ moisture was sealed in polyethylene bags and stored at –20 °C until used. The moisture of the grape pomace (before and after fluid-bed drying) was determined by drying in oven at 105 °C until constant weight.

Methods

Fluid-bed drying

Wet grape pomace (25 g) was dried in a bench-scale fluidized-bed drier (FBD 2000, UK) at different temperatures (60 °C, 70 °C or 80 °C) for 90 min, 135 min or 180 min. The dried grape pomace (DGP) was cooled in a desiccator, milled in a blender (HR 2860, Philips) and stored at +4 °C until used for extraction.

Extraction procedure

About 1 g of milled sample (WGP and DGP) was extracted with 40 mL of 50 % aqueous ethanol solution in capped flasks placed in a shaking water-bath at 200 rpm for 120 minutes at 80 °C. Following extraction, the suspension was centrifuged (Multifuge 3 L-R) at 15000g for 10 minutes at room temperature. The obtained supernatants were used for determination of phenolic compounds and antioxidant activities. All extractions were conducted in duplicate.

Determination of total phenolic compounds

The content of total phenolic compounds (TPC) in the extracts was determined using the Folin-Ciocalteu method with microscale protocol[41]. Gallic acid was employed as a calibration standard and the results were expressed as gallic acid equivalent per gram of dried grape pomace ($mg_{GAE}\ g_{db}^{-1}$). Determination of TPC was conducted in duplicate.

Determination of total flavonoids

The content of total flavonoids (TF) of grape pomace extracts was measured spectrophotometrically using the aluminum chloride colorimetric assay at 510 nm (UV-1700, Shimadzu) according to Marinova *et al.*[42]. Determination of TF in each extract was made in duplicate and the TF was expressed as (+)-catechin equivalent per gram of dried grape pomace ($mg_{CE}\ g_{db}^{-1}$).

Determination of extractable proanthocyanidins content

The content of total extractible proanthocyanidins (TPA) was determined using acid butanol as-

say where treatment in butanol leads to depolymerization of the proanthocyanidins to anthocyanidins, and due to formation of red colour, they could be detected by spectrophotometer at 540 nm[43]. Analyses were performed in duplicate and the results of TPA content were expressed on dry basis of grape pomace (mg g_{db}^{-1}).

HPLC analysis of phenolic compounds

The individual phenolic compounds analysis was carried out using a HPLC (Hewlett Packard, 1100 Series, Palo Alto, CA, USA) equipped with a C18 reverse-phase column (201TP54, Vydac, Hesperia, CA, USA) coupled with a DAD detector. The mobile phase was water/acetic acid (99:1 %, v/v) and methanol/acetonitrile (50:50 %, v/v), following the method described by De Faveri *et at*.[44] The results were expressed as the mass of individual phenolic compounds per gram of dried grape pomace (mg g_{db}^{-1}).

Determination of antioxidant activity

Antioxidant activity (AA) of grape pomace extracts was evaluated using 2.2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging assay as described by Brand-Williams *et al.*[45] with some modifications[43]. All tests were performed in duplicate and the results were expressed as the mass of inhibited DPPH per dry basis of grape pomace ($g_{inhDPPH}$ g_{db}^{-1}).

Statistical analysis

Statistica 12 (Stat Soft Inc., USA) was used for data analysing. All results were expressed as a mean value. Statistical significant difference ($p < 0.05$) between phenolic compound content and AA of different extracts was evaluated by oneway ANOVA coupled with LSD *post-hoc* test at confidence level of 95 %.

Results and discussion

Drying methods and process conditions play a significant role in the stability of phenolic compounds because high temperatures can lead to degradation of phenolic compounds and affect antioxidant activity of grape pomace[32]. In this study, the content of all quantified phenolic compounds and antioxidant activity of grape pomace extracts were influenced by using drying conditions.

The moisture content of WGP (66.26 $\%_{wb}$) reduced proportionately with the drying temperature and prolonged drying time, and the moisture of DGP amounted from 11.33 $\%_{wb}$ (at 60 °C for 90 min) to 9.58 $\%_{wb}$ (at 80 °C for 180 min). The influence of applied drying conditions (temperature and drying time) on moisture content of grape pomace was statistically significant at $p < 0.05$ (Fig. 1).

