

Effect of Different Heat Treatment on Alkylresorcinol Contents of Wheat Bran

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The influence of different heat treatment levels on the contents of alkylresorcinol and homologues (ARs) was evaluated using wheat bran from two different wheat cultivars. The ARs in the wheat bran were destroyed by all heat treatments investigated in this study (oven treatment, microwave treatment, autoclave treatment, and extrusion treatment). The results showed that the loss rate of ARs by different heat treatment was oven treatment > autoclave treatment > extrusion treatment > microwave treatment, indicating that microwave treatment was more suitable for stabilizing wheat bran than the other three heat treatments. Both temperature and time of heat treatment had effects on the ARs of wheat bran. Higher temperatures and longer times of the treatment resulted in higher loss rates of ARs of wheat bran. The thermal stability of ARs homologues was different between wheat varieties, which may result from the variance of their structure and other components in wheat bran.

Keywords: Wheat bran; Alkylresorcinol; ARs; Heat treatment

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INTRODUCTION

Lipase and lipoxygenase in wheat bran can catalyze lipid hydrolysis or oxidation rancidity during storage, which reduces the storage stability of wheat bran. These changes can affect the function, organoleptic, and nutrition characteristics of wheat bran, which limits its application (Srivastava *et al.* 2007; Sudha *et al.* 2007; Rikard *et al.* 2008). Heat inactivation of the enzymes in wheat bran and germ is an effective way to improve the storage stability of whole wheat flour (Rose *et al.* 2008; Doblado Maldonado *et al.* 2012). Generally, whole wheat flours are prepared by combining the bran and germ with fine flour (Podio *et al.* 2019). Stabilization treatment of bran and germ before combining can improve the storage stability of whole wheat flour (Li *et al.* 2007; Jacobs *et al.* 2016).

Heat treatment results in the loss of some heat-sensitive nutrients, such as phenols, anthocyanins, and alkylresorcinol and homologues (ARs) (Randhir *et al.* 2008; Demir and Elgün 2014; Wang *et al.* 2019). ARs are important phenolic component in whole wheat flour, and the ARs content is a quality evaluation index of whole wheat flour (Kozubek and Tyman 1999; Al-Ruqaie, and Lorenz 1992). Some potential health effects, such as anticancer, antioxidants, antimicrobial, and antimutagenic, are related to the intake of ARs in animal and model systems (Landberg *et al.* 2006). However, there has been less research about the effect of heat treatment on the ARs in wheat bran. In the process of different heat treatments, to reduce the loss of ARs and improve the quality of wheat bran, the change of ARs content was studied. Because ARs are absent in the endosperm and exclusively deposited in the outer layers of wheat, wheat bran is used instead of whole wheat flour (Bordiga *et al.* 2016; Majewski, and Cunha 2018).

The present study investigated the variation of ARs content and homologues compositions in wheat bran by different heat treatments using various cultivars of two different wheat brans. The wheat brans of two different types were heated by physical treatment methods such as oven, microwave, autoclave, and extrusion. The ARs and homologues in bran were collected by ultrasound-assisted extraction.

EXPERIMENTAL

Materials and Chemicals

Bran 1 was provided by Cofco Zhengzhou Haijia Food Co. Ltd. (Zhengzhou, China), and bran 2, both untreated and extruded forms, was provided by Hebei Black Horse Flour Co. Ltd. (Shijiazhuang, China). The contents of each component in wheat bran are listed in Table 1. Ethyl acetate and acetic acid were analytical grade and purchased from Tianjin HengXing Chemical Reagent Co. Ltd. (Tianjin, China). Methanol (chromatographic grade), Fast Blue B Zn, and the authentic standards 5-pentadecylresorcinol (C15:0) were obtained from Sigma-Aldrich (Saint Louis, USA). Nitrogen was purchased from Zhengzhou Zezhong Technology Co. Ltd. (Zhengzhou, China).

