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Effects of aroma additives on the release of smoke during the pyrolysis of reconstituted tobacco

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Abstract. The effects of typical acid and alkaline aroma additives on the release characteristics of aroma components from reconstituted tobacco were studied. In this study, the pyrolysis of reconstituted tobacco samples loaded with different aroma components was carried out, and the aroma components in the smoke were analyzed by the GC-MS. The results showed that adding a certain amount of acid aroma components in reconstituted tobacco could promote the release of aroma components in smoke. However, excessive acid aroma additives could inhibit the Maillard reaction to decrease the formation of additional aroma components during pyrolysis. The addition of ethanol in reconstituted tobacco could promote the release of olefin and acid aroma components in smoke. Alkaline aroma additives (sodium bicarbonate and potassium carbonate) could promote the release of all kinds of aroma components in smoke, because potassium and sodium ions could catalyze the pyrolysis of tobacco, which was preferred to generate aroma components. According to these results, the best promoting effect of the aroma components released in smoke was performed by adding 0.078 wt% of potassium carbonate and 0.01 wt% of citric acid in reconstituted tobacco.

Keywords: pyrolysis; aroma additives; reconstituted tobacco

1. Introduction

As people's health awareness increases year by year and with the strengthening of tobacco control policies, global cigarette sales decreases. The tobacco companies have deployed new tobacco products that can reduce health damage, among which heated not burn (HNB) cigarettes have the highest potential to replace traditional cigarettes. In recent years, the market consumption of HNB cigarettes is increasing rapidly[1].

Compared with traditional cigarettes, the most notable feature of HNB cigarettes is the low heating temperature of the tobacco stick during smoking, which is about 300-350°C. The lower heating temperature reduces the release of harmful components (such as tar, nitrosamines, phenolic compounds, etc.) in smoke by more than 80%, so it has a significant health advantage over traditional cigarettes. Meanwhile, HNB cigarettes can also release smoke that contains nicotine and other aroma components, which can satisfy the physiological needs of consumers to a certain extent [2]. However, the main shortcoming of HNB cigarettes is also due to the low heating temperature of the tobacco stick. The released smoke components are only the compounds that are easy to be cracked or the added components (such as atomizers) with the low-boiling point in the tobacco stick. Therefore, the content of aroma components can not effectively be improved the smoking feeling, the smoke is generally thinner, and the smoking feeling is weaker than traditional tobacco [3-6]. In consequence, increasing the release of effective components in smoke during smoking is the key factor in improving the quality of HNB cigarettes [7, 8].

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Adding additional aroma components to the tobacco stick is one of the effective methods to improve the aroma quality of HNB cigarettes [9, 10]. Through the process of adding additional aroma components to the tobacco stick, the aroma components in the smoke can be increased, the smoke concentration can be stabilized, and the smoking feeling can be enhanced[11-13]. In this study, the effects of typical aroma additives such as lactic acid, malic acid, citric acid, ethanol, sodium bicarbonate, and potassium carbonate (with increasing pH value) which were added in the reconstituted tobacco on the smoke release characteristics during pyrolysis of HNB cigarettes were studied. Among the above aroma components, lactic acid can maintain the humidity of tobacco, remove the impurities in tobacco, change the taste, and neutralize some nicotine in smoke at the same time, reducing harm to the human body [14]. Malic acid is a commonly used food additive, which has a soft taste (with a high buffer index) and a special fragrance, and it is a new generation of food sour agents[15]. Citric acid is a kind of edible acid, which is the largest organic acid produced by biochemical methods in the world[16]. Ethanol has a low boiling point and is easily miscible with other organic components. It also functions as an atomizing agent. Sodium bicarbonate and potassium carbonate are widely used acidity regulators and chemical leavening agents in the food industry [17-22]. Therefore, these aroma additives have great potential for application in tobacco, which is worth the in-depth study.

