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Effect of air drying temperature on the phenolics and antioxidant activity of Xuan-Mugua fruit

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Abstract

Xuan-Mugua is a Chinese traditional fruit and is mainly preserved by air drying. To explore the effect of temperature on the drying property and quality of the fruits, they were sliced and dried at 60-90 °C. The results indicated that the drying time shortened from 150 to 80 min with the increasing temperature and the minimal time was found at 90 °C. The total phenolic content and antioxidant activity generally decreased by 5.09-18.27% at higher temperature (90 °C) and their maximums respectively appeared at 60 and 80 °C. Compared with the medium temperatures (60 and 70°C), the phenolic profiles changed obviously at higher temperatures (80 and 90 °C), and a transformation of chlorogenic acid was detected; besides, with the increasing temperature, the main phenolics including catechin and chlorogenic acid respectively decreased by 29.3% and 18.4%. Taking the above findings together, 70 °C should be the proper temperature to dry Xuan-Mugua fruits.

Keywords: chaenomeles fruit; drying temperature; phenolic compound; antioxidant activity.

Practical Application: This study revealed that drying Xuan-Mugua fruits under high temperatures of 80 and 90 °C generally led to the decrease in phenolics and antioxidant activity, and the transformation of some phenolics, implying that the Xuan-Mugu fruits should dry at low temperatures (e.g. 60 and 70 °C) to maintain a high quality, which may provide useful guidance to the drying of Xuan-Mugua fruits in industry.

1 Introduction

'Mugua' is the Chinese name of Chaenomeles species and at least five cultivars have been found in China (Du et al., 2013). The cultivar *Chaenomeles speciosa* (Sweet) Nakai cultivated in Xuancheng city (Anhui, China) is called Xuan-Mugua (Shang et al., 2019), which has been served as traditional Chinese medicine and food for thousands of years (Zhang et al., 2014). The fruit of Xuan-Mugua is traditionally used to treat rheumatoid arthritis, hepatitis, asthma and common cold (Huang et al., 2018), and the main chemical components in the fruit are found as polysaccharides (Xie et al., 2016), polyphenols and triterpenes (Miao et al., 2016). Recent studies reveal that Xuan-Mugua fruit also shows protective activity against microbes (Wang et al., 2021), cardiovascular diseases and inflammatory (Zhang et al., 2010), and phenolics are considered to be responsible for these activities.

Phenolics are typical phytochemicals that are widely distributed in the different parts of plants such as roots (Wang et al., 2022), stems, leaves (Mazahir et al., 2022), fruits and seeds, showing dominant antioxidant (Mitrović et al., 2021), anti-wrinkling (Byun et al., 2021) and antidiabetic activity (Shafay et al., 2021). According to their structural characteristics, phenolic phytochemicals are generally divided into two main groups such as phenolic acids and flavonoids (Vargas-Madriz et al., 2021). As to the Xuan-Mugua fruit, the presentative phenolic acid is chlorogenic acid, whereas the main flavonoids are catechin, quercetin and their derivatives (Zhu et al., 2019).

Drying technology is widely used to preserve foods such as fruits, and air drying, freezing drying and microwave drying are commonly used methods (Khaing Hnin et al., 2019). Among these methods, air drying is frequently used in the industry (Yang et al., 2018). Temperature is the main parameter for air drying (Lewicki 2006), and it may affect the phenolics and antioxidant activity of the fruit (Kamiloglu et al., 2016), and these properties could reflect the quality of the dried fruit to some degree. Previous studies have shown that drying temperature had a significant effect on the phenolics and antioxidant activity of fruits slices, including apple (Vega-Gálvez et al., 2012), pear (Santos et al., 2014), and kiwi (Izli et al., 2017). However, this effect on the fruit slices varies with the fruit types. To date, the effect of air drying temperature on the phenolics and antioxidant activity of Xuan-Mugua fruit is still unknown.

