



## Research on variation of volatile compounds of *Cinnamomum cassia* Presl in different processing stage of stewed beef

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### Abstract

Spices play an important role in the formation of stewed meat flavor, and it is necessary to establish a rapid detection technology for flavor in stewed meat production. The aim of this work was to discover the variation of volatiles from *Cinnamomum cassia* Presl in different processing processes and to establish a real-time detection method during processing. The relative content of volatile components of *Cinnamomum cassia* Presl from powder to extract and from extract to beef was compared in different processes. The volatile compounds were analyzed by headspace sampling and gas chromatography-mass spectrometry (HS-GC-MS). A total of 20 components were detected in the cinnamon powder. In the marinade processing, the most volatile components was detected in marinade when marinating for 1 hour, such as cinnamic acid(4.39%), cinnamaldehyde (46.57%), anethole(8.05%),  $\alpha$ -pinene(7.38%), naphthalene,1,2,4a,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-(0.93%),  $\alpha$ -muurolene(8.99%), naphthalene,1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-,(1S-cis)-(20.31%), naphthalene,1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)-(3.03%) and  $\alpha$ -calacorene(0.35%). In the bitter processing, the flavor components in the bitter when cooking for 15 minutes were the most and 7 components were detected which were cinnamic acid(3.69%), cinnamaldehyde(70.67%), anethole(4.37%),  $\alpha$ -pinene(1.62%),  $\alpha$ -muurolene(3.69%), naphthalene,1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-,(1S-cis)-(14.17%) and naphthalene,1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)-(1.79%). The relative content of the volatile components in two bitter were slightly different. In two processes, only cinnamaldehyde was detected in the stewed beef, where a small amount of  $\alpha$ -pinene was detected when marinating for 1 hour and bittering for 45 minutes. Cinnamaldehyde can be used as the main indicator for the detection of processing. HS-GC-MS can be used as a real-time detection method in the marinade and bitter processing.

**Keywords:** HS-GC-MS; volatile compounds; stewed beef; variation.

**Practical Application:** In this experiment, HS-GC-MS was used to detect the relative content of volatile components in cinnamomum cassia presl during different processing. The relative content of volatile components of cinnamomum cassia presl from powder to extract and from extract to beef was compared in different processes. This study was designed to discover the variation of volatiles from cinnamomum cassia presl in different processing processes and to establish a real-time detection method during processing.

### 1 Introduction

As a traditional cooked meat product, stewed meat product is favored by most consumers in China by its beautiful color, special flavor and moderate palate. Due to China's vast territory and diverse ethnic characteristics, Chinese ethnic meat products have gradually developed various processing techniques and enjoyable flavors (Wang et al., 2013). In recent years, people paid more attention to the industrialization of traditional meat products, it has also put forward higher requirements to the safety and nutrition of traditional meat products. Many scholars devoted themselves to the theory of processing and the research of new techniques of stewed meat (Zeng et al., 2016). The processing technology of stewed meat products mainly including marinate and bitter processing. Traditional stewed products have many problems in processing, innovation and technology (Li et al., 2011). The main problems of stewed meat that aren't suitable for industrialization and standardized

production are unstable processing quality, poor equipment and short shelf life of products (Lu et al., 2010). Therefore, it is urgent to carry out standardization studying on stewed meat products. Spices are the important materials in the processing of meat products. The flavor and stability of the spices determined the flavor of stewed meat products. Carry out the variation of volatile compounds of spices in different processing stages was necessary in product standardization processing. HPLC method was used to detect the main components of common spices and the main components were studied (Ai et al., 2018a). In addition to regulating the flavor of meat products, spices can also act as natural preservatives (Kittisakulnam et al., 2017; Radha Krishnan et al., 2014a; Zhang et al., 2016; Chen et al., 2017), which have a protective effect on meat products and extend their shelf life (Hernández-Ochoa et al., 2014).

Received 03 Jan., 2022

Accepted 16 Feb., 2022

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According to GB/T21725-2017, Strong-flavor spices are the main taste factors which include cloves, star anise, cinnamon and nutmeg so on. The electronic nose technology was used to detect the distilling soup prepared by 12 kinds of edible spices and the effect of the overall flavor was analyzed. The order of the overall flavor affecting factor of the strong-flavor was cinnamon > nutmeg > octagon (Wang et al., 2017). Therefore, *Cinnamomum cassia* Presl (cinnamon) was chosen as the research object in this study. In related research, cinnamon extracts not only has the ability to change the flavor of stewed meat (Kim et al., 2013) and extensive antibacterial activities (Evrendilek, 2015), but also effective in inhibiting the growth of microbial populations and retarding lipid oxidation during refrigerated storage (4 °C) of raw beef meat (Radha Krishnan et al., 2014b; Han et al., 2014). There are also specific studies on the types and identification of volatile components in cinnamon (Wang et al., 2014).