2. Materials and methods

2.1 Materials, reagents and instruments

Materials: The original tobacco powder which was used to produce HNB cigarette reconstituted tobacco leaves (provided by Hubei Xinye Reconstituted Tobacco Development Co., Ltd., 150-300 mesh) was chosen as the sample in this study. Reagent: Lactic acid (Sinopharm Group, AR); Citric acid (Sinopharm Group, AR); Malic acid (Sinopharm Group, AR); Ethanol (Sinopharm Group, AR); Potassium Carbonate (Sinopharm Group, AR); Sodium Bicarbonate (Sinopharm Group, AR). Instrument: Tobacco pyrolysis device (specially made); gas chromatography-mass spectrometer (GC-MS, ThermoFisher, Trace 1300/ISQ); blast drying oven (Tianjin Test, 101-2AB); analytical balance (Ohao Sri Lanka, EX1100ZH).

2.2 Methods

2.2.1 Tobacco sample preparation

Different mass percentages of lactic acid, malic acid, citric acid, ethanol, sodium bicarbonate, potassium carbonate, and their mixtures (see Table 1) were added to the tobacco raw materials to prepare the samples, respectively. After thorough mixing, the tobacco powder and atomizer (Glycerin: propylene glycol=7:3) and other materials were used to produce the reconstituted tobacco sample. The content of each aroma component in the reconstituted tobacco sample was shown in Table 1.

Table1. Mass percentage of the additives used in the tobacco sample

Group	Added aroma substances	The added proportion（wt%）			
		Sample 1	Sample 2	Sample 3	Sample 4
1	lactic acid	0.470	0.780	1.250	/
2	malic acid	0.010	0.013	0.025	0.064
3	citric acid	0.010	0.013	0.025	0.064
4	ethanol	0.470	0.780	1.250	/
5	sodium bicarbonate	0.060	0.310	0.630	/
6	potassium carbonate	0.016	0.031	0.078	0.156

/: Unprepared sample

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2.2.2 Pyrolysis experiment

The pyrolysis experiment of tobacco samples was carried out on a special tobacco pyrolysis reaction system (Figure 1). The system was heated by a controllable temperature-resistance furnace. In order to simulate the smoking situation of HNB cigarettes, the reactor was designed as a small-diameter quartz tube (24 mm in diameter), and the tobacco sample was placed in the quartz tube through a quartz boat. The pyrolysis temperature was set to 330°C to simulate the actual heating temperature of HNB cigarettes. The outlet section of the reactor was connected to a U-shaped condenser (dry ice cooling), and the condensable components in the smoke were collected in the U-shaped tube and further analyzed. The other end of the U-shaped condenser was connected with a pair of flanges, and a Cambridge filter was sandwiched between the flanges to further collect the flue gas components; the right end of the flange was connected with a small suction pump to simulate the airflow in the suction process.

The experimental procedure of pyrolysis was as follows: Place the quartz tube in the electric furnace, connect the pipes, and set the temperature of the electric furnace to 330°C. The pyrolysis experiment was carried out in a nitrogen atmosphere with a gas flow rate of 150 ml/min (close to the real suction flow rate). After the temperature of the electric furnace was stabilized, a small quartz boat containing 1.0 ± 0.01 g of tobacco samples was quickly pushed into the center of the reactor, and after 5 minutes of reaction, the quartz boat was pushed out and cooled in a nitrogen atmosphere. Repeated the above experiment 10 times to increase the amount of collected product to reduce the error. Recorded the quality of the quartz tube, connected tube, U-shaped condenser, flange, and filter paper before and after pyrolysis. The mass difference between the quality before and after was the quality of the released liquid product, that was, the condensable components in the smoke. The non-condensable gases were collected with airbags.