Heat Treatments for Wheat Bran

Oven drying

A total of 60 g of each bran was placed on china plates (20 cm in diameter) to achieve a height of 2.5 cm and subjected to convective oven for 30 min at 90 °C, 110 °C, 130 °C, 150 °C, and 170 °C. A total of 60 g of each bran was placed on china plates and heated in an oven at 150 °C and 170 °C for 5 min, 10 min, 15 min, 20 min, 25 min, or 30 min.

Microwave treatment

A total of 150 g of each bran was placed on china plates to achieve a height of 2.5 cm. Bran specimens under the same conditions (microwave irradiation power 700 W, oscillation frequency of 2450 Hz) were treated for 30 s, 90 s, 150 s, 210 s, or 270 s, and the temperature of treated brans was measured.

Autoclave treatment

A total of 150 g of each bran was placed on china plates to achieve a height of 2.5 cm. Brans were steamed at the different temperatures (110 °C, 120 °C, 130 °C) for 5 min, 10 min, 15 min, 20 min, or 25 min.

Extrusion treatment

A DS32-II twin-screw extrusion tester (Saixin Puffing Machinery Co., Ltd. Jinan, China) was used for experiments. The temperature in zone I was 40 °C, zone II 140 °C, and zone III 160 °C. The prepared bran 2 were extruded under the condition of main machine frequency 15 Hz, feeding frequency 10 Hz, and rotary cutting frequency 60 Hz.

Alkylresorcinol Analyses

Extraction of ARs in bran

Ultrasound-assisted extraction of ARs was performed using a Scientz-IID ultrasonic cell disruptor (Ningbo, China), containing a $\Phi 6$ amplitude transformer. An aliquot of 0.8 g of whole wheat flour and 40 mL of ethyl acetate were sonicated for 2 min at 286 W (turn ultrasonic cell disruptor for one second, the effective ultrasonic time within 1 min is 30 s). To avoid the loss of solvent during heating, samples were cooled in an ice bath during ultrasonication. The extract was centrifuged for 10 min at 3000 r/min. After 5 mL of obtained suspension was evaporation by nitrogen, the dried extract was dissolved in 1 mL of methanol and filtered through a 0.22 μm membrane. All extract solutions were freshly prepared daily prior to use.

Determination of ARs in bran

A stock solution of 0.05% (w/v) Fast Blue B salt was prepared in methanol containing 1% (v/v) acetic acid (Gajda *et al.* 2008). A fresh working solution of Fast Blue B reagent was prepared by mixing 1 part stock reagent with 5 parts methanol. Stock solutions of pure olivetol were prepared in methanol at a concentration of 1 mg/mL. Aliquots of this solution between 1 and 10 μL (1 to 10 μL , or 6.3 to 62.9 nmol) were placed in assay tubes and made up to 200 μL with methanol. Then, 2 mL of working solution of Fast Blue B salt was added to each assay tube. Samples extracted from cereal grains were also assayed. The absorbance was measured after 60 min at 520 nm. Each experiment was done in triplicate.

Statistical Analysis

The results were expressed as mean \pm standard deviation (SD) of at least three independent experiments. Using SPSS 16.0 software (SPSS Inc., Chicago, IL, USA) for variance analysis, the Duncan test was selected to carry out significant and statistical analysis of data at the $P < 0.05$ level. Origin 9.0 software (Northampton, USA) was used for data visualization.

RESULTS AND DISCUSSION

Effect of Oven Treatment on ARs Contents

To ensure product quality, wheat bran must be stabilized before milling. Therefore, the effect of heat treatment on two kinds of bran ARs can reflect the ARs in different varieties of wheat bran. The results also reflect the effect of heat treatment on the ARs in different varieties of wheat bran.