The headspace injection method is a gas chromatography-specific injection method suitable for the analysis of volatile components, which eliminates the pretreatment of the sample and avoids contamination of the instrument system by non-volatile components in the sample. Headspace gas chromatography-mass spectrometry (HS-GC-MS) technology becomes more mature and its application in the testing industry is also diverse. It is commonly used for the detection of volatile compounds in certain substances, such as the analysis of volatile compounds in onions (Colina-Coca et al., 2013), red and white wines (Zhang & Guo, 2017) and hilsa (Ganguly et al., 2017). The advantage of headspace sampling technology is that it doesn't require complex sample preparation, and it combines with GC-MS to quickly and easily detect volatile compounds (Jia & Wang, 2014; Xie et al., 2013). Some studies have found that soybean is a raw material or precursor that is easy to form furan during hot processing of food by HS-GC-MS (Nie et al., 2013). HS-GC-MS was also applied directly to olive and olive oil to determine the single aromatic volatile BTEXS group (Gilbert-López et al., 2010).

In this study, headspace gas chromatography-mass spectrometry (HS-GC-MS) was used to identify the volatile compounds by comparing GC retention time with those of standard compounds and by NIST 11 standard mass spectrometry library. The data representing regularity were analyzed using Excel (Microsoft Office 2010). This study compared the relative contents of volatile compounds of cinnamon in marinate and bitter process of stewed beef, in order to determine the variation of cinnamon flavor in the processing.

## 2 Materials and methods

### 2.1 Pretreatment of raw materials

*Cinnamomum cassia* presl was purchased at the spice wholesale market in Wuhan. The cinnamon samples were air dried in an oven (DGG-9240A, Shanghai Linpin Experiment Instrument Co., Shanghai, China) at 35 °C for 8h. Then they were subjected to pulverization by a cutting mill (DFT-50, Lingda Mechanics Co, Zhejiang, China). They were continuously pulverized twice and sieved through a 60-mesh sieve.

Beef was bought at the Wushang Supermarket Chains Company. Took the same batch of beef and cut into pieces (20 mm × 20 mm × 10 mm) and stored at low temperature (4 °C).

### 2.2 Sample preparation for the marinade process

Weigh 0.2 g of cinnamon powder accurately and put them into a test tube with stopper. Then added 60 mL of distilled water according to the ratio of 1:300 and marinated respectively for 15 minutes, 30 minutes, 1 hour, 2 hours, 4 hours, 6 hours, 12 hours and 24 hours at 4 °C. When they were finished, marinated extracts of different time periods were obtained by vacuum filtration.

Weighed 0.5 g of cinnamon powder accurately and put them into a test tube. Add 50 mL of distilled water at a faint boiling water (95 °C) for cooking 15 minutes. Took the beef cubes in a clean test tube with stopper and seal with sealing film. The extracting solution was added to meat in a ratio of 2:1 and marinated the beef at 4 °C for 30 minutes, 1 hour, 2 hours, 4 hours, 6 hours, 12 hours and 24 hours respectively.

The entire experiment was done in a sealed environment.

### 2.3 Sample preparation for the bitter process

Weigh 0.2 g of cinnamon powder accurately and put them into a test tube with stopper. Added 60 mL of distilled water (95 °C) respectively and seal them with sealing film for cooking 5 minutes, 10 minutes, 15 minutes, 30 minutes, 45 minutes, 60 minutes and 90 minutes. When they were finished, cooked extracts of different periods were obtained by vacuum filtration.

Weighed 0.5 g of cinnamon powder accurately and put them into a test tube. Add 50 mL of distilled water (95 °C) for cooking 15 minutes. Took the beef cubes in a clean test tube with stopper and seal with sealing film. The extracting solution was added to meat in a ratio of 2:1 and cooked the beef at a faint boiling water (95 °C) for 15 minutes, 30 minutes, 45 minutes, 60 minutes and 90 minutes respectively.

The entire experiment was done in a sealed environment.

### 2.4 Headspace volatile sampling

From cinnamon powder to extract: took 2 g of cinnamon powder and 7 mL liquid of different treatments to headspace bottles respectively and the air was used as blank sample. The volatile components were separated by HS-GC-MS.

From extract to beef: took 3 g of beef (shredded) in the headspace bottle respectively and the untreated beef was used as blank sample. The volatile components were separated by HS-GC-MS.

### 2.5 Analysis of volatiles by GC-MS

The analysis of all samples was performed on Agilent 7890A GC, coupled to a mass spectrometer Agilent 5975C single quadrupole mass spectrometer and equipped with a G1888 static headspace sampler (Agilent Technologies, Palo Alto, USA). The samples were contained in 10 mL headspace bottle, which were

placed in the auto sampler oven to be heated and agitated in order to generate the headspace. The incubation temperature was maintained at 60 °C with an equilibration time of 30 min. 1000 µL of the samples was injected automatically in split less mode. The components were separated on a HP-5 capillary column (30 m, 0.25 mm i.d, 0.25 µm film thickness). The carrier gas was helium, at a flow rate of 0.8 mL/min and septum purge flow rate of 3 mL/min. Column temperature was initially 50 °C, then gradually increased to 130 °C at 8°C/min, and finally increased to 180 °C at 5 °C/min for 10min.