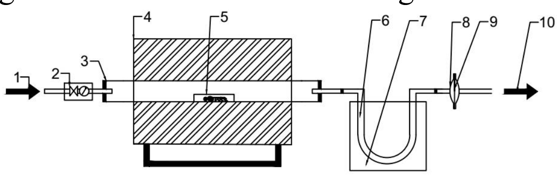

Figure 1. Schematic diagram of the tobacco pyrolysis reactor
1. Air inlet 2. Gas flow meter 3. Reaction tube 4. Furnace 5. Quartz boat 6. U-shaped condenser 7. Dry ice 8. Flange 9. Cambridge filter 10. Air bag

2.3 Analysis of condensable components in smoke

Gas Chromatography-Mass Spectrometer (GC-MS, ThermoFisher, Trace1300/ISQ) was used to characterize the condensable components in smoke, the quantification methods of the products were YC/T243-2008 and YC/T246-2008. The capillary column of GC-MS was HP-INNOWax (30 m × 0.25 mm × 0.25 μm). Before the test, the liquid product was diluted with a methanol/dichloromethane mixed solution, and the sample solution was filtered by a 0.45 μm filter. 1μL of the sample (concentrated to 1 wt%) was injected into the injection port in a splitless configuration. The column was operated in a constant flow mode using helium as the carrier gas (1 μL/min, purity > 99.99%). The temperature of the injection port was set at 250°C. The column was initially maintained at 40°C for 3 min before it was increased to 250°C at a heating rate of 5°C/min, and thereafter held for 10 min.

3. Results and discussion

3.1 Effects of acidic aroma substances on the release of smoke

3.1.1 Effects of lactic acid

The smoke released from the pyrolysis of tobacco samples with different contents of lactic acid was collected, and the composition and content of organic components in the smoke were analyzed by

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GC/MS. The results were shown in Figure 2. The results showed that the addition of lactic acid in tobacco had little effect on the release of aroma components in smoke, while it could still be observed that lactic acid slightly promoted the release of aroma components with the addition of 1.25 wt%. That might be due to the lactic acid being a weak acid. When the concentration of lactic acid added to the tobacco was low, the properties of the tobacco raw materials themselves changed very little. When the concentration of lactic acid in the tobacco sample increased, the acidity gradually also increased. If the tobacco sample was pretreated under stronger acidity, the structure and components of the tobacco sample would change more. For example, the content of hemicellulose in tobacco could be increased, and hemicellulose was more active, which made the tobacco components easier to be cracked, and released more smoke during pyrolysis [23]. By comparing the content of various aroma components in the smoke, it could be known that the release of acids, aldehydes, ketones, and alcohol aroma components in smoke obviously increased with the addition of lactic acid. When the additional amount of lactic acid was 1.25 wt%, the increase of acid aroma components was about 10 wt%, the increase of aldehyde aroma components was about 15 wt%, and the content of olefin and pyrrole aroma components increased slightly.

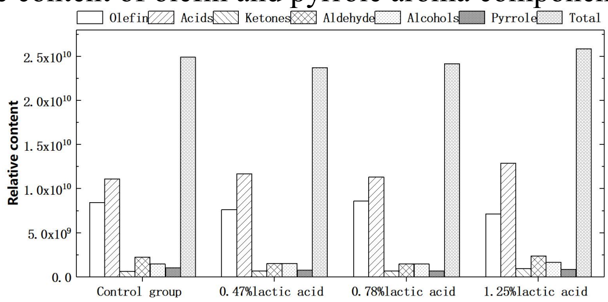

Figure 2. Content of the aroma components released from the pyrolysis of tobacco samples added with lactic acid

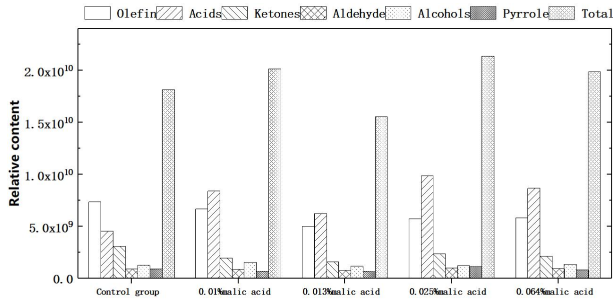

Figure. 3 Content of the aroma components released from the pyrolysis of tobacco samples added with malic acid

