

Research Article

Study on Cut Tobacco Drying Process Based on HS-GC/MS: Principal Component Analysis, Similarity Analysis, Drying Conditions, and Drying Mechanism

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In order to study the volatile and semivolatile components changes of cut tobacco in the drying process, the cut tobacco were determined by headspace-gas chromatography/mass spectrometry (HS-GC/MS). Principal component analysis (PCA) and similarity analysis were used to analyze the HS-GC/MS results. Based on the similarity, the drying conditions and drying mechanism have been studied. The results showed that the increase in water content would reduce the drying rate, and if the temperature increased, the drying rate increased. The preliminary study on the drying kinetics of the drying process shows that Henderson and Pabis model appeared to be the most suitable for describing the drying process of cut tobacco, and the drying activation energy is 111.9 kJ.

1. Introduction

In the cigarette production process, the drying process is an important step [1–3]. The setting of the process parameters not only has a direct influence on cut tobacco physical characteristics, such as moisture content, whole cut rate, and filling power, but it also has a critical impact on the chemical properties, especially aroma components [1, 4–6]. There are many studies reported on the physical and chemical changes during the tobacco drying process [7–12]. Zhu has studied the two-stage drying of cut tobacco [7, 8]. The results show that the drying temperature and the moisture content not only affect the drying rate but also affect the characteristic flavor components of cut tobacco. When the temperature increased from 100°C to 110°C, both Maillard reaction compounds and carotenoid degradation products were decreased by 10%; Maillard reaction compounds decreased with moisture content decreased [7].

Then, the drying process was fitted with developed heat and mass transfer model. The temperature and moisture evolution of cut tobacco at different air temperature combinations were in good agreement with simulation results by the model [8]. Li et al. reported that the drying temperature increase can result in decrease of the petroleum ether tobacco extract [9]. In Wang et al.'s research on the influence of tobacco drying on the subsequent pyrolysis and combustion characteristics, the results showed that low-temperature drying (at 70 and 110°C) had little effect on the pyrolysis and combustion properties, but high-temperature drying (at 150 and 170°C) led to a considerable reduction of sugar while producing new components that are harder to decompose [10]. With increasing the degree of drying, such as increasing the drying temperature and prolonging the drying time, the total sugar, total nitrogen, and nicotine content in the tobacco are decreased, but the organic acid content is slightly increased, and the difference in ammonia is not large [10, 13]. Pakowski et al.

modeled the drying process of CLED (closed loop expansion dryer) cut tobacco and optimized the drying parameters. Under optimal conditions, physical properties of cut tobacco were improved [11]. Xin et al. studied the drying kinetics of cut tobacco with Arrhenius-type equation of diffusion [12]. It found that the heat capacity and the thermal conductivity increase with an increasing moisture content of the tobacco. During the drying process, high humidity steam can improve the filling power of the cut tobacco [13].

Headspace gas chromatography-mass spectrometry (HS-GC/MS) analysis is an indirect measurement for the detection and analysis of the volatile and semivolatile compounds of samples [14–17]. After heating, the solid (liquid) phase and the gas phase achieve thermal equilibrium and then extract headspace gas for qualitative and quantitative analyses [16, 17]. The volatile and semivolatile components in cut tobacco can not only reflect the composition of cigarettes' flavor and fragrance but are also closely related to the inherent quality and style of tobacco [16]. Thus, the volatile and semivolatile components in cut tobacco analysis are the basis and important reference for evaluating the quality of tobacco. Therefore, it is an effective manner to qualify the cut tobacco via HS-GC/MS.

In this paper, the cut tobacco chemical contents, before/after the drying process, were studied by HS-GC/MS and then analyzed with principal component analysis (PCA) and similarity analysis. It is first to use similarity as an analytical indicator, the effects of temperature, water addition, processing time, etc. The chemical changes of cut tobacco were systematically studied, during the drying process, while the drying thermodynamics and drying kinetics of the drying process were studied. The research will provide technical support for the determination of the process parameters of drying and the control of cut tobacco quality after processing.

2. Materials and Methods

2.1. Reagents. N-hexane (HPLC grade) was purchased from Sinopharm Chemical Reagent Co., Ltd. Deuterated toluene ($\geq 99\%$ purity), used as an internal standard, and was purchased from Shanghai Aladdin Biochemical Technology Co., Ltd.