For GC-MS detection, it used an election ionization system. The ion temperature is 230°C and the quadrupole temperature is 150 °C. The mass spectrometer was operated in electron impact (EI) mode at 70eV. Temperatures of the ion source, the quadrupole, and the interface were set at 230,150 and 280 °C, respectively. The Mass scanning range is between 45~450U and the scanning speed is set in 0.20s/scan. Each compound was identified using the Wiley/NBS, NIST, and other published mass spectral libraries of GC-MS data. The selection of GC-MS conditions was based on the conditions of use of the relevant studies (Tung et al., 2010; Li et al., 2013) and was appropriately adjusted in conjunction with the results of the pre-experiments.

## 2.6 Statistical analysis

After GC-MS detection method, identification of all volatile compounds was carried out by comparing GC retention time with those of standard compounds, and by NIST 11 standard mass spectrometry library. The data representing regularity were analyzed using Excel (Microsoft Office 2010).

## 3 Results and discussion

### 3.1 Volatile components of the cinnamon powder

In this work, volatile components of *Cinnamomum cassia presl* powder were extracted using headspace method and then analysis by GC-MS. Peak area normalization method was adopted to determine the relative content of each component. Most of the compounds in the powder were separated and identified, and some of them had not been found before. About 4-6 hours is required for traditional methods of extraction, whereas only 30 minutes was needed for the headspace method. This method is suitable for the extraction of volatile compounds from *Cinnamomum cassia presl*. The detection sensitivity of the method is high and the detection speed is fast. It can detect more active ingredients without complicated pretreatment (Chen et al., 2015; Peris & Blázquez, 2014).

The chemical compositions of the *Cinnamomum cassia presl* powder are presented in Table 1. Twenty volatile compounds were detected in the cinnamon powder which contained one acid, one alcohol, two aldehydes, thirteen alkene, four naphthalenes and five unidentified components. There were 16 components of the matching rate was above 85% in the 20 compounds. The identification results are shown in Table 1.

As can be seen from Table 1, there are 7 components with a relative content of 1% or more. The major components in *Cinnamomum cassia presl* powder were cinnamaldehyde (55.27%), anethole(1.09%),

α-pinene(18.9%), naphthalene,1,2,4a,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-(2.11%), α-murolene(6.96%), naphthalene,1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-,(1S-cis)-(9.92%) and naphthalene,1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)-(1.49%). The number of volatile compounds detected was less than that of the open literature, and fewer types of fatty acids are detected (Wang et al., 2014).

The volatile components of cinnamon were mostly small molecular compounds of 15 carbons or less. It is mainly composed by aromatic aldehydes, aromatic lipids and moss. Cinnamaldehyde is the main active ingredient in the volatile matter of cinnamon, which has good anti-bacterial, anti-diabetic and anti-neuritis effects (Shen et al., 2015; Guo et al., 2017; Ho et al., 2013). According to related literature, different origins, planting techniques and storage time have effect on the active ingredients of cinnamon (Li et al., 2013). Therefore, the composition analysis described in this experiment cannot cover all cinnamon varieties.

### 3.2 The marinade processing

It can be seen from Table 2 that there were nine volatile compounds that were detected in different time of marinated processing. There were one acid, one aldehydes, four alkene and three naphthalenes, which were cinnamic acid, cinnamaldehyde, anethole, α-pinene, naphthalene,1,2,4a,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-, α-murolene, naphthalene,1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-,(1S-cis)-, naphthalene,1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)- and α-calacorene. The substances with a relative content of 1% or more in Table 1 were detected. Nevert heless,naphthalene,1,2,4a,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)- and α-calacorene were detected when marinade for 1 hour, while Naphthalene,1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)- was not detected when marinade for 6 hours and 24 hours. These major volatile compounds that were detected were consistent with the volatile substances of cinnamon powder (Tung et al., 2010).

Six components were detected in eight marinating times, such as cinnamic acid, cinnamaldehyde, anethole, α-pinene, α-murolene and naphthalene,1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-,(1S-cis)-. The relative content of volatile components measured by the eight marinated times in Figure 1 shows that the relative of cinnamic acid has a significant difference when marinated for 15 minutes and 24 hours. The relative content of cinnamaldehyde that marinated for 1 hour is reduced. There was no significant change in the relative content of anethole at different marinating times. However, the relative content of α-pinene, α-murolene and naphthalene,1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-,(1S-cis)- were obviously increase when marinated for 1 hour. Compared with the volatile components of cinnamon powder, the marinade liquid which marinated for 1 hour has the most abundant flavor.