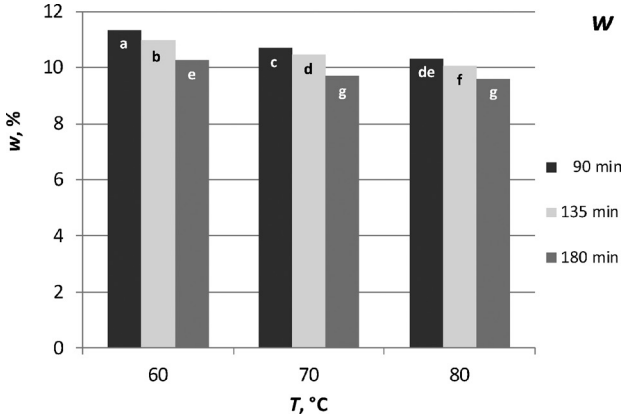

Fig. 1 – *Effect of drying air temperature and duration of fluid-bed drying on moisture content (w) of grape pomace (statistically significant difference is marked with different Latin letters, $p < 0.05$)*

TPC in DGP extracts ranged from 64.12 mg_{GAE} g_{db}^{-1} to 72.30 mg_{GAE} g_{db}^{-1} (Fig. 2a). Owing to the data dispersion, it was difficult to define a clear dependence of TPC on the drying conditions. Generally, it can be seen that TPC of DGP obtained for drying time of 90 minutes and 180 minutes at 60 °C and 70 °C was not significantly different in relation to WGP extracts (73.83 mg_{GAE} g_{db}^{-1}), while the increase in drying temperature up to 80 °C caused the highest drop in TPC, down to 13.2 % with regards to WGP extracts. In addition, TPC was lower at 135 minutes drying than for 180 minutes, regardless of the applied drying temperature. It can be assumed that prolonged exposure of grape pomace to drying treatment may result in the formation of new compounds (*e.g.* Maillard products) which could react with Folin-Ciocalteu reagent among other compounds and interfere with the determination of total phenolic compounds[46].

The content of TF in DGP extracts were in the range 35.79 – 40.62 mg_{CE} g_{db}^{-1} (Fig. 2b) and ANOVA showed a statistically significant ($p < 0.05$) reduction in the content of TF (down to 15.3 %) either at higher temperatures (> 70 °C) or at 70 °C, and a longer drying time (> 135 min) compared with WGP extracts (42.24 mg_{CE} g_{db}^{-1}). In contrast, there were no statistically significant differences between TF in WGP and DGP dried at 60 °C during all times, and at 70 °C for 90 minutes and 135 minutes.

TPA in DGP was in the range 17.38 – 19.45 mg g_{db}^{-1} (Fig. 2c). TPA in DGP obtained under used fluid-bed drying temperature-time conditions was significantly ($p < 0.05$) decreased (down to 43.1 %) compared to WGP (30.53 mg g_{db}^{-1}). It can be seen

