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Effects of different tobacco materials on the release characteristics of aroma components

Qunshan Yan[1,2,3], Jiazhi Ni[1,2,3], Rui Peng[1,2,3], Song Gao[1,2,3], Xiangbin Tang[1,2,3,*], Shuoguo Wei[1,2,3], Liangcai Liu[1,2,3], Pei Wei[1,2,3], Zhe Xiong[4], Yi Wang[4], and Yuxing Tong[1,2,3,*]

[1]China Tobacco Hubei Industrial Co., Ltd., Wuhan 430056, China

[2]Hubei Xinye Reconstituted Tobacco Development Co., Ltd, Wuhan 430056, China

[3]Applied Technology Research of Reconstituted Tobacco Hubei Province Key Laboratory, Wuhan 430056, China

[4] State Key Laboratory of Coal Combustion, Huazhong University of Science and Technology, Wuhan, Hubei 430074, China

*Corresponding author e-mail: tangxb@hbtobacco.cn; tong_yx@126.com.

Abstract. In order to reveal the release characteristics of different tobacco materials, the characteristics of tobacco materials and the release characteristics of smoke were analyzed. This paper studied 17 kinds of tobacco materials, the thermogravimetric analyzer was used to study the thermogravimetric characteristics of the tobacco materials, meanwhile the gas chromatography was used to analysis original aroma composition characteristics of the raw tobacco material, the smoke released from the pyrolysis of tobacco in a fixed bed reactor was also studied. The results showed: ① The elemental composition of different kinds of tobacco materials was similar, but there were significant differences in the volatile content (aroma components), among which the aroma components of tobacco stem were the highest. ② Different categories of tobacco raw materials have different properties of the aroma components. Among them, the nicotine content of roasted tobacco was higher, spice tobacco and tobacco stems mainly contain acid and ketone aroma components. However, burley tobacco and cigar tobacco are distinguished from other types of tobacco by the presence of aromatic substances such as new phytodiene.③ Through the analysis of thermal release characteristics of aroma components of different tobacco raw materials, it was found that the yield of aroma components was positively correlated with the volatile content of tobacco materials. The results of this study clarified the effects of different tobacco raw materials on the release characteristics of aroma components and provided theoretical guidance for the subsequent compound utilization of tobacco.

Keywords: Tobacco; Aroma components; Extraction; Pyrolysis; Nicotine

1. Introduction

The tasting quality style of tobacco is determined by the chemical composition of smoke produced from the pyrolysis and combustion of tobacco. The results showed that there were significant differences in chemical and physical characteristics among different types of tobaccos [1-4], resulting in significant differences in the release characteristics of aroma components and quality styles of different tobacco materials [5-9]. The production of different types of tobacco products relies on the blending of different leaf groups of tobacco raw materials. Clarifying the release characteristics of aroma components of different tobacco materials is the theoretical basis for guiding the development of tobacco products. On the other hand, tobacco flavoring additives can be extracted and prepared from tobacco materials. The components and taste of tobacco flavoring additives obtained from different tobacco materials are different, so it is also necessary to clarify the aroma components of different tobacco materials.

In recent years, in order to study the release characteristics of aroma components of different types of tobacco, researchers mainly studied the smoke release characteristics of tobacco samples by thermal analysis [10-14]. The thermal decomposition processes of tobacco, cigar, and burley tobacco were studied by thermogravimetry analysis. It was found that the characteristics of different types of tobacco could be identified by distinguishing the thermal reaction stages of tobacco combustion, such as distillation, cracking, and combustion [15]. The results showed that the structure difference of tobacco cellulose was one of the fundamental reasons for the different release characteristics of tobacco smoke [16]. Based on the combustion and pyrolysis characteristics of different structures of tobacco cellulose, different structural characteristics of tobacco celluloses were extracted from tobacco, burley tobacco, spice tobacco, and tobacco stem. The combustion and pyrolysis processes of different types of tobacco cellulose were compared through thermogravimetric analysis and micro combustion calorimetry, in the meanwhile, the composition of pyrolysis products and the release rule of carbonyl compounds were detected, and the differences in smoke release characteristics of different types of tobacco were found. Such as the spice tobacco cellulose has a high degree of crystallinity, resulting in the coking being easily performed during the process of combustion, however, there are more light molecule components in the smoke of tobacco stem cellulose. Researchers in the domestic representative of roasted tobacco, slice tobacco, expansion silk, burley tobacco, and spice tobacco as the research object, found that glove box moisture content test method as testing means, physical moisture protection performances of different tobacco materials are different, thus affect the absorption process of smoke release, and confirmed that the Weibull model can better characterize tobacco samples drying dynamics [17]. Existing studies have fully shown that there are significant differences in the release characteristics of aroma components of different types of tobacco, but the composition of smoke released by different types of tobacco and their differences are not cleared yet.