Table 1. Contents of each Component in Wheat Bran (%)

Variety	Protein	Fat	Starch	Ash	Total dietary fibre
Bran1	17.66 \pm 0.38	4.16 \pm 0.03	7.90 \pm 0.50	17.58 \pm 0.91	45.31 \pm 1.12
Bran 2	17.81 \pm 0.09	2.89 \pm 0.01	14.03 \pm 0.84	24.74 \pm 0.37	38.09 \pm 0.87

Oven treatment had a strong influence on ARs content in wheat bran. The change of total ARs content in the bran is shown in Fig. 1. The oven treatment damaged the ARs. As the temperature of the oven increased, the total content of ARs in bran gradually

decreased, while the loss rate of ARs gradually increased. Initially, the ARs content of two different brans were $1802.7 \pm 7.4 \mu\text{g/g}$ and $1615.3 \pm 5.0 \mu\text{g/g}$. When the temperature was raised to 110°C , the content of ARs dropped significantly to $1723.2 \pm 31.8 \mu\text{g/g}$ and $1481.8 \pm 25.5 \mu\text{g/g}$ ($P < 0.05$). After the temperature rise to 130°C , the loss rate of ARs kept increasing, and the content of ARs in bran were significantly different when comparing the total content ($P < 0.05$). When the heat temperature was 170°C , the total content of ARs in the two kinds of bran decreased to $1275.3 \pm 0.0 \mu\text{g/g}$ and $953.8 \pm 0.0 \mu\text{g/g}$.

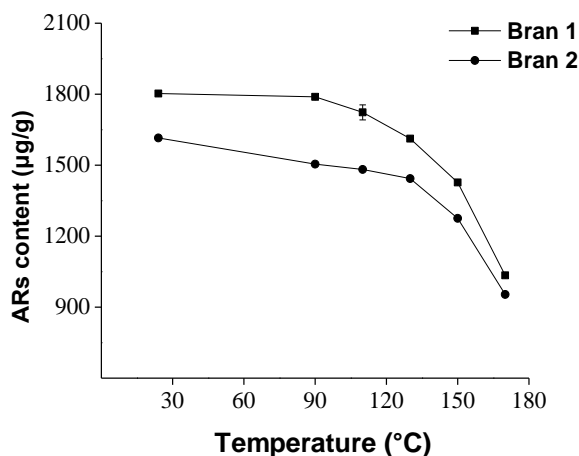


Fig. 1. Effect of oven treatment temperature on ARs content of wheat bran

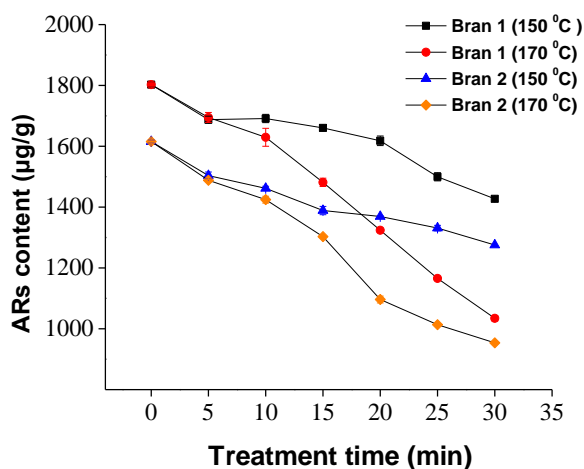


Fig. 2. Effect of oven treatment time on ARs content of wheat bran

To elucidate the relationship between ARs total content and heating time, two kinds of brans were treated at 150°C and 170°C for 5 min, 10 min, 15 min, 20 min, 25 min, and 30 min. The ARs total content in bran is shown in Fig. 2.

Figure 2 shows that ARs total contents in bran 1 and bran 2 were significantly decreased after 5 min at 150°C and 170°C in the oven ($P < 0.05$). The ARs total content in bran 1 and bran 2 were $1802.7 \pm 7.4 \mu\text{g/g}$ and $1615.3 \pm 5.0 \mu\text{g/g}$, while heated bran1 and bran2 at 150°C contained $1687.4 \pm 11.7 \mu\text{g/g}$ and $1503.6 \pm 11.7 \mu\text{g/g}$. When bran 1 and bran 2 were heated at 170°C for 5 min, the ARs total content was reduced to 1694.8