3.1.2 Effects of malic acid

The smoke released from the tobacco samples with different contents of malic acid was collected and analyzed by the GC-MS, as shown in Figure 3. The results showed that the addition of malic acid in tobacco significantly increased the release of aroma components in smoke, and the increased aroma components were mainly the acid aroma components, and the release of other types of aroma components did not change much. When the amount of malic acid added to tobacco was 0.025 wt%, the total amount of aroma components released via the pyrolysis increased by more than 20%, and the increase of acid aroma components reached nearly 40%, but the release of other types of aroma components decreased. Due to the stronger acidity of malic acid, free hydrogen ions would be generated during the acid pretreatment process of the tobacco sample. These free hydrogen ions could react with some components in the tobacco to release more free acids. Thus the content of acidic components in tobacco increased significantly. In addition, although acid pretreatment could increase the content of hemicellulose in tobacco and make it easier to pyrolyze, excessive acidity could inhibit the Maillard reaction during smoking. With the increase of acidity during pretreatment, the inhibition of the Maillard reaction strengthened, so fewer aroma components were produced.

3.1.3 The effects of citric acid

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The results of GC-MS characterization and analysis of smoke released from the pyrolysis of tobacco samples with different contents of citric acid were shown in Figure 4. The results showed that citric acid can significantly promote the thermal release of aroma components in tobacco, and only the addition of 0.01 wt% of citric acid could increase the release of aroma components by more than 30%. Among the increased aroma components, the increase in acid aroma components exceeded 50%, and the release of other types of aroma components had been slightly reduced. With the further increase of citric acid concentration, the release of aroma components gradually decreased. This was because the acidity of citric acid is stronger than that of malic acid. The addition of 0.01 wt% citric acid could significantly promote the release of acid aroma components. At the same time, the inhibitory effect on the Maillard reaction was also more obvious due to the stronger acidity of citric acid. Therefore, the release of aroma components in tobacco samples with high addition of citric acid decreased more significantly.

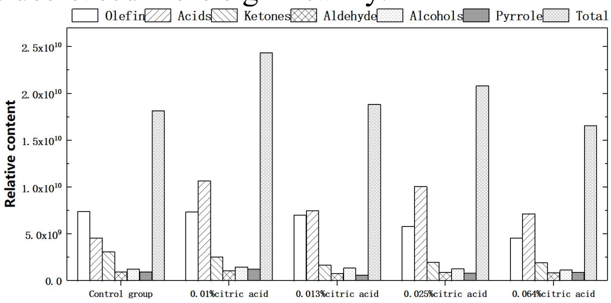

Figure. 4 Content of the aroma components released from the pyrolysis of tobacco samples added with citric acid

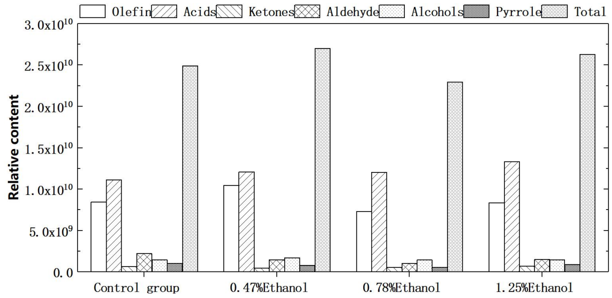

Figure. 5 Content of the aroma components released from the pyrolysis of tobacco samples added with ethanol