In combination with Table 2 and Figure 1, the species and content of flavor substances detected in marinade liquid at different marinating times were obviously different. The most volatile compounds were detected when marinating for 1 hour

**Table 1.** Volatile components of the cinnamon powder.

Retention time(min)	Component	CAS number	Molecular formula	Molecular weight	Relative amount (%)
2.585	Cinnamic acid	000621-82-9	C <sub>9</sub> H <sub>8</sub> O <sub>2</sub>	148.159	0.21
10.197	3-Phenyl-2-propyn-1-ol	001504-58-1	C <sub>9</sub> H <sub>8</sub> O	132.159	0.19
11.142	Cinnamaldehyde,(E)-	014371-10-9	C <sub>9</sub> H <sub>8</sub> O	132.159	55.27
11.36	Anethole	000104-46-1	C <sub>10</sub> H <sub>12</sub> O	148.202	1.09
12.395	Cedrene	011028-42-5	C <sub>15</sub> H <sub>24</sub>	204.351	0.059
12.986	1,2,4-Metheno-1H-indene, octahydro-1,7a-dimethyl-5-(1-methylethyl)-, [1S-(1.alpha.,2.alpha.,3a.beta.,4.alpha.,5.alpha.,7a.beta.,8S*)]-	022469-52-9	C <sub>15</sub> H <sub>24</sub>	204.351	0.61
13.167	α-Pinene	003856-25-5	C <sub>15</sub> H <sub>24</sub>	204.351	18.9
13.458	Aromandendrene	000489-39-4	C <sub>15</sub> H <sub>24</sub>	204.351	0.49
13.594	1,4-Methano-1H-indene,octahydro-4-methyl-8-methylene-7-(1-methylethyl)-,[1S-(1.alpha.,3a.beta.,4.alpha.,7.alpha.,7a.beta.)]	003650-28-0	C <sub>15</sub> H <sub>24</sub>	204.351	0.60
13.894	Isosativene	024959-83-9	C <sub>15</sub> H <sub>24</sub>	204.351	0.23
14.066	Caryophyllene	000087-44-5	C <sub>15</sub> H <sub>24</sub>	204.351	0.37
14.484	1H-3a,7-Methanozulenene,2,3,4,7,8,8a-hexahydro-3,6,8,8-tetramethyl-,[3R-(3.alpha.,3a.beta.,7.beta.,8a.alpha.)]-	000469-61-4	C <sub>15</sub> H <sub>24</sub>	204.351	0.05
14.757	Humulene	006753-98-6	C <sub>15</sub> H <sub>24</sub>	204.351	0.36
15.175	Naphthalene,1,2,4a,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-	031983-22-9	C <sub>15</sub> H <sub>24</sub>	204.351	2.11
15.429	Naphthalene,1,2,3,5,6,7,8,8a-octahydro-1,8a-dimethyl-7-(1-methylethenyl)-,[1S-(1.alpha.,7.alpha.,8a.alpha.)]-				0.17
15.656	α-Muurolole	017627-24-6	C <sub>15</sub> H <sub>24</sub>	204.351	6.96
15.965	g-Muurolole	000483-74-9	C <sub>15</sub> H <sub>24</sub>	204.351	0.25
16.137	Naphthalene,1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-, (1S-cis)-	000483-76-1	C <sub>15</sub> H <sub>24</sub>	204.351	9.92
16.337	Naphthalene,1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)-	016728-99-7	C <sub>15</sub> H <sub>24</sub>	204.351	1.49
16.573	α-Calacorene	021391-99-1	C <sub>15</sub> H <sub>20</sub>	200.319	0.21

**Table 2.** The relative contents of main components of cinnamon from cinnamon powder to marinated extract.

Component	15min	30min	1h	2h	4h	6h	12h	24h
Cinnamic acid	0.84%	9.77%	4.39%	9.10%	10.07%	7.42%	10.49%	17.91%
Cinnamaldehyde	74.93%	69.31%	46.57%	69.70%	72.78%	76.85%	72.84%	70.21%
α-Anethole	12.68%	11.54%	8.05%	12.63%	12.01%	12.64%	12.36%	8.96%
α-Pinene	3.31%	1.97%	7.38%	1.39%	0.71%	0.66%	0.82%	0.65%
Naphthalene,1,2,4a,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-	-	-	0.93%	-	-	-	-	-
α-Muurolole	1.99%	1.70%	8.99%	1.48%	0.75%	0.57%	0.64%	0.52%
Naphthalene,1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-, (1S-cis)-	5.53%	5.08%	20.31%	5.01%	3.15%	1.86%	2.37%	1.75%
Naphthalene,1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)-	0.72%	0.63%	3.03%	0.69%	0.53%	-	0.48%	-
α-Calacorene	-	-	0.35%	-	-	-	-	-

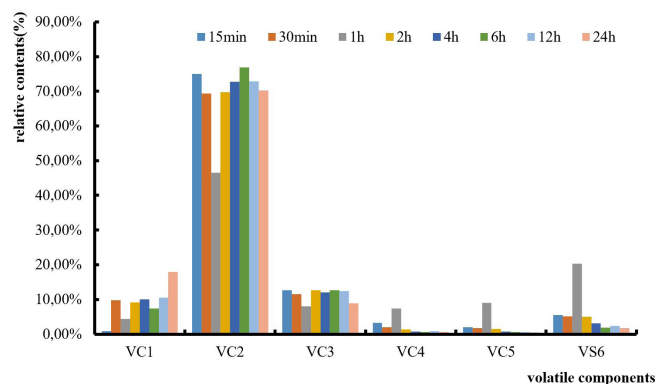
and the proportion of the components closest to the cinnamon powder, which is the most suitable time for making the marinade.