In this study, to explore the effect of air drying temperature on the phenolics and antioxidant activity of Xuan-Mugua fruit, its slices were dried under different temperatures. The drying

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curves, phenolic content and antioxidant activity of the samples were further studied.

2 Materials and methods

2.1 Materials and reagents

Xuan-Mugua fruits were collected from the plantation of Xuancheng (Anhui, China). Chemicals including Folin-Ciocalteu phenol reagent, 2,2'-azinobis-(3-ethylbenzo -thiazoline-6-sulfonate) (ABTS), and 2,2-diphenyl-1-picrylhydrazyl (DPPH) were purchased from Aladdin Chemicals Ltd. (Shanghai, China). Other chemicals were bought from Sinopharm group Ltd. (Shanghai, China). All solvents used were of analytical or HPLC grade.

2.2 Drying experiments

Xuan-Mugua fruits were cut into slices with a thickness of 2 mm and dried at different temperatures (60, 70, 80 and 90 °C) to constant weight in a drying oven. The samples were weighed at different time courses during the drying process to obtain the drying curves, including moisture ratio (MR) and drying rate (DR), and these parameters were calculated by following equations (Equation 1 and Equation 2) (Deng et al., 2020):

$$MR = \frac{M_t - M_e}{M_0 - M_e} \tag{1}$$

$$DR = \frac{M_{t_1} - M_{t_2}}{t_2 - t_1} \tag{2}$$

Where M_t is the moisture content at a certain drying time, M_e is the moisture content at equilibrium, M_0 is the initial moisture content; M_{t1} and M_{t2} are the moisture content at drying time t_1 and t_2, and t_1 and t_2 are the various times (min).

The final samples with constant weight were grounded to powder and stored at -20 °C until use.

2.3 Extract preparation

Xuan-Mugua dried powder (0.5 g) was extracted with 70% ethanol aqueous (10 mL) under sonication for 60 min, and the clear extract was obtained by centrifugation (8000 rpm, 10 min). The extracts were kept at -20 °C before analysis.

2.4 Total phenolic compound content (TPC)

The method was adapted from (Ma et al., 2021) with small modification. The extract (400 μL) and Folin-Ciocalteu reagent (50 μL) were mixed in a centrifuge tube and kept in the dark for 5 min, followed by adding Na_2CO_3 (10%, 500 μL); the mixture was then kept in the dark for another 30 min at room temperature before recording the absorbance at 730 nm by a UV-8000S spectrophotometer (Metash, China). The TPC was expressed as gallic acid equivalents (GAE) per 1 gram dried weight (D.W.).

2.5 Total flavonoid compound content (TFC)

The method was adapted from (Ma et al., 2021). The extract (250 μL) was mixed with $NaNO_2$ (1.6%, *m/v*, 250 μL) and kept in the dark for 5 min, followed by addition of $AlCl_3$ (3.2%, *m/v*, 250 μL); after keeping for 5 min, the mixture was mixed with NaOH (8%, *m/v*, 250 μL) and kept for another 15 min in the dark at room temperature before reading the absorbance at 510 nm on the above-mentioned spectrophotometer. The TFC was expressed as quercetin equivalents (QE) per 1 gram dried weight (D.W.).

2.6 DPPH radical scavenging activity

The DPPH radical scavenging activities of the samples were determined as follows (Ma et al., 2020): the extract (50 μL) and DPPH (0.2 mM, 500 μL) were mixed and kept in the dark for 30 min at room temperature, and the absorbance at 513 nm was recorded. The DPPH radical scavenging activities were expressed as trolox equivalents (TE) per 1 gram dried weight (D.W.).

2.7 ABTS radical scavenging activity

The scavenging activities of the samples against ABTS radical were determined as follows (Ma et al., 2020): to a mixture of the extract (50 μL) and ethanol (450 μL), freshly diluted ABTS (the 723 nm absorbance is around 0.7, 500 μL) was added. The mixture was incubated in the dark at room temperature for 5 min and the absorbance at 723 nm was read. The ABTS radical scavenging activities were expressed as trolox equivalents (TE) per 1 gram dried weight (D.W.).

