Effects of Threshing, Redrying with Collaborative Aging on the Conventional Chemical Components of Tobacco Leaves

Yaping Ma[#], Minchang Liu[#], Xiaodong Gu, Tingjie Bao, Ruifang Gu, Jiaxi Ni, Lingying He, Zhengrong Zeng, Xiaoqin Yang, Rongya Zhang[a*], Wu Wen[b*]

Technology Center, China Tobacco Sichuan Industrial Co., Ltd., Section 2, Chenglong Avenue, Longquanyi District, Chengdu, Sichuan 610100, China
[#]Co-first author

Corresponding Authors: [a*]rongyazhang@126.com, [b*]wenwu@sctobacco.com

Abstract. To enhance the quality of tobacco leaves, the suitable conditions for the coordination of threshing, redrying with collaborative aging were investigated. Simple correlation analysis and one-way ANOVA were employed to analyze the effects of threshing and redrying parameters and aging time on the conventional chemical components of tobacco leaves. The results indicated that: (1) As the aging time (0-30 months) increased, the reducing sugar generally showed a fluctuating trend of first rising and then falling, the total sugar generally showed a trend of first decreasing and then fluctuating slightly, the total plant alkaloids showed no significant change, the total nitrogen generally showed an "M" shaped change trend, the potassium generally showed a slight trend of first stabilizing, then increasing, then decreasing, and then stabilizing, and the chlorine generally showed a trend of first stabilizing, then decreasing, and then increasing. The reducing sugar and potassium both reached their peak at 12 months, the total sugar and total nitrogen reached their trough at 18 months, and the chlorine reached its trough at 24 months. (2) The key parameter a1 of threshing and redrying mainly positively regulated the reducing sugar, total sugar, and chlorine, a2 mainly negatively regulated the total plant alkaloids and positively regulated the potassium, a3 mainly positively regulated the total sugar, and a4 mainly positively regulated the reducing sugar, total sugar, and negatively regulated the chlorine. The key parameters of threshing and redrying had no regulatory effect on the total nitrogen. The regulation of chlorine by the key parameters of threshing and redrying mainly occurred during the threshing and redrying stage, while the regulation of reducing sugar, total sugar, total plant alkaloids, and potassium continued into the aging process. The study effectively regulates the conventional chemical components of tobacco leaves by optimizing the parameters of leaf threshing and redrying, as well as the aging time. This significantly enhances the aroma and taste quality indicators of the tobacco leaves, while also improving their usability. It provides important technical guidance and application value for the tobacco industry.

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1 Introduction

Threshing and redrying refer to the process activities that involve vacuum rehumidification, bunch control, first rehumidification, impurity removal, second rehumidification, leaf-stem separation, and leaf redrying, which are beneficial for maturation and storage [1]. The effects of threshing and redrying on aroma compounds, conventional chemical components, amino acids, polyphenols, and sensory quality have been extensively studied [2-4]. Yang Jie et al. [5] found that among the main phenolic compounds in tobacco leaves, the order is B3F > C3F > X2F, and the factors affecting the release of phenols are redrying > first rehumidification > second rehumidification (for B3F and X2F), and first rehumidification > redrying > second rehumidification (for C3F). Lu Youxiang et al. [6] demonstrated that among the key parameters of redrying, the moisture content of the feed and the hot air temperature are the main factors affecting sensory quality. Minghai Long et al. [7] showed that low-temperature slow redrying of tobacco leaves can improve moisture content and color uniformity, reduce the degradation rate of tobacco colorants and polyphenol precursors, and is more suitable for medium and high-grade tobacco compared to high-temperature redrying and steam-water mixture redrying. Aging is an important step in improving the quality of tobacco leaves, playing a significant role in improving appearance, coordinating chemical components, and enhancing sensory quality [8]. Aging has significant effects on aroma compounds, conventional chemical components, sensory quality, and microbial colonies [9-14]. Liu Hongguang et al. [15] found that the sensory quality and chemical component usability index of redried tobacco leaves both show an initial increase followed by a decrease as the aging time extends. The suitable aging time for initial tobacco leaves is 3 to 5 months, and for redried tobacco leaves, it is 15 months. Wang Peng et al. [16] observed that the total amount of amino acids decreases during the aging process, but the rate of decrease gradually diminishes. The impact of redrying process parameters on amino acids is less significant than that of aging time. Zhou Ping et al. [17] found that the changes in conventional chemical components vary greatly during the drying process at different temperatures.