Therefore, this article selects the representative different types of 17 kinds of tobacco materials, including roasted tobacco, burley tobacco, cigar, spice tobacco, and tobacco stem, producing areas including Yunnan, Sichuan, Guizhou, Hubei, Hunan, Fujian, Henan, Shandong, Shanxi, Heilongjiang, Zimbabwe, etc. The characteristics of materials and the release of aroma components were studied, clarifying the release characteristics of aroma components of different materials and providing theoretical support for the formulation development of tobacco products.

2. Materials and methods

2.1 Materials, reagents, and instruments

Dichloromethane (HPLC, Sinopharm); Methanol (HPLC, Sinopharm); Methanol (SP, Sinopharm); Nicotine (99.7% purity, provided by Hubei Xinye Tobacco Chip Development Co., Ltd.); Nitrogen and helium (purity > 99.999%, supplied by Wuhan Huerwen Industrial Co., Ltd); 0.45 μm microporous oil filter membrane (Tianjin Jinteng Company, China); φ 44 mm Cambridge filter (Whatman Company, UK); Otf-1200x type tube furnace (Hefei Kojing Material Technology Co., Ltd.); Trace 1300 ISQ gas chromatography-mass spectrometer (Thermo Fisher Technologies); EX224ZH electronic balance (sensing: 0.0001g, Ohaus Company, USA); CS200A mass flowmeter (Beijing Sevenstar Electronics Co., Ltd); Air-drying oven (101-2AB, Tianjin Tester); 5E-CHN2200 elemental Analyzer (Changsha Kaiyuan Instrument Co., Ltd); 5E-MAG6700 industrial analyzer (Changsha Kaiyuan Instrument Co., Ltd).

There were 17 kinds of tobacco materials, such as roasted tobacco, burley tobacco, cigar, spice tobacco, and tobacco stem, tobacco areas included Yunnan, Sichuan, Guizhou, Hubei, Hunan, Fujian, Henan, Shandong, Shanxi, Heilongjiang, Zimbabwe, etc (The details were shown in Table 1). Before use, samples were crushed and screened, and parts with particle sizes ranging from 0.106-0.150mm were selected. The samples were baked at 80°C for 24 hours and sealed for use.

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Table. 1 The information about the tobacco samples

Serial number	Materials	Area	Recommended level	Recommended year
1	Roasted tobacco	Yunnan	234	2018
2		Sichuan	364	2018
3		Guizhou	442	2018
4		Hubei	009	2018
5		Hunan	604	2018
6		Fujian	585	2018
7		Henan	632	2018
8		Shandong	692	2018
9		Shanxi	782	2018
10		Heilongjiang	742	2018
11		Zimbabwe	801	2017
12	Burley tobacco		904	2012
13	Cigar		9953	2018
14			9947	2018
15	Spice tobacco		924	2012
16	Tobacco stem		274	2019
17	Shredded tobacco from Wuhan			

2.2 Methods

2.2.1 Industrial analysis and elemental analysis of tobacco samples.

17 kinds of raw materials were analyzed by the industrial analyzer (Changsha New Century, 5E-MAG6700) and elemental analyzer (Changsha New Century, 5E-CHN2200) respectively. For industrial analysis, GB/T 28731-2012 standard was referred to. For element analysis, refer to GB/T 214-2007 and GB/T 30733-2014.

2.2.2 Extraction of aroma components from tobacco samples.

According to industry standard "tobacco essence ethanol, 1 2-propylene glycol, glycerol content determination gas chromatography YCT 242-2008", tobacco raw materials extraction experiment. Specific experimental steps were as follows: Each tobacco sample was accurately weighed as 1.0± 0.1g by using a high-precision electronic balance and was mixed with 25 mL methanol in a centrifuge tube. The centrifuge tube was placed on a rotating shaker and fully mixed for 4 hours before being removed for use.