Ultrapure water was produced from a Milli-Qs Integral system (Merck Millipore DE, Germany).

2.2. Samples Collection and Preparation. The cut tobacco, which is not added with any flavor and fragrance, is Hongtashan (classic) brand obtained from Yuxi Cigarette Factory. The cut tobacco are subjected to an in-line drying at drying oven (DHG-9140A Series, Youyi Instrument Company, Shanghai, China) to simulate the cut tobacco drying process, and the thickness of the sample is 3 mm. Take the cut tobacco, spray a predetermined amount of deionized water, then put it into the oven, drying at a preset temperature for a certain time, and take it out for use.

Accurately weigh 0.7 g of tobacco sample and place it in a 20 mL brown headspace vial. An aliquot of 10 mL of deuterated toluene ($0.25 \mu\text{g}/\mu\text{L}$ n-hexane) as an internal standard was added to each vial before testing.

2.3. HS-GC-MS Analysis. Agilent model 7890A-5975C series gas chromatography/mass spectrometer equipped with an Agilent model 7694E headspace sampler (Agilent Technologies, Santa Clara, California, USA). Chromatographic separation was performed on capillary column ($30 \text{ m} \times 0.25 \text{ mm I. D.}$, $0.25 \mu\text{m}$ film thickness, DB-5MS 5% Phenyl Methyl Siloxane, Agilent Technologies); 20 mL headspace vials were sealed air-tight with a silicone/aluminum septum.

2.3.1. Headspace Operating Conditions. The oven temperature was set at 70°C , the loop temperature was set at 110°C , the transfer line was set at 119°C , and the thermal equilibration time was 40 min with low shaking. The injection time was 0.2 min, and vial was pressurized to 20 psi in 2 min. The operating mode is constant.

2.3.2. GC-MS Operating Conditions. The injection mode: split ratio was 5:1. The following GC oven temperature was applied: initial oven temperature was set at 50°C and held for 2 min. The oven temperature was then increased to 220°C at a rate of $5^\circ\text{C}\cdot\text{min}^{-1}$. After that, the oven temperature was then increased to 250°C at a rate of $10^\circ\text{C}\cdot\text{min}^{-1}$ which was held for 1 min. The injector temperature was at 250°C , and a constant flow rate of $1.5 \text{ mL}\cdot\text{min}^{-1}$.

2.3.3. Mass Selective Detection Parameters. The EI ion source and transfer line temperatures were at 230°C and 250°C , respectively, and the quadrupole temperature set at 150°C . The MS operating conditions are positive electron ionization mode using automatic gain control with 70 eV of electron energy. The mass spectrometer was operated in scan mode from 40°amu to 450°amu . The solvent delay was set for 5.2 min.

2.4. Data Processing. Principal component analysis (PCA) analysis and cosine similarity calculation were performed using Chemmind Chempattern 2017 software.

3. Results and Discussion

3.1. HS-GC/MS Analysis of Cut Tobacco. The volatile and semivolatile compounds of cut tobacco were extracted and analyzed by HS-GC/MS. The sample was measured 7 times, respectively, and the relative peak areas were averaged. A total of 37 volatile and semivolatile aroma components were identified by HS-GC/MS in cut tobacco. The results are shown in Table 1. The aroma components in Table 1 are mainly derived from the cut tobacco in different drying processes. From Table 1, it can be found that in the drying temperature range, it is less change in the species of volatile compounds of the tobacco and mainly change in the composition of the contents. This indicates that the cut tobacco drying conditions are relatively mild, which makes less volatile compound species change. The results are consistent with Wang's research [10]. In Wang's article, it showed that low-temperature drying (below 150°C) scarcely

TABLE 1: Data of HS-GC/MS results of various drying parameters.