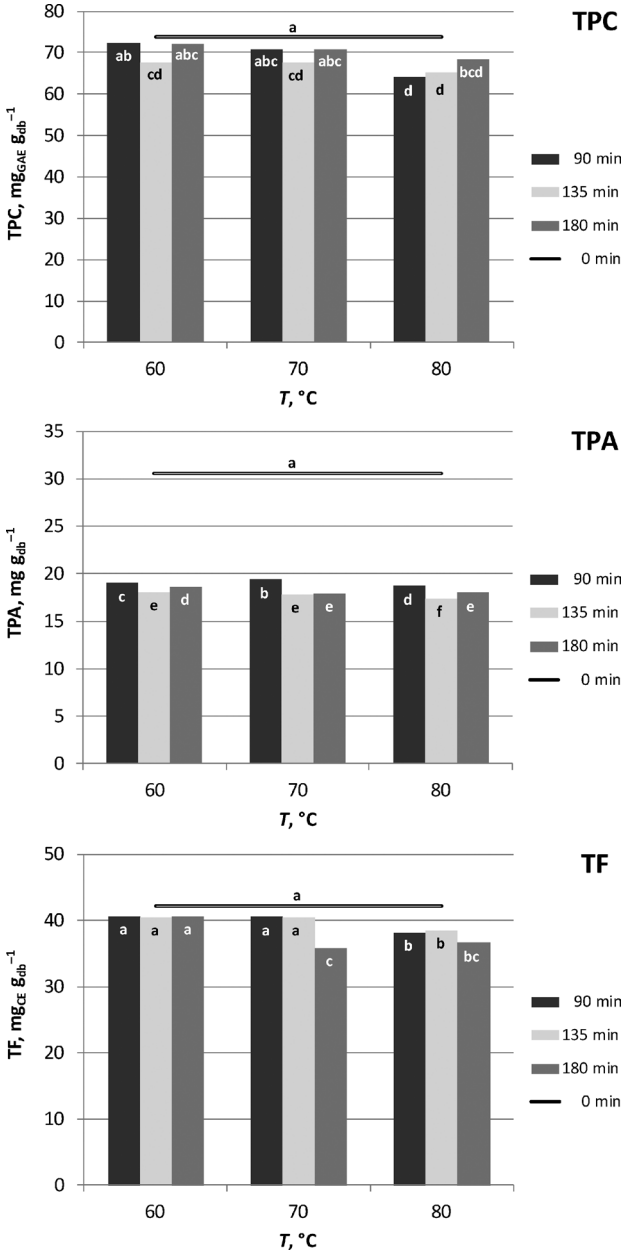

Fig. 2 – *Effect of drying air temperature and duration of fluid-bed drying on (a) total phenolic compounds (TPC), (b) total flavonoids (TF) and (c) total extractible proanthocyanidins (TPA) of grape pomace in comparison with TPA, TF and TPA in extracts of wet grape pomace marked with a line (statistically significant difference is marked with different Latin letters, $p < 0.05$)*

that the highest TPA in DGP was reached in 90 minutes of drying, regardless of drying temperature, and prolonged drying caused a decrease in TPA. Additionally, the greatest decrease in TPA for DGP was observed at 80 °C at all drying times studied. Larrauri *et al.*[32] reported that TPC and TPA in oven-dried DGP was affected with temperatures higher than 100 °C, while drying at 60 °C did not cause significant changes in TPC and TPA compared to freeze-dried samples.

Other authors have published more or less similar results regarding the phenolic compounds content in WGP extract and DGP extracts. Makris *et al.*[47] reported lower TPC (54.02 mg$_{GAE}$ g$_{db}^{-1}$) and TF (23.49 mg$_{CE}$ g$_{db}^{-1}$) for red WGP extracts obtained by maceration with 0.1 % HCl in methanol/acetone/water (60/30/10 v/v/v) during 30 min. Casazza *et al.*[8] used high-pressure and temperature reactor to extract polyphenols from grape pomace of Pinot Noir cultivar. They noticed that the highest TPC (60.7 mg$_{GAE}$ g$_{db}^{-1}$) and TF (15.1 mg$_{CE}$ g$_{db}^{-1}$) were obtained working at 150 °C and 270 min, and 150 °C for 15 min, respectively.

Furthermore, the content of six individual phenolic compounds were determined using HPLC in DGP extracts as follows: catechin (5.14 – 8.52 mg g$_{db}^{-1}$), epicatechin (2.42 – 3.02 mg g$_{db}^{-1}$), syringic acid (0.80 – 0.92 mg g$_{db}^{-1}$), *t*-resveratrol (0.47 – 0.62 mg g$_{db}^{-1}$), *p*-coumaric acid (0.24 – 0.33 mg g$_{db}^{-1}$), and gallic acid (0.07 – 0.14 mg g$_{db}^{-1}$). These compounds in WGP were 8.61, 2.86, 0.97, 0.60, 0.32 and 0.09 mg g$_{db}^{-1}$, respectively (Fig. 3).The interaction between drying temperature and duration of drying on measured GA concentration in extracts was observed. A negative effect of prolonged drying time was observed when the temperature of 60 °C was applied, while the positive influence of drying time was observed when higher temperatures (70 °C and 80 °C) were investigated. The reason for this could be the fact that higher temperatures and prolonged time cause the change of material structure, polymer degradation, as well as enzyme degradation, which all may affect the availability and extractability of GA from DGP.

Rubilar *et al.*[48] reported for WGP as well as Lu and Foo[49] for freeze-dried grape pomace that phenolic acid, flavan-3-ols, flavonoids were dominant compounds among others. Lafka *et al.*[50] testified that gallic acid, catechin and epicatechin were the major phenolic compounds in ethanol extract of winery waste (grape skin and seeds) from red wine-making.