2.2.3 Characterization of aroma components in tobacco samples.

The pyrolysis oil solution was characterized and analyzed by Gas chromatography-mass spectrometry (Thermo Scientific, Trace 1300/ISQ) with the hp-innowax column (length 30 m, inner diameter 0.25 mm, thickness 0.25 mm). Before the test, the sample solution was filtered by an oil filter of 0.45 μm. The sample volume was set to 1 μL/ time, the carrier gas was high purity helium (purity > 99.99%), the flow rate was 1.0 mL/min, and the injection mode was not split. The inlet temperature was set at 250 °C, and the column temperature program was as follows: after staying at 40 °C for 3 min, the heating rate was increased to 250 °C at 5 °Cmin-1 and staying at 250 °C for 10

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min. After the test, the peak area of each component was multiplied by the correction factor "mass of solution/mass of raw material" to obtain the peak area data based on "per gram of raw material".

2.2.4 Thermogravimetric characterization of tobacco samples.

Thermogravimetry refers to a technology that measures the relationship between the mass and temperature of a substance under program-controlled temperature. The measured curve is the TG curve, the Y-axis is the mass of the sample, and the X-axis is temperature or time. In this study, the PESTA8000 synchronous thermal analyzer was used to measure the weight loss and heat absorption during the heating process, and the temperature range of subsequent rapid pyrolysis experiments was determined according to the results. The heating procedure was set as follows: the heating rate was 10 °Cmin-1 from 30 °C to 1100 °C, the carrier gas was N2, the flow rate was 100 mLmin-1, and the 40 mg sample was placed each time.

2.2.5 Pyrolysis of tobacco samples.

The pyrolysis experiment of tobacco stalk waste was carried out in a self-made fixed-bed quartz reactor. In the pyrolysis process, high purity nitrogen (purity > 99.999%) was used as carrier gas and reaction gas, and the total airflow was 200 mLmin-1. Before sample injection, reaction gas was first introduced and purged for 30 minutes to ensure a set atmosphere in the reactor. The quartz reactor was heated to the target temperature and kept steady for 15 min. Before the experiment, 5.0±0.1 g tobacco samples were evenly spread in the quartz boat, and the quartz boat was placed at the inlet end of the quartz reactor. During sample injection, the quartz boat loaded with tobacco samples was quickly pushed into the center of the heating zone for pyrolysis reaction. After 20 minutes of reaction, the quartz boat was pulled out of the high-temperature zone, and the nitrogen atmosphere was switched for cooling. The reaction time of 20 minutes could ensure sufficient pyrolysis of the tobacco. The volatiles produced by pyrolysis enter the U-tube with the carrier gas and were completely condensed by liquid nitrogen. After the experiment, the yield of pyrolysis oil was calculated by the quality difference between the u-tube and Cambridge filter before and after the experiment. After weighing the u-tube and Cambridge filter, the organic solvent (methanol to methylene chloride 1:4 by volume) was used to dissolve the pyrolysis oil, and the solution was collected. The collected solution was stored in a refrigerator (-16 °C) and the samples were analyzed and tested within one week.

3. Results and Discussion

3.1 Analysis of composition characteristics of multiple tobacco raw materials

After tobacco samples are burned in the elemental analyzer, carbon converted into carbon dioxide form, hydrogen in water form, nitrogen in monomer form or nitrogen oxide form and sulfur dioxide form can be quantitatively determined, and then the percentage content of C, H, N, S, and O in tobacco can be calculated. The elemental analysis results of 17 tobacco material samples were shown in Table 2. The results of the elemental analysis showed that the elemental compositions of different tobacco materials were similar. The highest content of oxygen and carbon in tobacco was 34.98-45.80 wt.% and 37.22-43.98 wt.%, respectively. Compared with other herbaceous biomass, tobacco contained nitrogenous compounds such as nicotine, so it contained more nitrogen elements. The nitrogen content of 17 tobacco material samples ranged from 1.61 wt.% to 4.89 wt.%.