Compound name	Content ($\mu\text{g}/\text{kg}$)				
	CK	90°C	100°C	115°C	120°C
Cyclopentane, methyl-	0	0	56.64	0	0
Isovaleraldehyde	2.49	2.79	2.48	3.98	0.8
Butanal, 2-methyl- + acetol	1.26	3.1	3.59	20.22	5.44
Propanoic acid	0.77	1.53	1.2	2.12	0.53
1-Penten-3-one	0.37	0.5	1.39	0.06	0.03
2,3-Pentanedione	0.69	1.07	0	6.82	1.89
2-Butanone, 3-hydroxy-	0.44	0.43	0.5	2.64	0.64
2,3-Butanediol (Z)	0.32	1.23	0.82	1.33	0.3
2,3-Butanediol (E)	0.39	2.09	1.72	1.49	0.33
Hexanal	0.45	0.34	0.32	0.42	0.1
3(2H)-Furanone, dihydro-2-methyl-	0.52	0.74	0.93	6.57	1.35
Furfural	1.91	3.46	5.11	22.04	9.44
Furfuryl alcohol	1.13	2.18	3.31	24.15	7.93
2-Propanone, 1-(acetyloxy)-	0.12	0.19	0.43	2.51	0.72
2-Cyclopentene-1,4-dione	0.16	0.49	0.65	2.92	1.08
2-Acetylfuran	0.11	0.14	0.27	2.42	0.91
γ -Butyrolactone	0.13	0.33	0.39	1.75	0.53
2-Furanmethanol, 5-methyl-	0.14	0.18	0.27	1.41	0.28
Benzaldehyde	0.2	0.15	0.13	2.7	1.05
Furfural, 5-methyl-	0.06	0.14	0.22	2.7	1.05
2,4-Dihydroxy-2,5-dimethyl-3(2H)-furan-3-one	0.27	0.73	0.84	3.54	1.16
5-Hepten-2-one, 6-methyl-	0.33	0.4	0.38	0.85	0.24
Benzyl alcohol	0.35	0.36	0.23	2.51	0.17
Benzeneacetaldehyde	0.48	0.57	0.52	0.55	0.22
2-Acetylpyrrole	0.26	0.38	0.37	0.97	0.23
Pentanoic acid, 2-methyl-, anhydride	0.1	0.33	0.19	0.25	0.09
Undecane	0	0.28	0.26	0.2	0.11
2-Decene, 5-methyl-	0.09	0.14	0.12	0.1	0.05
Phenylethyl alcohol	0.13	0.17	0.17	0.28	0.02
4H-Pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl-	5.45	8.75	9.42	48.52	18.21
Dodecane	0	0.6	0.52	0.57	0.36
Hexadecane	0	0.19	0.17	7.01	0.16
Nicotine	132.3	239.7	188.2	204.7	81.6
Solanone	1.04	1.46	1.69	4.03	1.06
Actinidiolide, dihydro-	0.08	0.14	0.08	0.14	0.03
Maltolazine	1.34	2.61	1.61	7.1	0.4
Neophytadiene	2.49	47.82	39.28	30.18	16.27

producing new components, which had little effect on the pyrolysis and combustion properties of cut tobacco.

3.2. Principal Component Analysis of Cut Tobacco. To study the cut tobacco aroma composition changes during the drying process, the principal component analysis was applied [18, 19]. The HS-GC/MS data of cut tobacco were introduced into the software ChemPattern 2017 Pro for principal component analysis. Figure 1 is a principal component analysis diagram of cut tobacco for 90 min at different drying temperatures. The first principal component (PC1) explained 76.87% of total variability, and the second principal component (PC2) explained 8.94%, the total contribution rate is 85.81%, and this indicates that these first two components could present the majority of all significant composition changes among the samples.

The PC1/PC2 scores plot (Figure 1) shows that four distinct clusters are formed and distributed over four regions, corresponding to the four different drying temperatures studied.

Therefore, the influence of different parameters on the drying process can be distinguished by PCA analysis of the HS-GC/MS components of cut tobacco. However, it should be noted that distances between the samples are different, and the accurate difference value could not be observed from PCA.

3.3. Effects of Different Drying Conditions on Cut Tobacco Basis on the Similarity Analysis. To distinguish the changes accurately, it was necessary to quantify the difference between the HS-GC/MS results [18, 19]. This can be achieved by the calculation of cosine similarities value between the drying tobacco and the untreated tobacco. Then, using similarity as an analytical indicator, the effects of different process parameters on cut tobacco have been studied.