2.8 HPLC-MS analysis

The extracts of dried Xuan-Mugua fruit were analyzed by M5100 high performance liquid chromatography system (HPLC, Dalian Elite, China) equipped with a reverse phase column (250 × 4.6 mm, 5 μm, Shimadzu) and a Chrom5100 workshop (Dalian Elite, China). The samples were eluted with water (solvent A) and acetonitrile (solvent B), and both solvents containing 0.8% acetic acid. The elution program was as follows: 0-6 min, B increased from 2 to 8%; 6-26 min, B increased from 8 to 12%; 26-50 min, B increased from 12 to 24%; 50-55 min, B increased from 24 to 100%; 55-60 min, B decreased from 100 to 2%; 60-65 min, B maintained at 2%. The flow rate was 1 mL/min and UV detection was carried out at 280 nm. LC-MS analysis was performed using an Agilent 1260/6460 LC/MSD system, and the mass spectra were obtained using electrospray ionization in the negative ionization mode in the range of m/z 100-1000; the rest of conditions were identical to those of the HPLC analysis. The content of the individual phenolics were quantified by reference standards and expressed as mg per 1 gram dried weight (D.W.).

2.9 Statistical analysis

The results in one experimental group were compared by one-way ANOVA, and mean difference was determined by Tukey's multiple comparison test at $p < 0.05$. All data were expressed as mean ± standard deviation for replicate analyses (n ≥ 3).

3 Result and discussion

3.1 Drying curves of Xuan-Mugua fruit

To explore the effect of temperature on the drying curves of Xuan-Mugua fruit slices, the moisture ratio (MR) and drying rate (DR) of the samples were determined. As shown in Figure 1A, to reach the desired moisture content, the drying time of Xuan-Mugua fruit slices was respectively about 150, 120, 90, and 80 min at temperatures of 60, 70, 80, and 90 °C, indicating that the drying duration was obviously shortened with the increasing temperatures. The result was consistent with that of pumpkin (Ouyang et al., 2021) and orange peel (Deng et al., 2020). Besides the moisture ratio, the drying rate of Xuan-Mugua fruit slices was also affected by drying temperatures. As it is indicated in Figure 2B, the drying rates of the samples accelerated with the increase of drying temperature and higher rates were found at the temperature of 90 °C, and this result may be explained by the enhanced vaporization of water under higher temperatures, and similar finding was also reported in apricot (Ihns et al., 2011). Besides the temperature, the drying rates were also affected by the moisture content. High moisture content samples contain more evaporable water (free water) that could quickly be removed in the early stage of the drying, showing a high drying rate; however, with the prolonging of drying duration, the content of free water was decreased, leading to a decrease in drying rate (Karathanos, 1999). The drying rate finally decreased to approximate zero and reached the drying equilibrium, which may result from the almost complete removal of free water from the samples. In this step, the remaining water should be the bound water, which could hardly remove from the samples, and this water led to the relatively constant moisture of the final dried samples.

3.2 Phenolic content of dried samples

To detect the effect of drying temperature on the phenolic content of Xuan-Mugua fruit slices, the TPC and TFC of the dried samples were shown in Figure 2. As it is indicated in Figure-2A, the content of TPC gradually decreased from 78.69 ± 8.25 to 72.86 ± 2.97 (mg GAE per 1 g D.W.) when the drying temperature increased from 60 to 90 °C, and only a slight decrease of the TPC was observed, indicating that the phenolics of Xuan-Mugua fruit slices was almost stable during this drying. As to the TFC (Figure 2B), with the increase of drying temperature from 60 to 90 °C, the content of TFC decreased from 208.50 ± 22.76 to 170.61 ± 9.65 (mg LTE per 1 g D.W.), indicating that drying temperature had a greater effect on flavonoids. These results in phenolic content were similar to the findings found in *Stevia rebaudiana* leaves (Gąsecka et al., 2020), and the partial degradation or transformation of the phenolics including flavonoids under the high temperature may be the reason for the change.