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Table 2 The results of elemental analysis of tobacco samples (as received basis)

Sample	C(wt.%)	H(wt.%)	O(wt.%)*	N(wt.%)	S(wt.%)
Roasted tobacco 1	41.52	5.10	40.86	2.35	0.49
Roasted tobacco 2	42.25	5.02	42.30	2.58	0.41
Roasted tobacco 3	38.85	4.49	34.98	4.89	0.26
Roasted tobacco 4	40.79	5.11	42.62	1.86	0.49
Roasted tobacco 5	42.08	5.27	42.95	2.11	0.23
Roasted tobacco 6	37.22	4.08	39.51	4.01	0.32
Roasted tobacco 7	35.81	4.15	45.80	1.79	0.18
Roasted tobacco 8	40.15	4.75	44.36	2.15	0.59
Roasted tobacco 9	36.33	4.67	37.56	4.76	0.32
Roasted tobacco 10	40.79	5.36	42.71	1.84	0.28
Roasted tobacco 11	41.57	5.30	43.44	2.35	0.33
Burley tobacco 12	39.38	4.91	45.03	1.61	0.30
Cigar 13	41.33	5.05	41.17	2.27	0.44
Cigar 14	41.13	5.02	42.38	1.82	0.18
Spice tobacco 15	40.64	4.86	43.12	2.32	0.23
Tobacco stem 16	42.58	5.07	41.63	2.69	0.34
Shredded tobacco from Wuhan 17	43.98	5.78	35.42	2.15	0.32

The industrial analysis could detect the contents of water (M), ash (A), volatile matter (V), and fixed carbon (FC) in tobacco samples. The smoke released by tobacco was mainly composed of volatile components, so the volatile content of tobacco materials determined the release amount of aroma components. The industrial analysis results of 17 tobacco samples were shown in Table 3. The results of the industrial analysis showed that although the elemental composition of different tobacco samples was similar, there were still significant differences in volatile content. Among them, the volatile content of roasted tobacco was 58.475-66.610 wt.%, burley tobacco was 64.120 wt.%, cigar was 64.630-65.415 wt.%, and spice tobacco was 64.485 wt.%. The volatile content of the tobacco stem was 66.375 wt.%. According to the volatile content of different kinds of tobacco samples, tobacco stem could produce more aroma components, while some kinds of roasted tobacco produced slightly lower aroma components.

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Table 3 The results of proximate analysis of tobacco samples (as received basis)

Sample	Moisture(wt.%)	Ash(wt.%)	Volatile(wt.%)	Fixed carbon(wt.%)
Roasted tobacco 1	12.565	10.170	65.965	11.300
Roasted tobacco 2	14.550	7.855	65.325	12.270
Roasted tobacco 3	12.985	16.795	63.460	6.760
Roasted tobacco 4	12.775	9.625	65.465	12.135
Roasted tobacco 5	11.890	7.595	66.610	13.905
Roasted tobacco 6	18.450	15.185	58.475	7.890
Roasted tobacco 7	16.675	12.455	58.820	12.050
Roasted tobacco 8	16.525	8.590	63.890	10.995
Roasted tobacco 9	16.540	16.680	60.080	6.700
Roasted tobacco 10	11.135	9.305	66.145	13.415
Roasted tobacco 11	13.425	7.340	65.250	13.985
Burley tobacco 12	14.770	9.075	64.120	12.035
Cigar 13	12.600	10.185	65.415	11.800
Cigar 14	12.165	9.655	64.630	13.550
Spice tobacco 15	13.775	9.060	64.485	12.680
Tobacco stem 16	12.865	8.030	66.375	12.730
Shredded tobacco from Wuhan 17	11.73	12.35	63.390	12.53

3.2 Analysis of the characteristics of aroma components in tobacco materials

To clarify the characteristics of different kinds of aroma components in tobacco materials, based on the method of tobacco industrial standard YC/T243-2008, methanol was used as the extraction agent to extract aroma components in tobacco, and GC-MS was used to detect various aroma components. To accurately quantify nicotine content, the external standard method was used to quantify nicotine and calculate the nicotine content of tobacco materials. Table 4 showed the amount of nicotine in 17 different aromatic-producing components of tobacco materials obtained in the experiment.