Boonchu and Utama-ang[3] found in red WGP a similar content of TPC (86.3 – 17.8 mg g$_{db}^{-1}$), catechin (9.93 – 16.1 mg g$_{db}^{-1}$) and epicatechin (1.6 – 2.5 mg g$_{db}^{-1}$), while the content of *t*-resveratrol was 100 times lower (0.0020 – 0.0074 mg g$_{db}^{-1}$). Rockenbach *et al.*[51] reported similar values for TPC (75.75 mg g$_{db}^{-1}$) but lower for values of catechin and *t*-resveratrol corresponding to 1.51 and 0.04 mg g$_{db}^{-1}$ of WGP. Tseng and Zhao[36] reported lower values for TPC (18.08 – 40.98 mg g$_{db}^{-1}$) detected in DGP variety Merlot obtained using four drying methods, including conventional drying in oven at 40 °C, vacuum drying at 40 °C, ambient air drying at 25 °C, and freeze drying. Literature data are often non-comparable since it is known that different factors (culti-

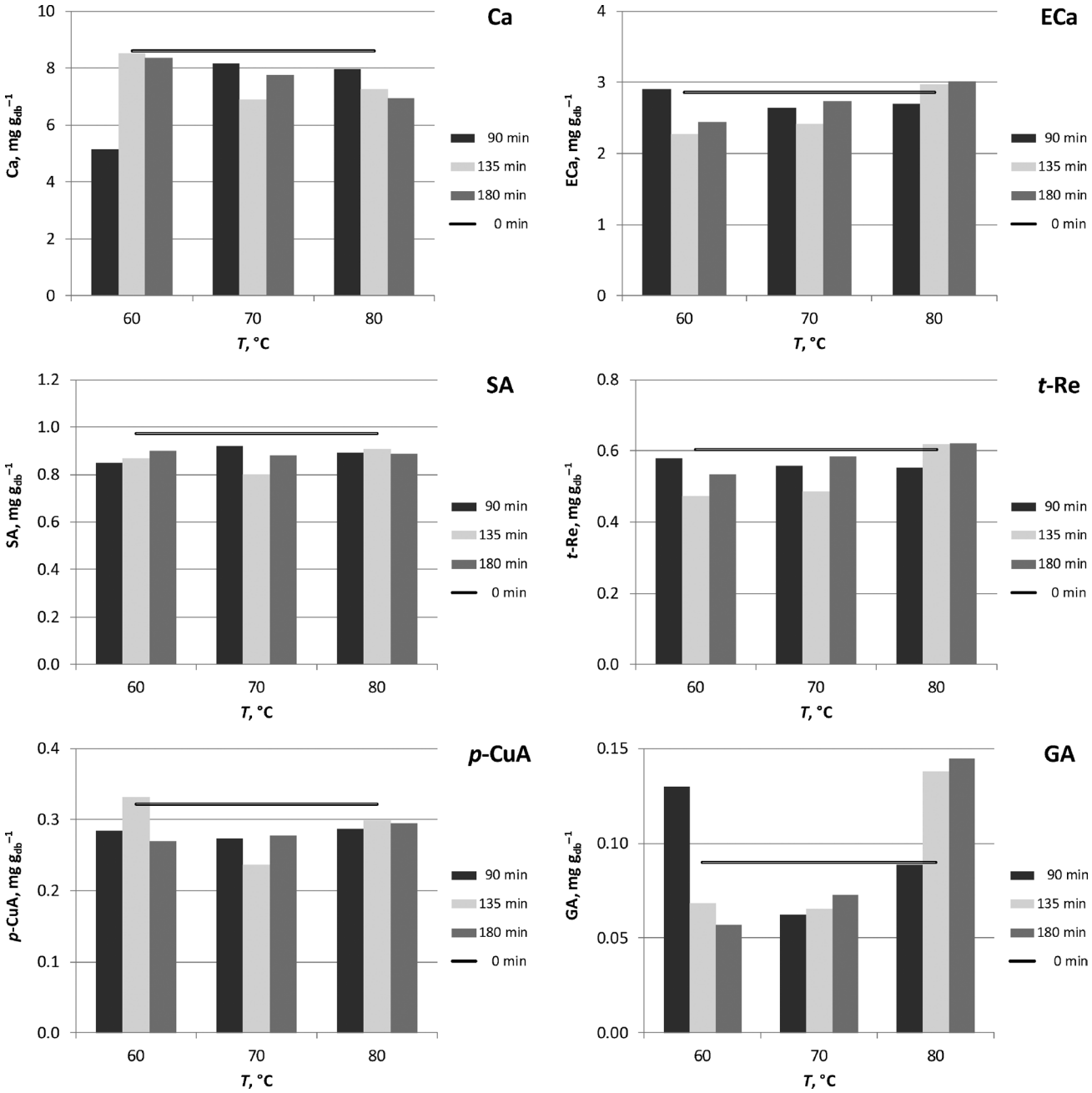

Fig. 3 – *Effect of drying air temperature and duration of fluid-bed drying on (+)-catechin (Ca), (–)-epicatechin (ECa), syringic acid (SA), t-resveratrol (t-Re), p-coumaric acid (p-CuA) and gallic acid (GA) of grape pomace in comparison with concentration of same individual phenolic compounds in extracts of wet grape pomace marked with a line*

vars, growing environment, harvest time, winemaking conditions, storage conditions, extraction conditions, analytical method, etc.) affect the content of phenolic compounds in extracts.