$\pm 16.3 \mu\text{g/g}$ and $1488.1 \pm 2.3 \mu\text{g/g}$, respectively. Heating time had a significant effect on the total content of ARs in the bran within 0 to 30 min ($P < 0.05$), which showed a linear reduction trend. The ARs total content and heating time in bran 1 at 150°C was as follows,

$$\text{ARs} = -11.25 \times \text{treatment time} + 1795.4 \quad (R^2 = 0.9188) \quad (1)$$

The ARs total content and heating time in bran 1 at 170°C was as follows,

$$\text{ARs} = -26.21 \times \text{treatment time} + 1840.6 \quad (R^2 = 0.9856) \quad (2)$$

The ARs total content and heating time in bran 2 at 150°C was as follows,

$$\text{ARs} = -10.413 \times \text{treatment time} + 1576.9 \quad (R^2 = 0.9543) \quad (3)$$

The ARs total content and heating time in bran 2 at 170°C was as follows,

$$\text{ARs} = -23.299 \times \text{treatment time} + 1620.1 \quad (R^2 = 0.9798) \quad (4)$$

Oven treatment in certain conditions destroyed the ARs in wheat bran. This result was similar to the impact of heat treatment on ARs total content in mango peel (Christian *et al.* 2015). During the drying process, the ARs were more stable at lower temperature. As the treatment temperature was increased, the stability and the total content of ARs decreased. The extension of treatment time could accelerate the loss of ARs. The reduced ARs contents of brans after convective oven heating might be due to aeration. The circulating gas in the oven provided sufficient oxygen for the oxidation reaction of ARs. In addition, the thermal ARs degradation during oven heating might also contribute to ARs losses (Randhir *et al.* 2008; Geerkens *et al.* 2015). Therefore, it should avoid high temperature and long treatment time to reduce the loss of ARs in the process of oven treatment.

Effect of Microwave Treatment on ARs Contents

Microwave processing has the characteristics of time-saving, high efficiency, and less nutrition loss, but it results in changes in the quality of lipids. Because ARs are commonly phenolic lipids, microwave treatment may cause the content to change (Vadivambal and Jayas 2007). To study the effect of microwave treatment on ARs in wheat bran, the brans were treated by microwave for 30 s, 90 s, 150 s, 210 s, and 270 s. The ARs total content and temperature of bran 1 and bran 2 are shown in Fig. 3.

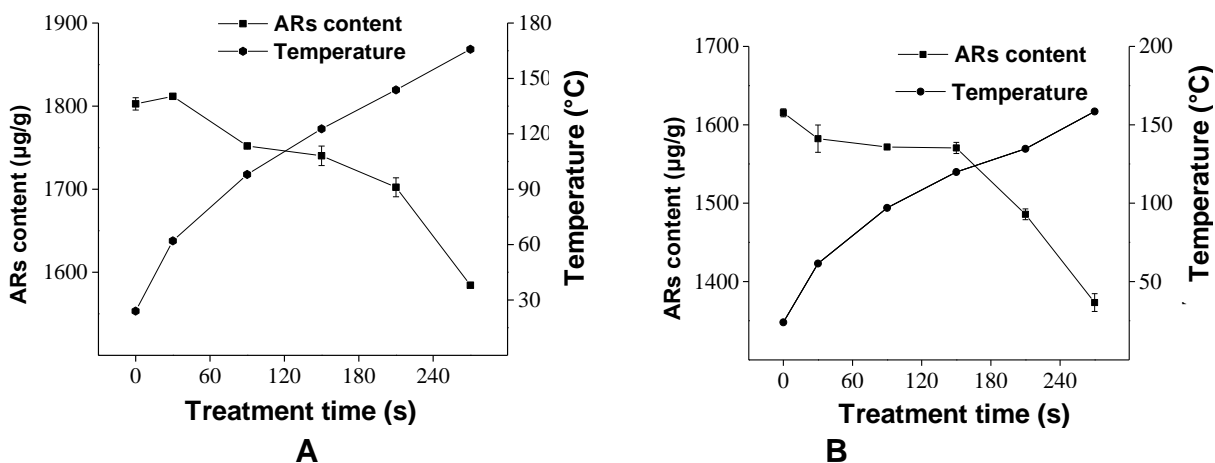


Fig. 3. Effect of microwave treatment on ARs content of wheat bran (A: bran1; B: bran 2)