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Table. 4 The yield of nicotine in the aroma components of tobacco

Serial number	Materials	Area	Recommended level	Recommended year	Nicotine content (wt.%)
1	Roasted tobacco	Yunnan	234	2018	0.1031
2		Sichuan	364	2018	0.1091
3		Guizhou	442	2018	0.0700
4		Hubei	009	2018	0.1580
5		Hunan	604	2018	0.1388
6		Fujian	585	2018	0.0806
7		Henan	632	2018	0.0745
8		Shandong	692	2018	0.0707
9		Shanxi	782	2018	0.0706
10		Heilongjiang	742	2018	0.1547
11		Zimbabwe	801	2017	0.0569
12	Burley tobacco		904	2012	0.1673
13	Cigar		9953	2018	0.0287
14			9947	2018	0.0122
15	Spice tobacco		924	2012	0.0105
16	Tobacco stem		274	2019	0.0038
17	Shredded tobacco from Wuhan				0.0730

It could be seen from the above table that the nicotine content of the 17 kinds of tobacco materials was 0.0038-0.1673 wt.%, among which the nicotine content of cigar, spice tobacco, and tobacco stem was relatively low. Further, according to GC-MS detection results, the extracted aroma components were characterized and analyzed, and different categories of aroma components in the aroma components were classified and analyzed to obtain the relative contents of different categories of aromatic substances, as shown in Figure 1-3. As could be seen from the results in Figure 1-3, the aroma components extracted from Sample 1-11 and Sample 15-17 were similar in type, mainly including ketones, acids, alcohols, and furans. Among them, acid aroma components and ketone aroma components were the main components with high content. However, the compounds detected in the extracts of Sample 12-14 tobacco (Sample 12 was burley tobacco, and Sample 13-14 was cigar) were less, mainly acetic acid, neophytadiene, palmitic acid, stearic acid, chlorophyll, and other compounds, among which the content of neophytadiene in burley tobacco was significantly higher than other compounds. The results for burley tobacco and cigar were significantly different from those for other samples and the summary results were shown below. The results showed that burley tobacco and cigar were significantly different from other types of tobacco (roasted tobacco, spice tobacco, and tobacco stem).

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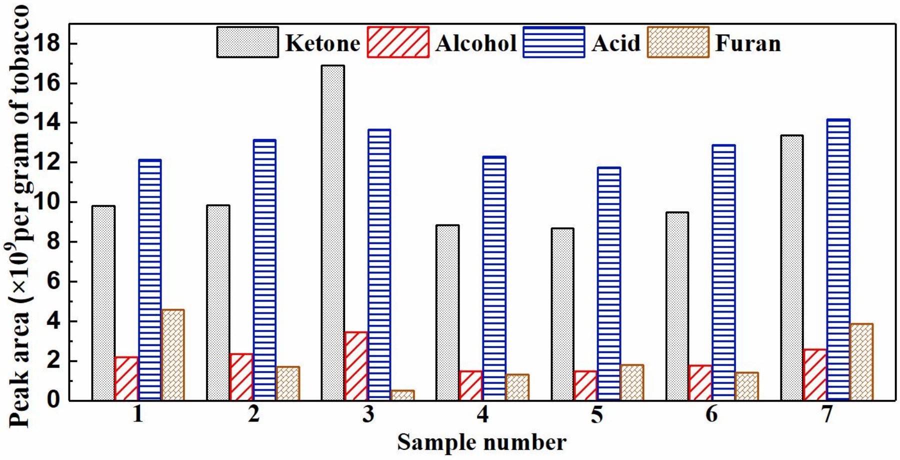

Figure 1. The yield of the light compounds in aroma components of tobacco from Sample 1-7