In order to ensure that the starting conditions of the stewed beef are the same except the different processing, and to obtain the extract of cinnamon quickly, the bittern was chosen to cooking for 15 minutes to treat the beef (Ai et al., 2018b). Then compare

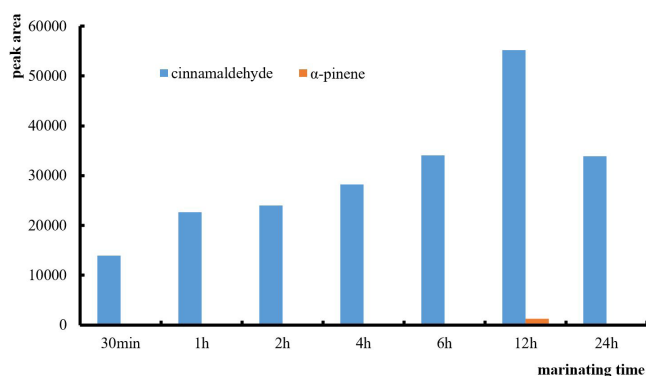
the relative content of volatile components of cinnamon from extract to beef during the different marinated times.

Since the active ingredient of cinnamon detected in beef is extremely small, the practical significance can't be reflected by the relative percentage of each component by the area normalization method for determining, therefore the peak area is used for

explanation. In the processing of marinade, cinnamaldehyde was detected in the beef during different marinating time. As can be seen from the figure 2, the content of cinnamaldehyde rose first and then declined throughout the marinating process. As far as this figure is concerned, the content of cinnamaldehyde reached the maximum when marinated for 12 hours. And another volatile substance  $\alpha$ -pinene was detected at this moment which is consistent with the volatile components of cinnamon powder. Compared with marinade processing of chicken, the



**Figure 1.** Trend diagram of relative content of volatile components in cinnamon with different marinating time. Notes: VC1, Cinnamic acid; VC2, Cinnamaldehyde; VC3, Anethole; VC4,  $\alpha$ -Pinene; VC5,  $\alpha$ -Muuroleone; VC6, Naphthalene, 1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-, (1S-cis)-.



**Figure 2.** Trend diagram of volatile components content of cinnamon in beef with marinating time.

ingredients detected in beef were less but the major ingredients of cinnamon was consistent (Liu et al., 2013). The result shows that best process is marinating for 12 hours.

### 3.3 The bitter processing

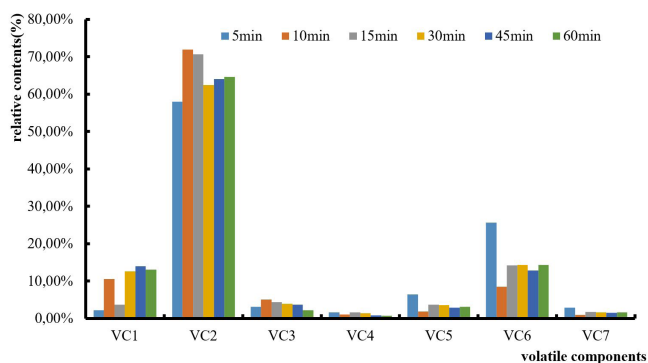
It can be seen from Table 3, seven components are detected in the volatile during different time of bitter processing which contain one acid, one aldehyde, three alkene and two naphthalenes. They are cinnamic acid, cinnamaldehyde, anethole,  $\alpha$ -Pinene,  $\alpha$ -muuroleone, naphthalene, 1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-, (1S-cis)- and naphthalene, 1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)-. However,  $\alpha$ -pinene and naphthalene, 1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)- were not detected at 90 minutes of cooking. When cooked for 10 minutes and 15 minutes, the relative content of cinnamaldehyde reached to 71.92% and 70.67%. The relative percentage of  $\alpha$ -pinene was minimal during the seven periods which was only lower than cinnamaldehyde in cinnamon powder. Cinnamic acid with content of 0.21% in cinnamon powder was detected in the marinade but naphthalene, 1,2,4a,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)- with content of 2.11% in cinnamon powder wasn't detected. The seven main components and their proportions are consistent with the published literature (Wang et al., 2014).

Since seven substances were detected except for cooking for 90 minutes, only the relative content of volatile components in the first six cooking times was shown in Figure 3. In compare with other time periods, the relative percentage of cinnamaldehyde reached the maximum at 10 minutes and 15 minutes of cooking. The relative content of anethole,  $\alpha$ -pinene,  $\alpha$ -muuroleone and naphthalene, 1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)- have no significant difference. When cooked for 5 minutes, the relative amount of cinnamic acid was lower, but the content of naphthalene, 1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-, (1S-cis)- was higher. Considering the relative content of cinnamic acid, cinnamaldehyde and naphthalene, 1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-, (1S-cis)- in different cooking time, the composition in brine was more flavor when cooking for 10 minutes and 15 minutes.