As shown in Fig. 3, the bran temperature increased and the total content of ARs in the bran decreased gradually with the increase of microwave treatment time. After two kinds of bran were treated by microwave for 270 s, the loss rate of ARs was $12.1 \pm 0.1\%$ and $15.0 \pm 0.7\%$, and the bran temperature were as high as $165.8 \text{ }^\circ\text{C}$ and $165.8 \text{ }^\circ\text{C}$, respectively.

These results showed that the longtime microwave treatment reduced the ARs total content in wheat bran. Because the bran temperature was increased by microwave treatment, the reduced ARs content might be due to the oxidation, thermal degradation, or polymerization of ARs during processing (Geerkens *et al.* 2015). It may also relate to the non-thermal effect of microwave, such as magnetic field effect. Microwave induces the production of free radicals, which can lead to different chemical reactions and cause changes in oil quality (Hoogenboom 2009). The ARs of bran is likely to be changed in the process of scavenging free radicals. Therefore, long-time microwave heating of bran should be avoided to reduce damage in ARs.

Effect of Autoclave Treatment on ARs Contents

The changes of ARs total content in the bran during autoclave treatment are shown in Fig. 4. Under the different temperatures, the total ARs contents in two kinds of brans decreased gradually with the extension of treatment time. The loss rate of ARs in two kinds of bran were similar and less affected by the initial content. When the temperature was set to $110 \text{ }^\circ\text{C}$, $120 \text{ }^\circ\text{C}$, and $130 \text{ }^\circ\text{C}$, the ARs total content in bran were significantly lower than the control after 5 min ($P < 0.05$). After 10 min, the total contents of ARs in bran tended to be steady as the treatment time increased further. In autoclave treatment process, brans had a high water content, and water was a good heat transfer medium, which promotes the catalytic reaction of ARs by enzymes and accelerated the damage of ARs by heat treatment. With the further increase of brans temperature, the enzyme activity decreased, and the change of the total ARs content was steady. In addition, the total content of ARs in the bran was affected by the treatment temperature. At the treatment condition of $130 \text{ }^\circ\text{C}$ and 25 min, the ARs loss rate of the two kinds of bran were $27.3 \pm 0.1\%$ and $34.1 \pm 0.8\%$.

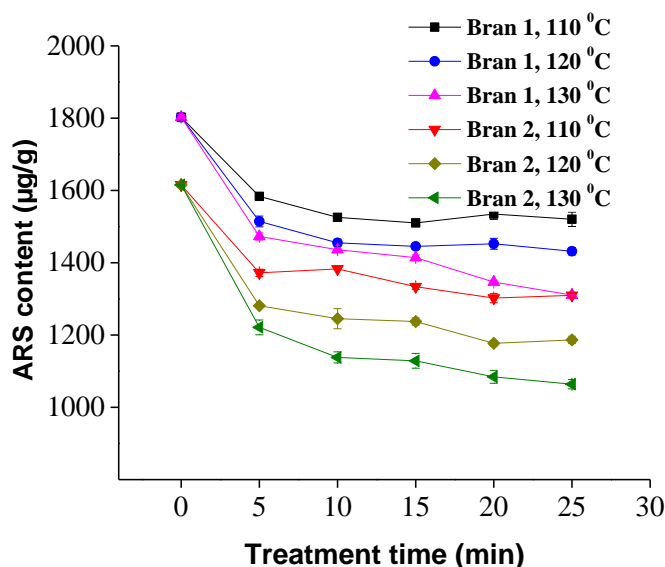


Fig. 4. Effect of autoclaving on ARs content of wheat bran

According to the above results, autoclave treatment can destroy ARs in wheat bran, resulting in a decrease in its content, and this change mainly occurs in the first 10 min. The total content of ARs was affected by both heating temperature and time. A higher temperature resulted in a lower total amount of ARs. Under the same temperature, a longer processing time resulted in a lower total content of ARs, until it tends to be stable.