3.2 Effects of alkaline aroma substances

3.2.1 Effects of ethanol

The composition and content of aroma components in the smoke released from the pyrolysis of the tobacco samples prepared by adding different amounts of ethanol were shown in Figure 5. The results showed that the addition of ethanol to tobacco could increase the release of aroma components during the pyrolysis of tobacco. When the amount of ethanol added was 0.47 wt%, the release of olefins and alcohol aroma components would increase; but as the amount of ethanol increased, the release of various aroma components would decrease. Ethanol would promote the release of acid aroma components, and with the increase of the amount of ethanol in the tobacco sample, the release of acid aroma components would gradually increase. After the addition of ethanol, the aldehydes, ketones, and pyrrole aroma components released from the pyrolysis of the tobacco sample were reduced, and the reduction was most obvious when the amount of ethanol added was 0.78 wt%.

3.2.2 Effects of sodium bicarbonate

The results of GC-MS characterization and analysis of smoke released from the pyrolysis of tobacco samples with different contents of sodium bicarbonate were shown in Figure 6. The results showed that the addition of sodium bicarbonate into tobacco samples could promote the release of aroma components during smoking, and when the additional amount of sodium bicarbonate was

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0.06 wt%, the promotion effect was the most obvious; as the addition amount of sodium bicarbonate increased, the promotion effect was slightly reduced instead. Among various aroma components, sodium bicarbonate had a certain degree of increase in the release of acid and alcohol aroma components during tobacco pyrolysis, and the promotion trend was similar. The promotion effect was best when the addition amount of sodium bicarbonate was 0.06 wt%. Sodium bicarbonate also promoted the release of ketones and pyrrole aroma components, but as the additional amount of sodium bicarbonate increased, the promotion effect would decrease. This was because the sodium salt would be adsorbed on the surface of tobacco components in the form of sodium ions to catalyze the pyrolysis process of tobacco and make the tobacco components active, which could reduce the activation energy of the pyrolysis process, thereby accelerating the pyrolysis of tobacco components. At the same time, sodium bicarbonate had a certain alkalinity, which could generate some free hydroxide ions, which was conducive to the Maillard reaction and had a certain promotion effect on the release of alcohol aroma components[24].

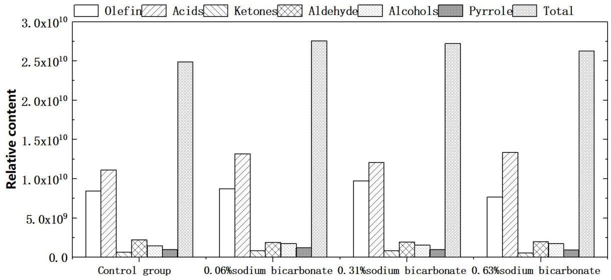

Fig. 6 Content of the aroma components released from the pyrolysis of tobacco samples added with sodium bicarbonate

3.2.3 Effects of potassium carbonate

The results of GC-MS characterization and analysis of smoke released from the pyrolysis of tobacco samples with different contents of potassium carbonate were shown in Figure 7. The results showed that the addition of potassium carbonate into tobacco could also greatly increase the release of aroma components during the pyrolysis process, and with the increase of potassium carbonate addition, the release of aroma components also increased. In the process of tobacco pyrolysis, potassium ions had a significant catalytic effect. The presence of potassium ions reduced the activation energy required for tobacco pyrolysis, the temperature required for decomposing the main components of tobacco was reduced, and the catalytic effects of potassium ions would change the evolution and chemical reaction pathways of macromolecular components such levoglucan, xylan and pyran rings and phenylpropane monomers. In addition, potassium ions would attach to the surface of the tobacco, changing the original specific surface area and pore density of the tobacco components, hindering the heat transfer inside the tobacco itself, inhibiting the rapid release of volatiles, and promoting the Secondary cracking of volatiles generated during the pyrolysis process[25-27]. Based on the above factors, the presence of potassium carbonate could catalyze the thermal cracking of tobacco and have the ability to significantly increase the amount of smoke released. However, when the additional amount of potassium carbonate was increased to 0.156 wt%, the amount of aroma components released began to decrease, indicating that the promotion effect of potassium carbonate on aroma components did not increase linearly, instead there was an optimal value. The best addition amount of potassium carbonate was between 0.031 and 0.156 wt%. Alkenes, acids, and ketones had similar thermal release behaviors. They all increased with the increase of potassium carbonate. The best release effect was achieved when the amount of potassium carbonate was increased to 0.078 wt%, and the release of aroma components increased by nearly 30%. The release of pyrrole aroma components also reached the maximum when the mass ratio of potassium carbonate was 0.078 wt%, but the presence of potassium carbonate would inhibit the release of aldehydes to a certain extent.