However, research on the combined process of threshing, redrying, and aging is currently scarce. P Previous research on threshing and redrying generally ends before the redrying and packaging process, while research on aging begins after the tobacco is stored in the warehouse. There is a disconnect between threshing and redrying and aging, and there is a lack of corresponding relationships. Additionally, research on the effects of key parameters of threshing and redrying on the conventional chemical components of tobacco leaves is still insufficient. Therefore, this study focuses on B2F, C3F, and X2F grades of Yunyan 85 from the Liangshan production area, investigating the impact of key threshing and redrying parameters and aging on the conventional chemical components of tobacco leaves. This provides direction for targeted regulation of conventional chemical components of tobacco leaves.

2 Materials and Methods

2.1 Materials

Three grades of initial flue-cured tobacco leaves from the Liangshan production area in Sichuan, purchased by Sichuan Zhongyan Industrial Co., Ltd. in 2012, were used in this study. The grades were B2F (upper), C3F (middle), and X2F (lower), all of the Yunyan 85 variety.

2.2 Experimental Equipment

The redrying line at Hui Li Redrying Factory in Sichuan.

2.3 Methods

2.3.1 Uniform Design Method

Uniform design is a method of experimental design that selects representative experimental points from a comprehensive set of experimental points. These points are evenly dispersed within the experimental range, reflecting the main characteristics of the system. Unlike orthogonal design, which emphasizes "neat comparability," uniform design focuses on "even distribution." Therefore, compared to orthogonal design, uniform design offers better uniformity of experimental points and requires fewer experimental runs.

2.3.2 Preparation of Redried Tobacco Samples

After conducting a literature review [5-6] and combining it with actual production, it has been determined that the temperature of the first moisturizing process and the temperatures of the first to fourth zones of the redrying process have a significant impact on the aroma components and sensory quality of tobacco leaves. Therefore, the temperature of the first moisturizing process and the temperatures of the first to fourth zones of the redrying process have been identified as experimental parameters. The temperature of the first moisturizing process is obtained by using temperature sensors to monitor the outlet temperature of the tobacco leaves in real time during the moisturizing process. The temperatures of the first to fourth zones of the redrying process are recorded and adjusted in real time by installing thermocouples in each drying zone of the redrying machine.Samples were prepared on the redrying line at Hui Li Redrying Factory in Sichuan. The process parameters for the first rehumidification and drying zones are shown in Tables 1 to 3. The process parameters for the other steps were not adjusted and were executed according to the current process parameters for each grade. To ensure the uniformity and consistency of the raw materials, manually selected initial flue-cured tobacco bundles were used, with each arrangement station placing the same total quantity and ending the arrangement simultaneously. The three grades of initial flue-cured tobacco leaves were processed according to the adjusted processing schemes, resulting in 18 batches of redried tobacco flakes, which were labeled as B0 to B5, C0 to C5, and X0 to X5, respectively.

Table 1. Experimental Parameter Table of B2F

Sample Number	R (°C)	Tobacco Leaf Redrying Drying Zone Temperature (°C)			
		First Zone	Second Zone	Third Zone	Fourth Zone
B0	50	60	73	69	65
B1	50	70	85	61	55
B2	65	65	55	73	60
B3	60	50	79	67	70
B4	45	55	71	80	50
B5	40	60	63	55	65

Table 2. Experimental Parameter Table of C3F

Sample Number	R (°C)	Tobacco Leaf Redrying Drying Zone Temperature (°C)			
		First Zone	Second Zone	Third Zone	Fourth Zone
C0	55	63	78	75	65
C1	50	70	85	61	55
C2	65	65	55	73	60
C3	60	50	79	67	70
C4	45	55	71	80	50
C5	40	60	63	55	65

Table 3. Experimental Parameter Table of X2F

Sample Number	R (°C)	Tobacco Leaf Redrying Drying Zone Temperature (°C)			
		First Zone	Second Zone	Third Zone	Fourth Zone
X0	58	55	67	65	60
X1	50	70	85	61	55
X2	65	65	55	73	60
X3	60	50	79	67	70
X4	45	55	71	80	50
X5	40	60	63	55	65

R: Tobacco Leaf Outlet Temperature of the First Rehumidification Process

2.3.3 Sampling of Tobacco Samples After Threshing, Redrying, and Aging, and Preparation of Cigarette Samples

Samples were taken from the 18 batches of finished tobacco flakes (B0-B5, C0-C5, and X0-X5) after threshing and redrying using a method of taking small amounts multiple times. During sampling, any insect spots, impurities, and foreign objects were removed to ensure the purity of the samples. The quartering method was used to retain the samples. Sampling after threshing and redrying was considered the zeroth sampling, with an aging time of 0. Additionally, five pieces of each finished product after redrying were selected and transported to the Liziyuan warehouse in Shifang for aging. Starting from the time the tobacco flakes were stored in the warehouse, one box was randomly selected for sampling each time (excluding boxes that had been sampled previously). Samples were taken from three points: top, middle, and bottom, with sampling conducted every six months for a total of six cycles.