Comparison of the Effects of Different Heat Treatment on ARs Contents

Figure 5 compares the total amount of the ARs after oven processing (under the condition of 170 °C for 30 min), microwave treatment (270 s), autoclave treatment (under the condition of 130 °C 25 min), and bran extrusion processing.

After the four kinds of heat treatment, the total ARs content of both bran decreased significantly ($P < 0.05$), suggesting that the oven treatment, microwave treatment, autoclave treatment, and extrusion treatment damage the ARs in bran (Fig. 5). Under the same heating conditions, two brans displayed a small difference in the ARs loss rate, which may be due to the difference in the composition and structure of ARs homologues in different bran (Gunenc *et al.* 2013). In addition, some material in the bran, such as lipase or antioxidant activity components, may have impacted the ARs during the treatment and cause differences in ARs loss rate in different bran varieties.

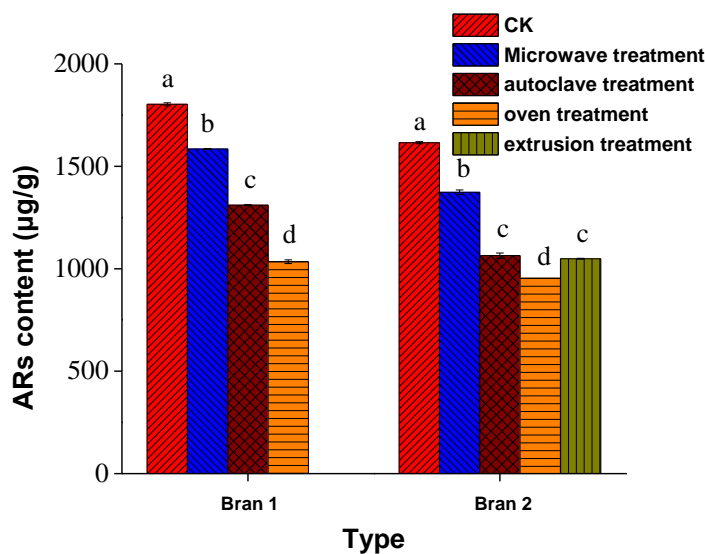


Fig. 5. Effect of heat treatments on ARs content of bran from different wheat varieties. The bran treated by different treatment methods is marked with different lowercase letters, indicating that the difference between different treatment methods is significant ($P < 0.05$).

The variation in ARs damage induced by different heat treatments may be related to the effect of different heating patterns. During oven processing, the bran is mainly affected by high temperature. In autoclave treatment, the bran is subjected to high temperature and high pressure, and the moisture content in the sample is high. During extrusion, the bran is subjected to high temperature, high pressure, and shear force (Rose *et al.* 2008; Oladiran *et al.* 2018). For both bran types, oven treatment led to the biggest reduction in ARs content, followed by autoclave and microwave. Therefore, microwave treatment is more suitable for stabilizing wheat bran due to less ARs loss.

CONCLUSIONS

1. Oven treatment, microwave treatment, autoclave treatment, and extrusion treatment have destructive effects on ARs in wheat bran to different degrees. The effect of heat treatments on ARs was oven treatment > autoclave treatment, extrusion treatment > microwave treatment, indicating that microwave treatment was more suitable for stabilizing wheat bran than the other three heat treatments.
2. The ARs were affected by heat treatment time and temperature. A higher temperature and longer processing time resulted in higher loss rates of ARs.
3. The effect of heat treatment on ARs in different varieties of wheat bran was different, which may be caused by the differences in the composition and structure of ARs homologues and the influence of other substances in the bran.

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CONFLICT of INTEREST

The authors declare that there is no conflict of interest.

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