According to the results presented in Fig. 4, it is evident that extracts of DGP have lower antioxidant activity based on DPPH assay (0.28 – 0.31 $g_{inhDPPH}$ g_{db}^{-1}) in comparison to the extracts of undried samples of grape pomace, WGP extract (0.35 $g_{inhDPPH}$ g_{db}^{-1}), regardless of the drying conditions. Although it can be assumed from Fig. 4 that the influence of different drying conditions on AA is negligible, statistical analysis (one-way ANOVA analysis with LSD post-hoc test at confidence level 95 %) suggests that variances in AA of different DGP extracts are statistically significant. According to the obtained results, it can be concluded that higher drying temperatures and prolonged drying process had greater impact on AA (lower AA was measured) in comparison to the milder drying temperature-time treatment. Thus, the extracts of grape pomace that was dried 180 min at 80 °C had 20 % lower AA, whereas the grape pomace sample dried for 90 min at 60 °C had 11.43 % lower AA with regard to WGP extract. The 20 %

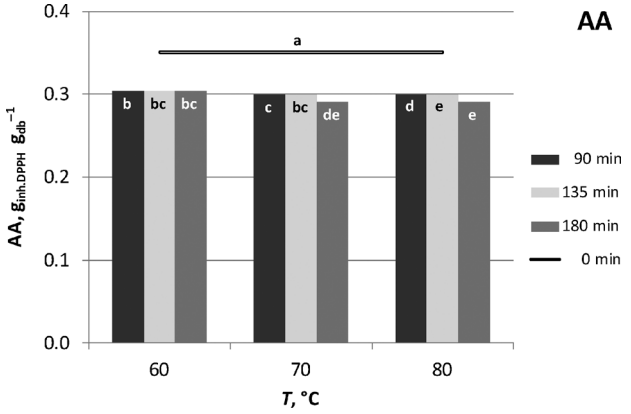

Fig. 4 – *Effect of drying air temperature and duration of fluid-bed drying on antioxidant activity (AA) of grape pomace in comparison with antioxidant activity of wet grape pomace extract marked with a line (statistically significant difference is marked with different Latin letters, $p < 0.05$)*

lower AA was measured in the extract of the samples that were dried at 80 °C/180 min, while 11.43 % lower AA was obtained in the extracts of the samples that were dried at 60 °C/90 min.

Moderate positive correlation between AA and TPC (R = 0.66) and high positive correlation between AA with TF (R = 0.82) and TPA (R = 0.88) suggests that the determined phenolic compounds, particularly TPA, significantly contribute to the total antioxidant activity of WGP and DGP (Fig. 5). The reduction in AA for DGP (down to 21 %) compared to WGP is probably the result of the degradation of phenolic compounds during drying treatment caused by higher/longer temperature/time regimes. Considering DGP, the AA of the samples dried at 60 °C during all times, and the DGP at 70 °C during 135 min did not differ significantly ($p > 0.05$). The same observation was detected for samples dried at 60 °C for a longer time (135 min and 180 min) and the samples dried at 70 °C for 90 minutes and 135 minutes.

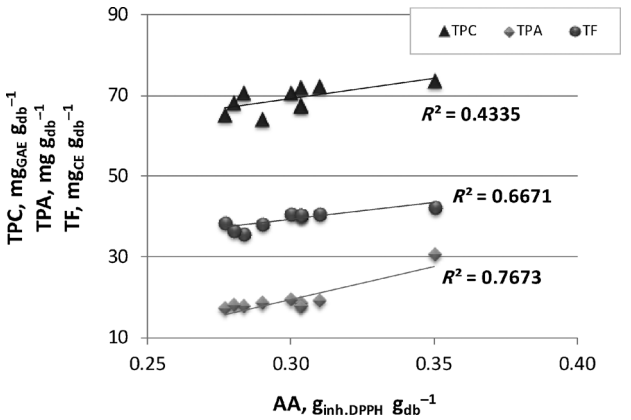

Fig. 5 – *Correlation of total phenolic compounds (TPC), total extractible proanthocyanidins (TPA), total flavonoids (TF) with antioxidant activity (AA) of grape pomace extracts*