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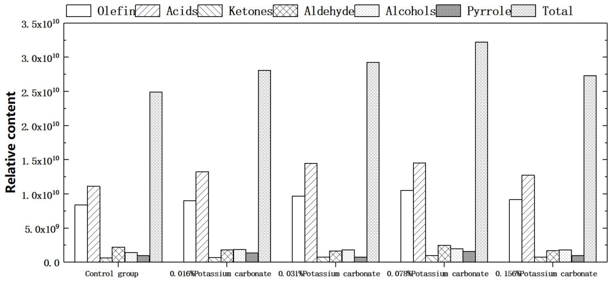

Figure. 7 Content of the aroma components released from the pyrolysis of tobacco samples added with potassium carbonate

3.3 The effect of synergistic effect between different aroma substances on smoke release

In order to further study the effects of various additives on the release of aroma components during the pyrolysis of tobacco, in this paper, we further studied the thermal release characteristics of aroma components from tobacco samples (as shown in Table 2) added with ethanol and lactic acid, ethanol and citric acid, lactic acid and citric acid, ethanol and malic acid, lactic acid and malic acid, respectively. A comparative analysis of the thermal release characteristics of aroma components in tobacco samples with the above-mentioned additives was carried out to clarify the effect of their synergistic effects on the release of aroma components when the additives were added together.

Table.2 The tobacco samples which were mixed with different aroma components used in this study

Contrast group	Additive1	Additive2	Mixed Additive3
1	0.47%Ethanol	0.94%Lactic acid	0.47%Ethanol+0.94%Lactic acid
2	0.47%Ethanol	0.013%Citric acid	0.47%Ethanol+0.013%Citric acid
2	0.94%Lactic acid	0.013%Citric acid	0.94%Lactic acid+0.013%Citric acid
4	0.47%Ethanol	0.013%Malic acid	0.47%Ethanol+0.013%Malic acid
5	0.94%Lactic acid	0.013%Malic acid	0.94%Lactic acid+0.013%Malic acid

The changes of aroma components released from the tobacco samples with mixed additives and the sum of those from the tobacco samples with a single additive were shown in Figure 8. The results showed that the synergy effects between Malic acid and Ethanol, Citric acid and Lactic acid during the pyrolysis process could further promote the thermal release of aroma components, and the promotion effect was greater than the sum of the promotion amount of the aroma components released when the two components were added separately. The synergy effects between Ethanol and Lactic acid, Malic acid, and Lactic acid had a certain inhibitory effect on the thermal release of aroma components, the promotion effect on the smoke released by the mixed additives was weaker than that of two components added separately. The effect of the mixed addition of Ethanol and Citric acid on the thermal release of aroma components was basically the same as that of single addition. There was only a weak inhibitory effect on the release of tobacco aroma components, so its synergy was weak. The effect of the mixed addition of ethanol and citric acid on the thermal release of aroma components was basically the same as that of single addition. There was only a weak inhibitory effect on the release of tobacco aroma components, so its synergy was weak. This was because ethanol and citric acid would neutralize with each other, which offsets the acid and alkali modification effects of tobacco samples that existed when they were added separately. Based on the above analysis, it could be seen that by adding different additives together, the synergy between the additives could be used to further increase the release of aroma components during the pyrolysis of tobacco.