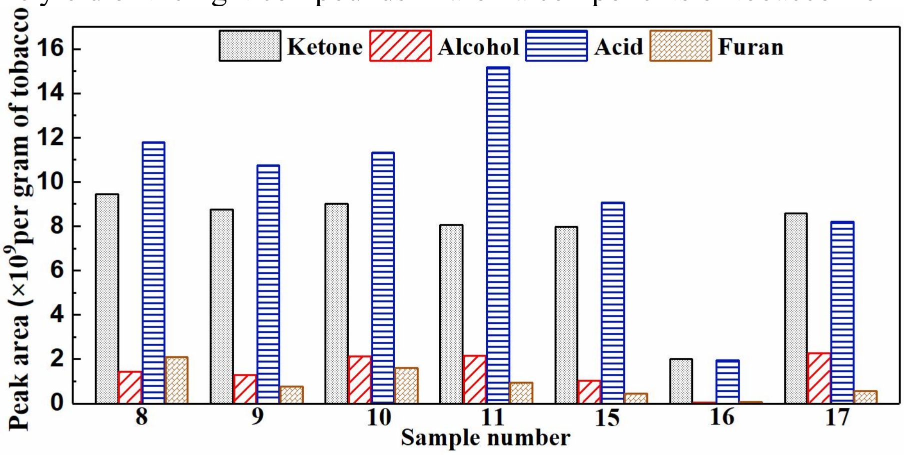

Figure 2. The yield of the light compounds in aroma components of tobacco from Sample 8-11 and Sample 15-17

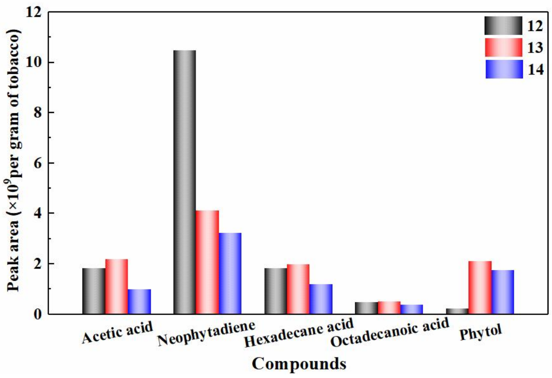

Figure 3. The yield of the light compounds in aroma components of tobacco from Sample 12-14

3.3 Analysis of thermal weight loss characteristics of various tobacco materials

Thermal release characteristics of tobacco leaf and stem samples were studied by using a thermogravimetric analyzer (NETZSCH STA449). Thermal release characteristics of typical tobacco samples of roasted tobacco, burley tobacco, cigar, and spice tobacco were shown in Figure 4. In general, there were three mass loss peaks in tobacco samples. The first mass loss peak was about 100 oC, which was mainly the sample dehydration peak. The second and third peaks were the main volatiles precipitation peaks of tobacco samples, which were significantly different due to the difference in species, origin, and year. The separation peaks of volatiles from roasted tobacco were about 200 oC and 350 oC, and the intensity of the third peak was significantly stronger than that of the second peak. The main volatiles release peaks of burley tobacco and cigar were similar and

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close to each other, about 260 oC and 320 oC, but the weight loss rate of the main volatiles release peaks of burley tobacco was lower than that of burley tobacco, and the weight loss rate of the secondary volatiles release peaks (such as 510 oC and 700 oC) was stronger than that of burley tobacco. The main volatiles were separated between roasted tobacco and burley tobacco, which were about 220 oC and 320 oC. The thermal release characteristic curves of the tobacco stem were different. Compared with the three major weightlessness peaks of tobacco, there were four major weightlessness peaks of tobacco stem. Besides the dehydration peak, there were also three volatile release peaks of 200 oC, 290 oC, and 320 oC. After pyrolysis, the mass fraction of residual char of tobacco stem was 30 wt.%, which was similar to other tobacco.

The same kind of tobacco also showed different thermal release characteristics due to the difference in producing area and year, as shown in Figure 4-5. In general, the thermogravimetric peak of roasted tobacco was mostly at 100 oC, 200 oC, and 350 oC, and the intensity of the third peak was significantly stronger than that of the second peak. However, the second peak of Sample 2 and Sample 9 from Sichuan and Shanxi was slightly stronger than the third peak. In addition, after pyrolysis at 800 oC, the residual char of Sample 3 from Guizhou had a minimum mass of 20%, which was lower than the 30 wt.% residual mass of other roasted tobacco samples. In Figure 4, the weight loss rate curves of Sample 13 and Sample 14 cigars were very similar. Two main volatiles precipitation peaks appeared at 260 oC and 320 oC, and two secondary volatiles precipitation peaks appeared at 510 oC and 700 oC. However, there were significant differences in the residual char mass of different cigar samples. The residual char mass fraction of Sample 13 after pyrolysis was 33 wt.%, while that of Sample 14 after pyrolysis was 25 wt.%. That was the thermal release characteristic curves of two kinds of cigars. It can be seen that the weight loss rate curves of the two cigars were very similar, with two main volatiles precipitation peaks appearing at 260 oC and 320 oC, and two secondary volatiles precipitation peaks appearing at 510 oC and 700oC. However, there were significant differences in the residual char mass of different cigar samples. The residual char mass fraction of Sample 13 after pyrolysis was 33 wt.%, while that of Sample 14 after pyrolysis was 25 wt.%.