Figure 2 is a plot showing the similarity change of cut tobacco for 90 min at different drying temperatures. In Figure 2, it can be seen that as the drying temperature increases, the similarity of cut tobacco decreases, indicating that as the temperature increases, the change of volatile

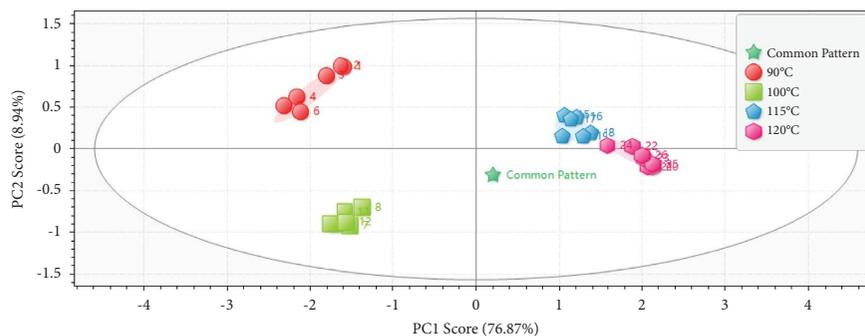


FIGURE 1: PCA of cut tobacco by drying at different drying temperatures (90°C, 100°C, 115°C, 120°C).

composition increases, and the tobacco drying degree increases. Also, when the drying temperature is higher than 115°C, the drying rate sharply increases, and the tobacco's volatile composition changes significantly.

Through external added water, the effect of humidity on the similarity of volatile components of cut tobacco after the drying process was studied. The similarity of the volatile components of cut tobacco drying at 115°C for 90 min, after adding 10% and 20% water, respectively, is shown in Figure 3. In Figure 3, it can be seen that the moisture content of cut tobacco increases, and the change of cut tobacco decreases, indicating that the moisture has a reducing effect on drying rate in drying process. This similar phenomenon also occurred in Xin's drying relative humidity research [12], and in their paper, it can be found that the higher the relative humidity was, the lower rate of reduction in moisture content was.

The effects of drying time on the similarity of tobacco volatile components were studied under the conditions of a drying temperature of 115°C. The results are shown in Figure 4. It can be found that with the increase of the drying time, the similarity of the volatile components of the tobacco has a tendency to decrease, indicating that the drying degree of the drying process increases with time; in addition, it can be seen that the similarity of tobacco volatile components shows a well linear correlation with the drying time in Figure 4. Therefore, it can be approximated that the process of changing the similarity of tobacco volatile components during the drying process is a one-dimensional linear process.

3.4. Preliminary Study on Drying Kinetics and Thermodynamics of Cut Tobacco. The thin-layer drying mathematical model is usually used to describe the drying process of agricultural products [12, 20, 21]. The drying process is also a water diffusion process which can be explained by Fick's diffusion law. Based on Fick's second law, there are three drying empirical equations often used for curve fitting of experimental data (shown in Table 2). At the same time, it is obvious that the greater the drying degree of the tobacco, the greater the similarity of the volatile components of the tobacco changes, and to some extent, they are correlated. Therefore, it can be studied the drying process by studying the change in the similarity of the volatile components of cut tobacco.

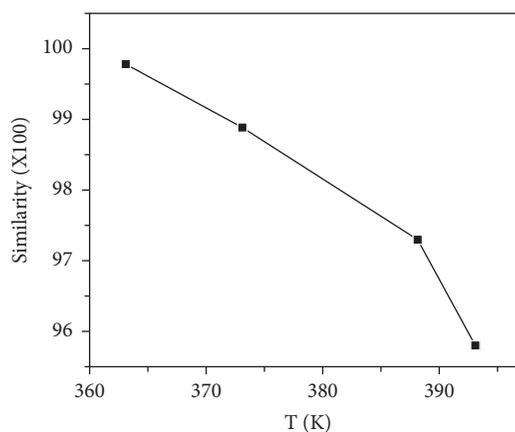


FIGURE 2: Similarity of cut tobacco after different drying temperatures (90°C, 100°C, 115°C, and 120°C).