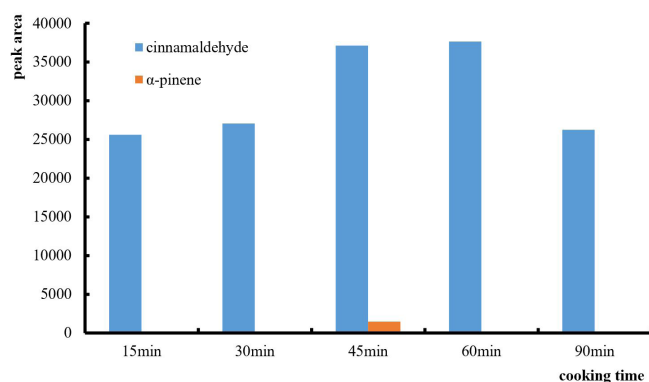
As can be seen from Table 3 and Figure 3, the relative content of volatile compounds in different cooking times is more stable than that of the marinating process. The volatile components detected in the extract are much less than those in the powder. According to the relative content of the main

**Table 3.** The relative contents of main components of cinnamon from cinnamon powder to cooking extract.

Component	5min	10min	15min	30min	45min	60min	90min
cinnamic acid	2.22%	10.52%	3.69%	12.61%	14.05%	13.11%	36.80%
cinnamaldehyde	57.98%	71.92%	70.67%	62.44%	64.02%	64.67%	55.47%
anethole	3.16%	5.10%	4.37%	3.95%	3.70%	2.23%	1.75%
$\alpha$ -Pinene	1.67%	1.12%	1.62%	1.45%	0.90%	0.74%	-
$\alpha$ -Muuroleone	6.46%	1.88%	3.69%	3.62%	2.88%	3.18%	1.18%
Naphthalene, 1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-, (1S-cis)-	25.64%	8.55%	14.17%	14.28%	12.87%	14.38%	4.80%
Naphthalene, 1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)-	2.87%	0.91%	1.79%	1.65%	1.58%	1.69%	-



**Figure 3.** Trend diagram of relative content of volatile components in cinnamon with different cooking time. Notes: VC1,cinnamic acid; VC2,cinnamaldehyde; VC3,anethole; VC4, $\alpha$ -Pinene; VC5, $\alpha$ -Muurolene; VC6,Naphthalene,1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-, (1S-cis)-; VC7,Naphthalene,1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)-.



**Figure 4.** Trend diagram of volatile components content of cinnamon in beef with cooking time.

seven active substances in the volatile components of cinnamon, the cooking 15 minutes of bittern is the most suitable time for making the marinade.

In order to ensure that the starting conditions of the stewed beef are the same except the different processing, and to obtain the extract of cinnamon quickly, the bittern was chosen to cooking for 15 minutes to treat the beef. Then compare the relative content of volatile components of cinnamon from extract to beef during the different cooking times.

The content of the substance was measured by the peak area. In the process of bittern, cinnamaldehyde was detected in the beef during different cooking time. It can be seen from the Figure 4 that the content of cinnamaldehyde reached a maximum at 60 minutes of cooking. The content of cinnamaldehyde at 45 minutes of cooking was slightly lower than that of 60 minutes and  $\alpha$ -pinene was detected at 45min. This result was slightly different from the study of Ai Youwei et al. (Ai et al., 2018a). In their research cinnamaldehyde reached the max at 30 minutes but the change trend was consistent, first increasing and then

decreasing. In summary, the 45 minutes of cooking was chosen the best bittern duration.

## 4 Conclusion

HS-GC-MS is simple to operate and doesn't require complicated pre-treatment. It is more suitable for the detection of volatile components than other traditional detection methods. It is fast and effective in the detection of volatile compounds in cinnamon powder. In the marinade processing, the most volatile components was detected in marinade when marinating for 1 hour, such as cinnamic acid(4.39%), cinnamaldehyde(46.57%), anethole(8.05%),  $\alpha$ -pinene(7.38%), naphthalene,1,2,4a,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-(0.93%),  $\alpha$ -muurolene(8.99%),naphthalene,1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-,(1S-cis)-(20.31%),naphthalene,1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)-(3.03%) and  $\alpha$ -calacorene(0.35%). In the bittern processing, the flavor components in the bittern when cooking for 15 minutes were the most and 7 substances were detected which were cinnamic acid(3.69%), cinnamaldehyde(70.67%), anethole(4.37%),  $\alpha$ -pinene(1.62%),  $\alpha$ -muurolene(3.69%), naphthalene,1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-,(1S-cis)-(14.17%) and naphthalene,1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)-(1.79%). The relative content of the volatile components in two bittern were slightly different. In both processing, the loss of effective flavor component of cinnamon is serious. The loss rate of the bittern is greater than the marinade process, but the flavor immersion in the bittern process is faster. Moreover, the volatile components can be detected in cinnamon extract were much less than the cinnamon powder. As the processing time changed, cinnamaldehyde is always the main forming substance of the flavor and the relative content of other active ingredients changed accordingly. In two processes, only cinnamaldehyde was detected in the stewed beef, while a small amount of  $\alpha$ -pinene was detected when marinating for 1 hour and cooking for 45 minutes. It can be seen from different processing that the spice effect the flavor of stewed beef, and the processing time isn't as long as possible but needs to be controlled at the right time. As the compounds with the largest content and being detectable, cinnamaldehyde can be used as a detection index to evaluate the processing. HS-GC-MS can be used as method to detect volatile components.