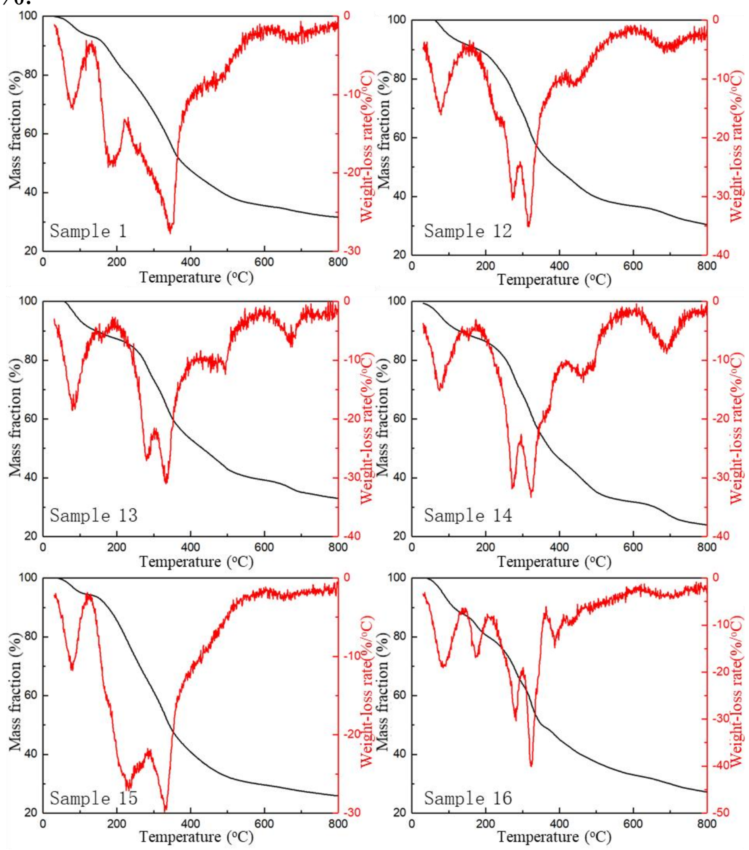

Figure 4. The weight loss characteristics of different tobacco samples

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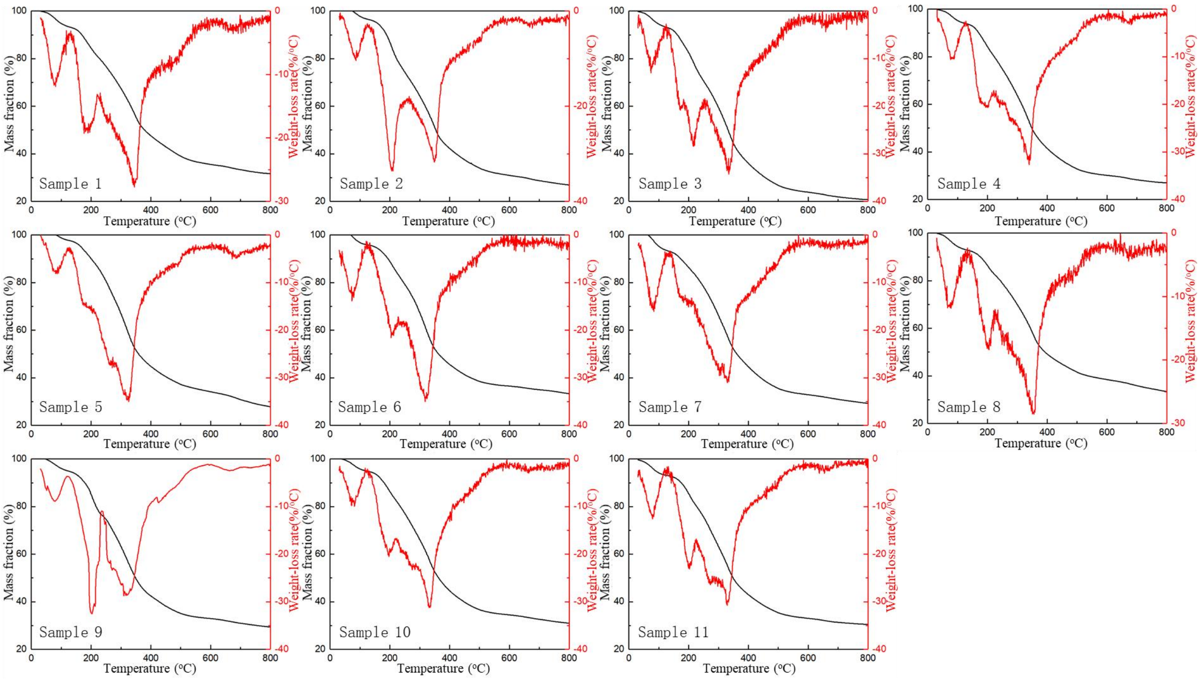