3.3 Antioxidant activity of dried samples

To detect the effect of drying temperature on the antioxidant activity of Xuan-Mugua fruit slices, their scavenging activity against DPPH and ABTS radicals were determined. As shown in Figure 3A, with the increasing of drying temperature from 60 to 80°C, the DPPH radical scavenging activity increased from 174.09 ± 38.66 to 199.38 ± 20.35 (mg TE per 1 g D.W.),

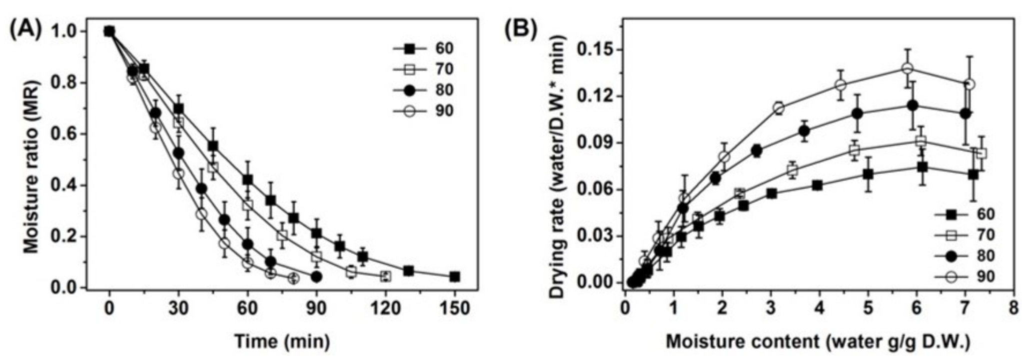

Figure 1. The MR (A) and DR (B) of Xuan-Mugua fruit slices dried under different temperatures.

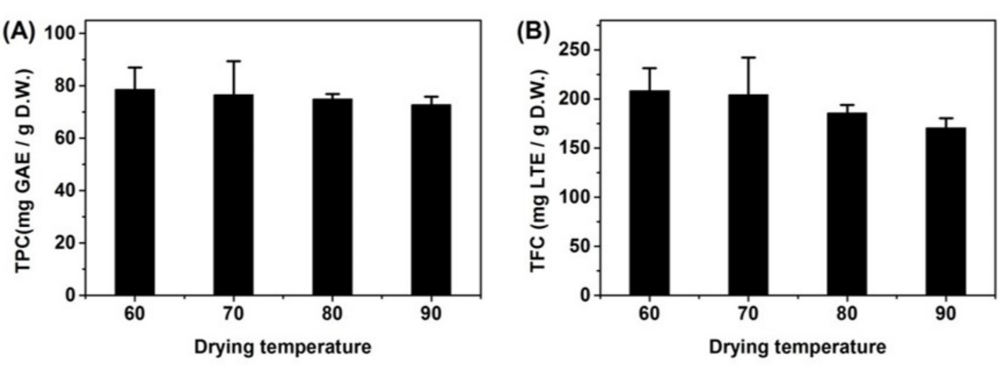

Figure 2. The TPC (A) and TFC (B) of Xuan-Mugua fruit slices dried under different temperatures.