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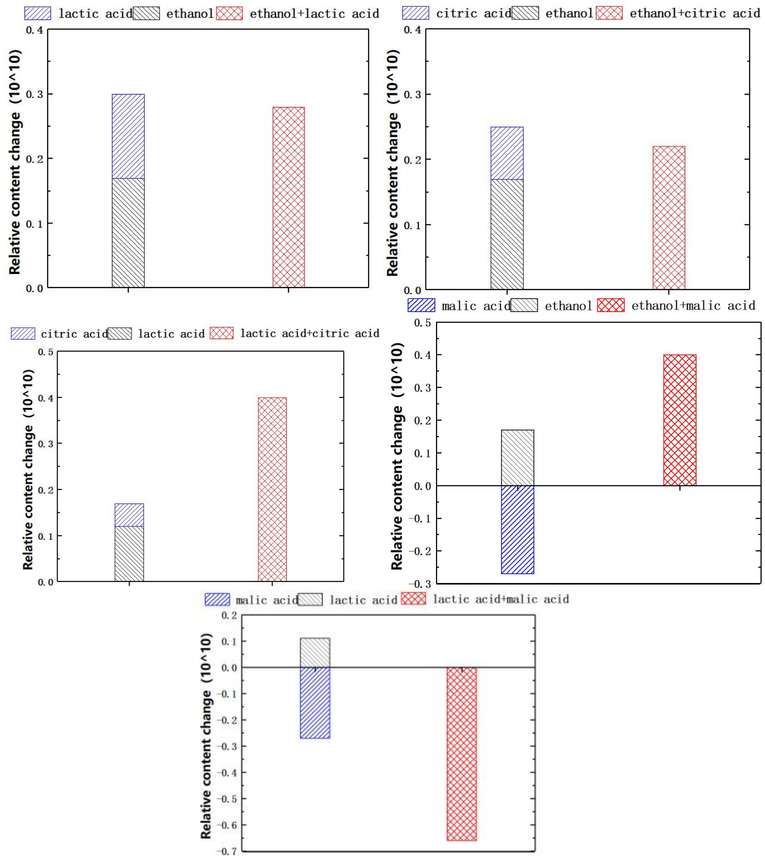

Figure. 8 Changes in the content of aroma components released from the pyrolysis of tobacco samples mixed with different aroma components.

4. Conclusion

This article studied the effects of adding lactic acid, citric acid, malic acid, ethanol, sodium bicarbonate, and potassium carbonate to tobacco on the release of aroma components from the pyrolysis of reconstituted tobacco during smoking, and analyzed the synergistic effects of the above-mentioned additives when they were added together. Research results showed that adding 0.078 wt% potassium carbonate or 0.01 wt% citric acid to tobacco raw materials could significantly increase the content of aroma components released during the pyrolysis of reconstituted tobacco. Compared with the original reconstituted tobacco samples, the samples added with potassium carbonate had the largest release amount of aroma components, and the amount of aroma components increased by nearly 30%. The main reason was that potassium ions could catalyze the thermal cracking of tobacco components, thereby promoting the thermal release of various aroma components. During the pyrolysis of reconstituted tobacco samples with two additives added at the same time, it was found that there was a synergistic effect between different additives, which further affects the thermal release of aroma components. Among these synergistic effects, the synergistic effect between malic acid and ethanol, citric acid and lactic acid further promoted the thermal release of aroma components; the synergistic effect between ethanol and lactic acid, ethanol and citric acid could inhibit the thermal release of aroma components; the synergistic effect between ethanol and citric acid was weak and had little effect on the release of aroma components. The research in this paper provided a theoretical basis and technical ideas for promoting the smoke-release effect of HNB cigarettes by adding modified components to the reconstituted tobacco of HNB cigarettes.

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