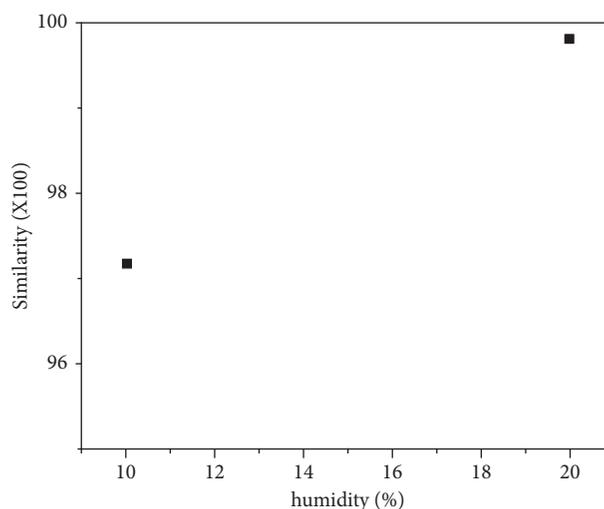


FIGURE 3: Similarity of cut tobacco after different drying humidity (10%, 20%).

As mentioned previously, the similarity of tobacco volatile components changes during the drying process can approximate to be a one-dimensional linear equation. Comparing the three commonly used thin-layer drying models (shown in Table 2), the similarity-time experimental drying data can be fitted to Henderson and Pabis model

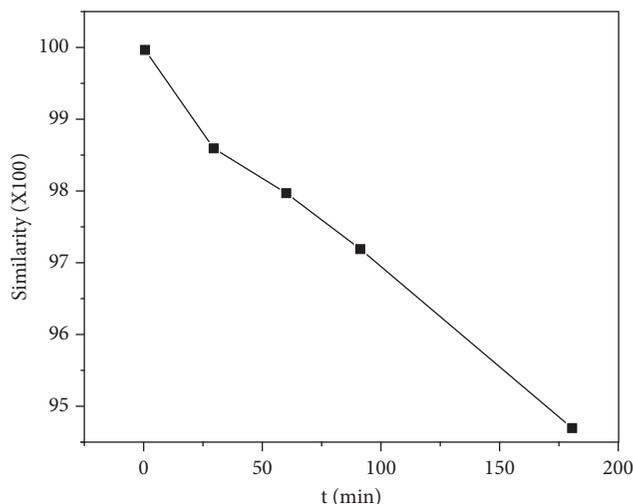


FIGURE 4: Similarity of cut tobacco after different drying times (30 min, 60 min, 90 min, 180 min).

TABLE 2: Thin-layer drying models.

Models	Model equation*	Linear equation*
Lewis	$MR = \exp(-kt)$	$\ln(MR) = -kt$
Page	$MR = \exp(-kt^n)$	$\ln[-\ln(MR)] = \ln k + n \ln t$
Henderson and Pabis	$MR = A \exp(-kt)$	$\ln(MR) = \ln A - kt$

*The MR is the moisture ratio, and the A , k , n are constants.

equation $\ln(MR) = \ln A - kt$, and the $S \propto \ln(MR)$, the slope of the $S-t$ curve K is proportional to k . Then, the drying kinetic equations at 90°C, 100°C, and 120°C, as well as the drying kinetics at 115°C, were fitted by Henderson and Pabis model equation. The equation was fitted, and the fitting results are shown in Table 3.

On the other hand, according to Fick's diffusion law, $\ln(MR)$ can be expressed by the following equation (1):

$$\ln(MR) = \ln \frac{8}{\pi^2} - \frac{\pi^2 D_{\text{eff}}}{4L^2} t, \quad (1)$$

where L is the average thickness of the tobacco; D_{eff} is the water-effective diffusivity.

According to Arrhenius equation [12, 22], the effective diffusivity was always described by (3).

$$D_{\text{eff}} = D_0 \exp\left(-\frac{E_a}{RT}\right). \quad (2)$$

After the natural logarithmic, D_{eff} can be written as follows:

$$\ln D_{\text{eff}} = \ln D_0 - \frac{E_a}{RT}, \quad (3)$$

where D_0 is the prefactor; E_a is the energy activation; R is the gas constant, $8.314 \text{ J mol}^{-1} \text{ K}^{-1}$; T is the Kelvin temperature, K .