2.3.4 Sample Analysis

In accordance with the tobacco industry standards, the contents of total sugar [18], reducing sugar [18], total plant alkaloids [19], chlorine [20], potassium [21], and total nitrogen [22] in tobacco leaves were determined using the continuous flow method.

2.4 Data Statistical Analysis

The uniform design method was used to design experiments for key parameters of threshing and redrying. Six conventional chemical components were measured in upper, middle, and lower tobacco leaves aged from 0 to 30 months. The data were statistically analyzed using simple correlation analysis, canonical correlation analysis, and one-way analysis of variance with SPSS 21.0.

3 Results and Analysis

3.1 Effects of Aging Time on Conventional Chemical Components

Figures 1 to 6 show the changes in six conventional chemical components of tobacco leaves from different threshing and redrying key parameters and three different positions as the aging time varies. It can be observed that the reducing sugar content ranges from 18% to 27%. The results of one-way ANOVA for reducing sugar in different positions indicate that there is no significant difference in reducing sugar among different positions at aging times of 0, 12, 24, and 30 months ($P > 0.05$). However, at aging times of 6 and 18 months, there is a significant difference between the middle and upper/lower positions ($P < 0.05$), while there is no significant difference between the upper and lower positions ($P > 0.05$). The average reducing sugar content is highest in the middle position, followed by the upper and lower positions. Reducing sugar is closely related to the quality of tobacco leaves, which is consistent with the superior quality of middle position tobacco leaves compared to the upper and lower positions. Overall, the reducing sugar content shows a fluctuating trend of first increasing and then decreasing, and it reaches its highest level at 12 months of aging. This may be due to the combined effects of the Maillard reaction and microbial growth metabolism, which consume reducing sugar, as well as the hydrolysis of starch and total sugar to produce reducing sugar. This trend is consistent with the research results of Hu Yajie et al. [23-24].

The total sugar content ranged from 19% to 31%. The results of one-way ANOVA for total sugar in different positions indicate that at an aging time of 0 months, there was a significant difference between the lower position and the upper and middle positions ($P < 0.05$), while there was no significant difference between the upper and middle positions ($P > 0.05$). At aging times of 6 and 30 months, there was a significant difference between the middle and lower positions ($P < 0.05$), but no significant difference between the upper position and the middle and lower positions ($P > 0.05$). At aging times of 12, 18, and 24 months, there were no significant differences between the middle, upper, and lower positions ($P > 0.05$). The average total sugar content was higher in the upper and middle positions than in the lower position. The total sugar content has an important impact on the quality of tobacco leaves, and within a certain range, it is positively correlated with the quality of tobacco leaves, which is consistent with the general observation that upper and middle positions are superior to the lower position. Overall, the total sugar content showed a trend of first decreasing and then fluctuating slightly, reaching its maximum at 0 months of aging and a lower level at 18 months of aging. The study by Hu Yajie et al. [24] also showed a trend of a significant initial decrease in total sugar followed by a slowdown.

The total plant alkaloids ranged from 1.6% to 3.4%. The results of one-way ANOVA for total plant alkaloids in different positions indicate that at aging times of 0, 12, 18, and 24 months, there were significant differences between the upper position and the middle and lower positions ($P < 0.05$), while there were no significant differences between the middle and lower positions ($P > 0.05$). At aging times of 6 and 30 months, there were significant differences among the upper, middle, and lower positions ($P < 0.05$). One-way ANOVA was conducted separately for the total plant alkaloids in the upper, middle, and lower positions at different aging times, and it was found that there were no significant differences among the total plant alkaloids at different aging times ($P > 0.05$). This suggests that aging time has little effect on total plant alkaloids, which is consistent with the research results of Wu Xinying et al. [25] .

The total nitrogen content ranged from 1.7% to 3.0%. The results of one-way ANOVA for total nitrogen in different positions indicate that at aging times of 0, 6, 18, 24, and 30 months, there were significant differences among the middle, upper, and lower positions ($P < 0.05$). At an aging time of 12 months, there were significant differences between the upper position and the middle and lower positions ($P < 0.05$), but no significant difference between the middle and lower positions ($P > 0.05$). The average total nitrogen content was highest in the upper position, followed by the middle and lower positions. Total nitrogen primarily affects the physiological strength of the smoke during smoking, and excessive amounts can increase irritation, which generally decreases from the upper to the lower positions of the tobacco leaves. Overall, the total nitrogen content showed a trend of first increasing, then decreasing, and then increasing and decreasing again, forming an "M" shape. During the aging period of 12-18 months, the total nitrogen content was relatively low. The fluctuation of total nitrogen may be related to the Maillard reaction involving amino compounds during the aging process.