Figure 5. The weight loss characteristics of roasted tobacco samples

3.4 Analysis of the release amount of aroma components from various tobacco materials

To clarify the release characteristics of aroma components from different kinds of tobacco materials, 17 different kinds of tobacco materials provided by Hubei Xinye Reconstituted Tobacco Development Co., Ltd were used in this experiment to extract aroma components. The experiment was carried out in a fixed-bed reactor (58 mm). The reaction conditions were as follows: the temperature was 800 oC, the heating rate was a fast heating condition, the carrier gas was high purity nitrogen, and the gas flow rate was 200 mLmin-1. The released aroma components were condensed and enriched through normal temperature water. The yield of aroma components prepared from 17 different tobacco materials obtained in the experiment is shown in Table 5.

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Table. 5 The thermal release characteristics of aroma components of tobacco

Serial number	Materials	Area	Recommended level	Recommended year	Yield (wt.%)
1	Roasted tobacco	Yunnan	234	2018	31.71
2		Sichuan	364	2018	31.22
3		Guizhou	442	2018	31.58
4		Hubei	009	2018	32.72
5		Hunan	604	2018	31.52
6		Fujian	585	2018	29.09
7		Henan	632	2018	32.32
8		Shandong	692	2018	29.23
9		Shanxi	782	2018	31.39
10		Heilongjiang	742	2018	34.20
11		Zimbabwe	801	2017	31.01
12	Burley tobacco		904	2012	30.10
13	Cigar		9953	2018	31.56
14			9947	2018	30.42
15	Spice tobacco		924	2012	30.75
16	Tobacco stem		274	2019	36.33
17	Shredded tobacco from Wuhan				30.19

Based on the industrial analysis data of 17 kinds of materials and the thermal release yield of aroma components, it could be concluded that the yield of aroma components was positively correlated with the volatile content of tobacco materials. The results showed that the yield of aroma components in tobacco stem was the highest (36.33 wt.%), the yield of aroma components in roasted tobacco 6 (Fujian) and 8 (Shandong) was the lowest (29.09 wt.% and 29.23 wt.%), respectively. When extracting aroma components from tobacco materials, we should refer to the yield of aroma components from different materials and select samples with a higher yield.

4. Conclusion

Based on the above results, it could be seen that ① the elemental composition of different kinds of tobacco materials was similar, but there were significant differences in the volatile content (i.e. aroma components), among which the aroma components of tobacco stem were the most. ② The characteristics of aroma components in different kinds of tobacco materials were different. Among them, the nicotine content of roasted tobacco was higher, and the aroma components of roasted tobacco, spice tobacco, and tobacco stem were mainly acids and ketones, but burley tobacco and cigar were different from other kinds of tobacco, mainly contain neophytadiene and other aroma components. ③ Through the analysis of thermal release characteristics of aroma components of different tobacco materials, it was found that the yield of aroma components was positively correlated with the volatile content of tobacco materials. The results of this study clarified the effects of different tobacco materials on the release characteristics of aroma components and provided theoretical guidance for the subsequent compound utilization of tobacco.

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