TABLE 3: First-order reaction model for similarity of cut tobacco volatile components at different drying temperatures.

T(K)	Linear equation**	R^2
363.15*	$S = -0.0022 t + 100$	1
373.15*	$S = -0.0122 t + 100$	1
388.15	$S = -0.0281 t + 99.761$	0.994
393.15*	$S = -0.0459 t + 100$	1

*Fitting by two points method. **where S is the similarity of tobacco volatile components, and t is the reaction time.

Comparing equation (1) and Henderson and Pabis model equation, we can get that the D_{eff} is proportional to k . And the slope of the $S-t$ curve K is proportional to k . Then, D_{eff} is proportional to K .

$$\ln D_{\text{eff}} = \ln D_0 - \frac{E_a}{RT} = \ln K + C. \quad (4)$$

By fitting plotted $\ln K$ against $1/T$, the plot is shown in Figure 5, and we obtained the regression equation of $\ln K$:

$$\ln K = 31.21 - \frac{13456.43}{T}. \quad (5)$$

According to equations (4) and (5), the D_{eff} can be expressed as follows:

$$\ln D_{\text{eff}} = \ln D_0 - \frac{E_a}{RT} + C = 31.79 - \frac{13456.43}{T}. \quad (6)$$

The activation energy E_a of the drying process can be obtained as presented in (1) and (6). Through calculation, the drying process activation energy E_a is 111.9 kJ, and the reaction is an endothermic reaction. As shown in Figure 5, it can be seen that the regression correlation index $R^2 = 0.9455$, and the fitting degree is relatively high, indicating that the whole inferred calculation process is reasonable, which is close to the actual experiment.

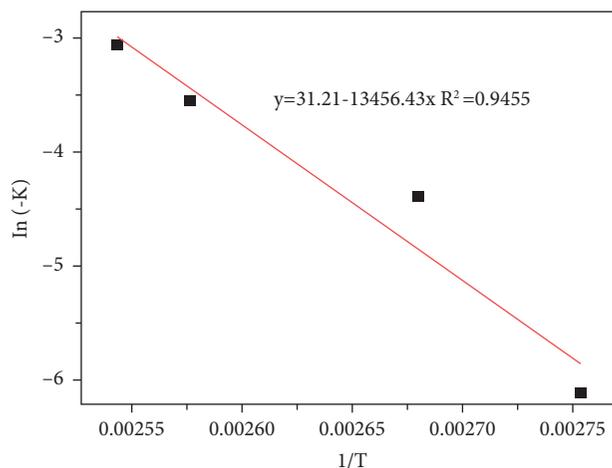


FIGURE 5: $\ln K - 1/T$ regression equation curve.

4. Conclusions

The cut tobacco drying process is very important in the cigarette production process. In order to study the drying mechanism of cut tobacco in the drying process, the volatile and semivolatile components of cut tobacco under different drying processes were determined by headspace-gas chromatography/mass spectrometry. Principal component analysis and similarity analysis were used to analyze and process the HS-GC/MS data. The similarity has been used to analyze the drying degree. Based on the similarity, it systematically studies the drying conditions such as temperature, time, and humidity, influence on the volatile components of tobacco. Furthermore, the drying kinetics and thermodynamics of the drying process were discussed preliminarily via Fick's diffusion law and the Arrhenius formula.

The results show that the change of the volatile and semivolatile chemical kinds of cut tobacco is small, but mainly composition content changed, under the conditions. Principal component analysis shows that different drying process tobacco can be clearly identified. Drying process study shows that an increase in water content will reduce the rate of drying reaction, and as time and temperature increase, the drying degree increases. The preliminary study on the drying kinetics of the drying process shows that the Henderson and Pabis model appeared to be the most suitable for describing the drying process of cut tobacco, and the energy of activation is 111.9 kJ,

The article developed new research methods for drying process and provided theoretical support for the selection of the cut tobacco drying process parameters.

Data Availability

The reference data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors are affiliated to China Tobacco Yunnan Industrial Co. and funded by China National Tobacco

Corporation, Ltd. The authors attest China Tobacco Yunnan Industrial Co., Ltd. and China National Tobacco Corporation have had no influence on the design of this study or its outcomes.

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