## Acknowledgements

This work is supported by Primary Research & Development Plan of Hubei Province (2020BBB074) and Primary Research & Development Plan of Hubei Province(2020BBA046). The authors have no financial interest or other potential conflict of interests.

## References

- Ai, Y., Hou, W., Zhang, J., & Wang, H. (2018a). Preliminary study on detection methods for the main effective components of common spices in marinated. *Zhongguo Tiaoweipin*, 43(6), 132-138.
- Ai, Y., Hou, W., Zhang, J., & Wang, H. (2018b). Research on change rule of main effective compositions of spices in sauce bittern product during processing. *Food Industries*, 39(8), 191-194.
- Chen, J., He, Z., Qin, F., Chen, J., Cao, D., Guo, F., & Zeng, M. (2017). Inhibitory profiles of spices against free and protein-bound heterocyclic

- amines of roast beef patties as revealed by ultra-performance liquid chromatography-tandem mass spectrometry and principal component analysis. *Food & Function*, 8(11), 3938-3950. <http://dx.doi.org/10.1039/C7FO00935F>. PMID:28933794.
- Chen, Y., Lin, C., Cheng, S., & Chang, S. (2015). Rapid Discrimination and Feature Extraction of Three *Chamaecyparis* Species by Static-HS/GC-MS. *Journal of Agricultural and Food Chemistry*, 63(3), 810-820. <http://dx.doi.org/10.1021/jf505587w>. PMID:25590241.
- Colina-Coca, C., González-Peña, D., Vega, E., de Ancos, B., & Sánchez-Moreno, C. (2013). Novel approach for the determination of volatile compounds in processed onion by headspace gas chromatography-mass spectrometry (HS GC-MS). *Talanta*, 103, 137-144. <http://dx.doi.org/10.1016/j.talanta.2012.10.022>. PMID:23200369.
- Ganguly, S., Mahanty, A., Mitra, T., Raman, R. K., & Mohanty, B. P. (2017). Volatile compounds in hilsa (*Tenualosa ilisha*, Hamilton) as detected by static headspace gas chromatography and mass spectrometry. *Journal of Food Processing and Preservation*, 41(6), e13212. <http://dx.doi.org/10.1111/jfpp.13212>.
- Gilbert-López, B., Robles-Molina, J., García-Reyes, J. F., & Molina-Díaz, A. (2010). Rapid determination of BTEXS in olives and olive oil by headspace-gas chromatography/mass spectrometry (HS-GC-MS). *Talanta*, 83(2), 391-399. <http://dx.doi.org/10.1016/j.talanta.2010.09.052>. PMID:21111151.
- Evrendilek, G. A. (2015). Empirical prediction and validation of antibacterial inhibitory effects of various plant essential oils on common pathogenic bacteria. *International Journal of Food Microbiology*, 202, 35-41. <http://dx.doi.org/10.1016/j.ijfoodmicro.2015.02.030>. PMID:25764982.
- Guo, X., Sun, W., Huang, L., Wu, L., Hou, Y., Qin, L., & Liu, T. (2017). Effect of cinnamaldehyde on glucose metabolism and vessel function. *Medical Science Monitor*, 23, 3844-3853. <http://dx.doi.org/10.12659/MSM.906027>. PMID:28790298.
- Han, C., Liu, G., Li, Y., Cui, Y., Tao, Y., & Wang, J. (2014). Preservation effect of rhubarb and cinnamon extracts on fresh beef. *Applied Mechanics and Materials*, 469, 175-179. <http://dx.doi.org/10.4028/www.scientific.net/AMM.469.175>.
- Hernández-Ochoa, L., Aguirre-Prieto, Y. B., Nevárez-Moorillón, G. V., Gutierrez-Mendez, N., & Salas-Muñoz, E. (2014). Use of essential oils and extracts from spices in meat protection. *Journal of Food Science and Technology*, 51(5), 957-963. <http://dx.doi.org/10.1007/s13197-011-0598-3>. PMID:24803704.
- Ho, S., Chang, K., & Chang, P. (2013). Inhibition of neuroinflammation by cinnamon and its main components. *Food Chemistry*, 138(4), 2275-2282. <http://dx.doi.org/10.1016/j.foodchem.2012.12.020>. PMID:23497886.
- Peris, I., & Blázquez, M. A. (2014). Comparative GC-MS analysis of bay leaf (*Laurus nobilis* L.) essential oils in commercial samples. *International Journal of Food Properties*, 18(4), 757-762. <http://dx.doi.org/10.1080/10942912.2014.906451>.
- Jia, S., & Wang, B. (2014). Determination of volatile components of Jiduhuo (*Angelica biserrata*) by HS-GC-MS. *Anhui Yiyao*, 18(8), 1429-1432.
- Kim, H., Cadwallader, K. R., Kido, H., & Watanabe, Y. (2013). Effect of addition of commercial rosemary extracts on potent odorants in cooked beef. *Meat Science*, 94(2), 170-176. <http://dx.doi.org/10.1016/j.meatsci.2013.01.005>. PMID:23501248.