A medium to weak positive correlation between individual phenolic compounds and AA was found with the following order from the highest to lowest value: GA > *t*-Re > C > *p*-CA > SA > EC (Fig. 6). Based on the above, it can be assumed that drying significantly affected the content of detected individual phenolic compounds. On the other hand, there is the possibility of interactions of detected phenolic compounds with other phenolic compounds of grape pomace, which may lead to unpredictable antioxidant efficiency, since phenomena synergism or antagonism can occur[47].

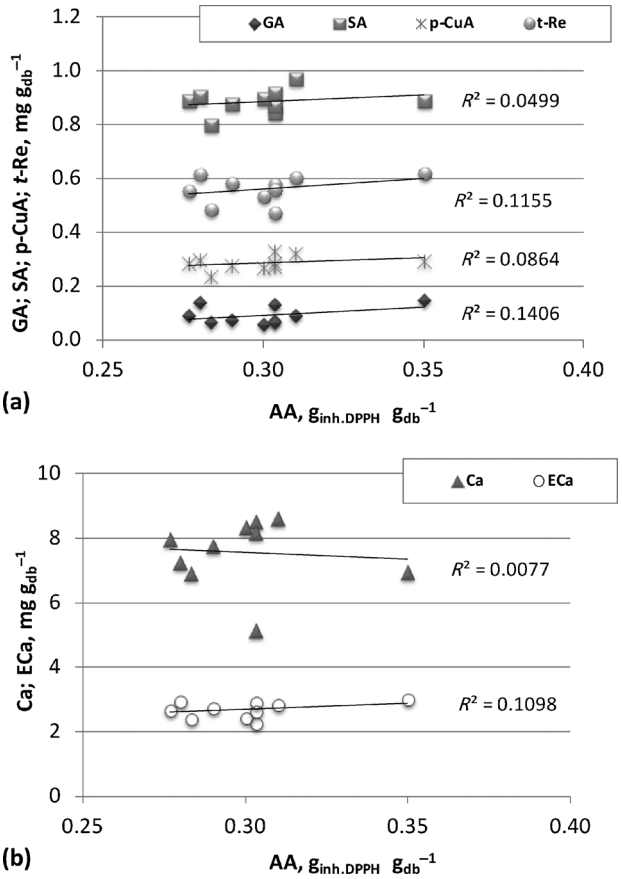

Fig. 6 – *Correlation of individual phenolic compounds: (a) gallic acid (GA), syringic acid (SA); galic acid (GA), p-coumaric acid (p-CuA), t-resveratrol (t-Re), (b) (+)-catechin (Ca), (–)-epicatechin (ECa) with antioxidant activity (AA) of grape pomace extracts*

Conclusion

In this study, the drying of grape pomace and extraction of the antioxidants contained in the wine industry by-products (pomace) was investigated. The effect of air temperature (60 °C, 70 °C, 80 °C) and drying time (90 min, 135 min, 180 min) of fluid-bed drying on phenolic compound extraction efficiency and antioxidant activities was examined.

The results suggest that fluid-bed drying at gentle temperature-time conditions is a promising

technique for preservation of grape pomace, and use as value added products like bioactive phenolic compounds. The best conditions obtained in this study were temperature lower than 70 °C and drying time of 90 minutes.

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Symbols/abbreviations used

AA – antioxidant activity, $g_{inhDPPH}\, g_{db}^{-1}$
Ca – (+)-catechin, mg g_{db}^{-1}
DGP – dried grape pomace
ECa – (–)-epicatechin, mg g_{db}^{-1}
GA – gallic acid, mg g_{db}^{-1}
p-CuA – *p*-coumaric acid, mg g_{db}^{-1}
SA – syringic acid, mg g_{db}^{-1}
T – drying air temperature, °C
TF – total flavonoids, $mg_{CE}\, g_{db}^{-1}$
TPA – total extractible proanthocyanidins, mg g_{db}^{-1}
TPC – total phenolic compounds, $mg_{GAE}\, g_{db}^{-1}$
t-Re – *t*-resveratrol, mg g_{db}^{-1}
w – moisture content, %
WGP – wet grape pomace

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