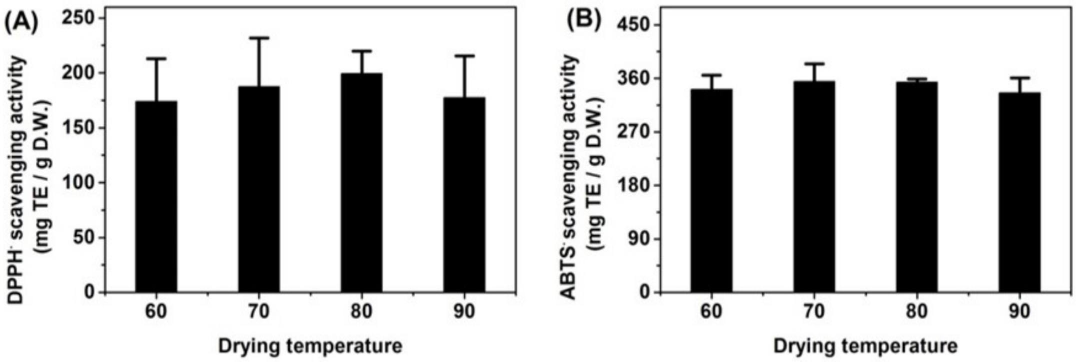

Figure 3. The DPPH (A) and ABTS (B) radicals scavenging activity of Xuan-Mugua fruit slices dried under different temperatures.

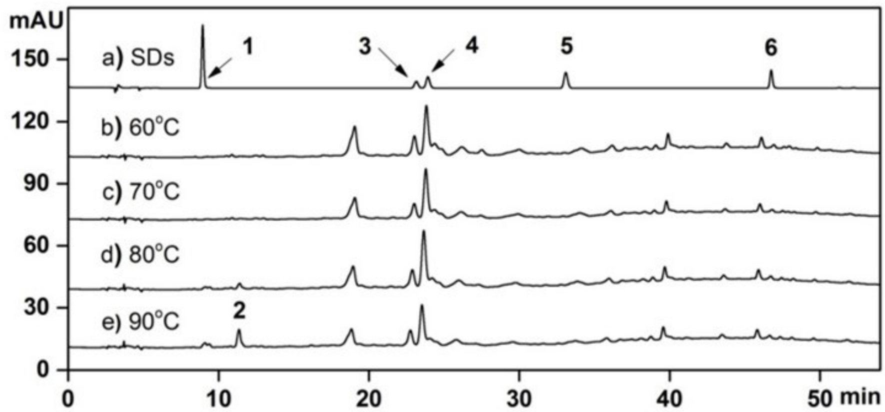

Figure 4. HPLC profiles of mixed standards (a) and Xuan-Mugua fruit slices dried under different temperatures (b-e). The standards are: 1-gallic acid, 3-catechin, 4- chlorogenic acid, 5-rutin, 6- quercetin.

whereas higher temperature of 90°C led to a decrease of the activity to 177.56 ± 37.79 (mg TE per 1 g D.W.). Similar to the change of DPPH radical scavenging activity, the ABTS radical scavenging activity was ranged from 335.83 ± 24.58 to 354.58 ± 29.99 (mg TE per 1 g D.W.) during the drying. These results indicated that the higher temperature may lead to the oxidation of some antioxidants, resulting in the decrease of antioxidant activities, and similar findings were reported in lotus pollen (Song et al., 2020).

3.4 HPLC profile of dried samples

To explore the effect of drying temperature on the phenolic profile of Xuan-Mugua fruit slices, HPLC analysis was carried out to the dried samples (Figure 4). Generally, the types of phenolics displayed no significant difference under the drying temperatures of 60 and 70 °C, whereas a new peak (compound 2) appeared when the temperatures increased to 80 and 90 °C, indicating the possible occurrence of transformation or degradation of some phenolics, and similar results were also found in the drying process of other plant materials, including black rice (Lang et al., 2019) and Oolong tea (Dou et al., 2007).