- Kittisakulnam, S., Saetae, D., & Suntornsuk, W. (2017). Antioxidant and antibacterial activities of spices traditionally used in fermented meat products. *Journal of Food Processing and Preservation*, 41(4), 1-8. <http://dx.doi.org/10.1111/jfpp.13004>.
- Li, H., Shang, Y., He, Z., Tang, J., Xia, Y., & Zhang, Y. (2011). The ideas about modernization development of chinese traditional meat foods. *Journal of Chinese Institute of Food Science and Technology*, 11(7), 1-3.
- Li, Y., Kong, D., & Wu, H. (2013). Analysis and evaluation of essential oil components of cinnamon barks using GC-MS and FTIR spectroscopy. *Industrial Crops and Products*, 41, 269-278. <http://dx.doi.org/10.1016/j.indcrop.2012.04.056>.
- Liu, X., Zhao, G., Tian, W., Liu, Y., Sun, L., & Liu, Y. (2013). Effects of cinnamon additions on volatile flavor compounds of stewed chicken. *Shipin Yu Fajiao Gongye*, 39(6), 34-40.
- Lu, J., Wang, Y., & Chen, Y. (2010). Systematic review on modernization of traditional Chinese meat products. *Meat Industry*, 11(11), 10-12.
- Nie, S.-P., Huang, J.-G., Zhang, Y.-N., Hu, J.-L., Wang, S., Shen, M.-Y., Li, C., Marccone, M. F., & Xie, M.-Y. (2013). Analysis of furan in heat-processed foods in China by automated headspace gas chromatography-mass spectrometry (HS-GC-MS). *Food Control*, 30(1), 62-68. <http://dx.doi.org/10.1016/j.foodcont.2012.07.020>.
- Radha Krishnan, K., Babuskin, S., Azhagu Saravana Babu, P., Sasikala, M., Sabina, K., Archana, G., & Sukumar, M. (2014a). Antimicrobial and antioxidant effects of spice extracts on the shelf life extension of raw chicken meat. *International Journal of Food Microbiology*, 171, 32-40. <http://dx.doi.org/10.1016/j.ijfoodmicro.2013.11.011>. PMID:24308943.
- Radha Krishnan, K., Babuskin, S., Babu, P. A. S., Fayidh, M. A., Sabina, K., Archana, G., & Sukumar, M. (2014b). Bio protection and preservation of raw beef meat using pungent aromatic plant substances. *Journal of the Science of Food and Agriculture*, 94(12), 2456-2463. <http://dx.doi.org/10.1002/jsfa.6580>. PMID:24425618.
- Shen, S., Zhang, T., Yuan, Y., Lin, S., Xu, J., & Ye, H. (2015). Effects of cinnamaldehyde on *Escherichia coli* and *Staphylococcus aureus* membrane. *Food Control*, 47, 196-202. <http://dx.doi.org/10.1016/j.foodcont.2014.07.003>.
- Tung, Y., Yen, P., Lin, C., & Chang, S. (2010). Anti-inflammatory activities of essential oils and their constituents from different provenances of indigenous cinnamon (*Cinnamomum osmophloeum*) leaves. *Pharmaceutical Biology*, 48(10), 1130-1136. <http://dx.doi.org/10.3109/13880200903527728>. PMID:20815702.
- Wang, B., Gong, W., Chen, G., Song, G., Jin, J., Qin, K., & Cai, B. (2014). Gas chromatography /mass spectrometry on volatile components of cinnamon. *World Journal of Integrated Traditional and Western Medicine*, 9(9), 941-943.
- Wang, C., Xiong, G., Jia, J., Yua, S., & Fang, Y. (2017). Exploiting Electronic Nose to detect braised soup which made by different spices. *Jiangsu Agricultural Science*, 45(10), 136-140.
- Wang, H., Liu, D., Xu, X., Zhou, G., Han, Y., & Xu, B. (2013). Trends in China's industrial development and research of traditional cured meat products. *Meat Research*, 27(9), 36-40.
- Xie, X., Chen, B., Liu, L., Peng, J., Xu, A., Jiang, S., & Lu, H. (2013). Analysis of volatile components from twelve rhodiola compounds by HS-GC-MS. *Acta Scientiarum Naturalium Universitatis Sunyatseni*, 52(5), 97-102.
- Zeng, W., Wen, W., Deng, Y., Tian, Y., Sun, H., & Sun, Q. (2016). Chinese ethnic meat products: continuity and development. *Meat Science*, 120, 37-46. <http://dx.doi.org/10.1016/j.meatsci.2016.04.007>. PMID:27091319.
- Zhang, C., & Guo, M. (2017). Analysis and differentiation of the volatile compounds in red and white wines using desiccated headspace gas chromatography-mass spectrometry coupled with chemometrics. *Food Analytical Methods*, 10(11), 3531-3537. <http://dx.doi.org/10.1007/s12161-017-0924-0>.
- Zhang, H., Wu, J., & Guo, X. (2016). Effects of antimicrobial and antioxidant activities of spice extracts on raw chicken meat quality. *Food Science and Human Wellness*, 5(1), 39-48. <http://dx.doi.org/10.1016/j.fshw.2015.11.003>.