To confirm the main compounds in the dried samples, HPLC-MS analysis was applied, and two of them were tentatively identified as catechin (compound 3) and chlorogenic acid (compound 4), which had been previously reported in Chaenomeles fruits (Miao et al., 2017). Regarding the newly appeared compound 2 at 80 and 90 °C, it had an MS spectrum of 341 ($[M-H]^-$), indicating that it may be the derivatives of caffeic acid including the dicaffeic acid or caffeic acid glycosides (Bravo et al., 2007; Torras-Claveria et al., 2007). Considering that chlorogenic acid (compound 4, also known as 3-*O*-caffeoylquinic acid) was the important source of caffeic acid and had decreased under the drying temperatures of 80 and 90 °C, therefore, compound 2 may be the transformation product of compound 4.

3.5 Content of main phenolics in dried samples

Beside the effect of drying temperature on the phenolic profile of Xuan-Mugua fruit slices, its effect on the two identified phenolics (catechin and chlorogenic acid) was determined. The content of these two compounds generally decreased with the increase in drying temperature. As shown in Figure 5A, with the increasing of drying temperature, the content of catechin almost showed a constant decrease, and the maximal and minimal contents were respectively found at 60 ^{o}C (2.49 ± 0.02 mg per 1 g D.W.)

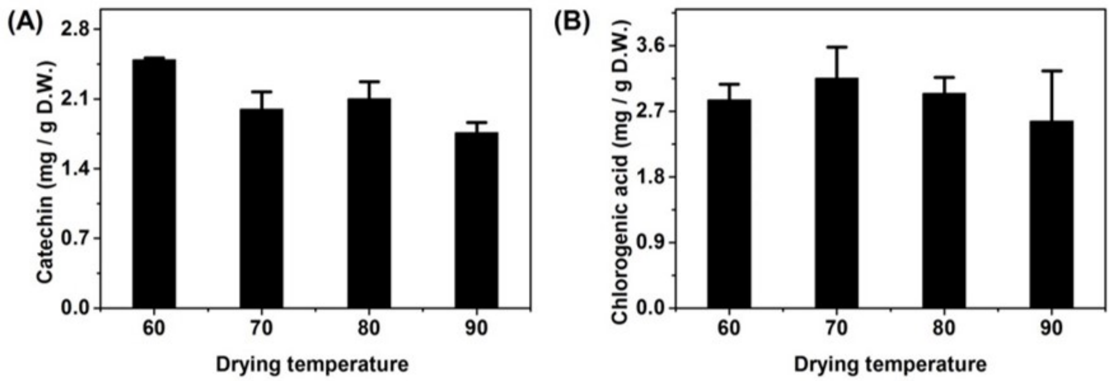

Figure 5. The content of catechin (A) and chlogenic acid (B) in Xuan-Mugua fruit slices dried under different temperatures.

and 90 °C (1.76 ± 0.10 mg per 1 g D.W.). But the change of chlorogenic acid was different from that of catechin. As it is indicated in Figure 5B, the content of chlorogenic acid showed a small increase when the drying temperature increased from 60 to 70 °C, and the maximal and minimal contents were respectively found at 70 °C (3.15 ± 0.42 mg per 1 g D.W.) and 90 °C (2.57 ± 0.69 mg per 1 g D.W.). These results indicated that the content of individual phenolics generally decreased with the increase of drying temperature, but this effect varied with the type of phenolics, which was similar to the findings reported in citrus seed (Al Juhaimi et al., 2018).

4 Conclusion

In this study, the Xuan-Mugua fruit slices were dried under different temperatures (60-90 °C). The moisture ratio and drying rates of the samples were respectively decreased and accelerated with the increase in drying temperature. The content of total phenolics and the antioxidant activity of the samples generally decreased with the increasing drying temperatures, but the content of the individual phenolics fluctuated with the drying temperature, and an obvious transformation of chlorogenic acid was found at higher temperatures (80-90 °C). Considering the phenolic profile and content, as well as the antioxidant activity, the suitable drying temperature for Xuan-Mugua fruit slices was 70 °C.

Conflict of interest

The authors declare that there are no conflicts of interest.

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