The potassium content ranged from 1.3% to 2.6%. The results of one-way ANOVA for potassium in different positions indicate that at an aging time of 0 months, there were significant differences between the upper position and the middle and lower positions ($P < 0.05$), while there were no significant differences between the middle and lower positions (P > 0.05). At an aging time of 12 months, there were significant differences between the upper and middle positions (P < 0.05), but no significant differences between the lower position and the upper or middle positions (P > 0.05). At aging times of 6, 18, 24, and 30 months, there were significant differences among the upper, middle, and lower positions (P < 0.05). The average potassium content was higher in the middle and lower positions than in the upper position. Potassium content is closely related to combustibility, and within a reasonable range, higher potassium content leads to better combustibility. Overall, the potassium content showed a trend of first stabilizing, then reaching a maximum at 12 months, followed by a decrease and then stabilization. The changes were relatively small. The results of one-way ANOVA indicate that there was a significant difference between the 12-month aging time and other aging times (P < 0.05), while there were no significant differences for the others (P > 0.05).

The chlorine content ranged from 0.04% to 0.46%. The results of one-way ANOVA for chlorine in different positions indicate that at aging times of 0, 6, 12, and 18 months, there were significant differences between the middle position and the upper and lower positions (P < 0.05), while there were no significant differences between the upper and lower positions (P > 0.05). At aging times of 24 and 30 months, there were significant differences among the upper, middle, and lower positions (P < 0.05). The average chlorine content was higher in the upper and lower positions than in the middle position. The chlorine content is closely related to combustibility, and within a reasonable range, lower chlorine content leads to better combustibility. Generally, the potassium-to-chlorine ratio is used to characterize combustibility and tobacco leaf quality, with the ratio being highest in the middle position and lower in the upper and lower positions. Overall, the chlorine content was relatively stable initially, began to decrease at 18 months of aging, reached its minimum at 24 months, and then increased.

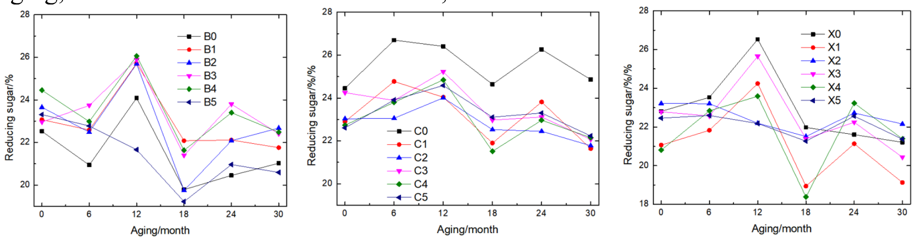

Fig. 1. Changes in Reducing Sugar Content in Upper, Middle, and Lower Positions at Different Aging Times

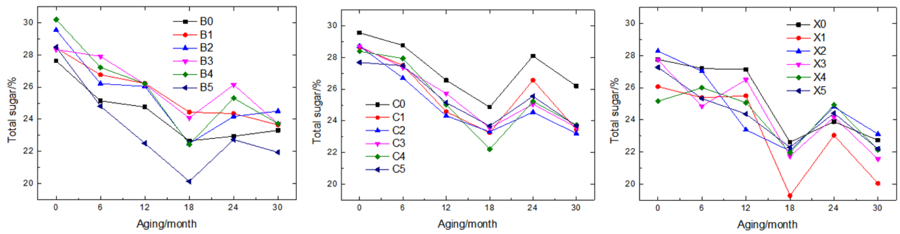

Fig. 2. Changes in Total Sugar Content in Upper, Middle, and Lower Positions at Different Aging Times

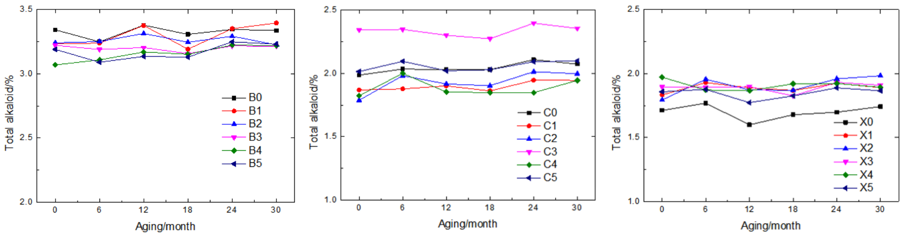

Fig. 3. Changes in Total Plant Alkaloid Content in Upper, Middle, and Lower Positions at Different Aging Times

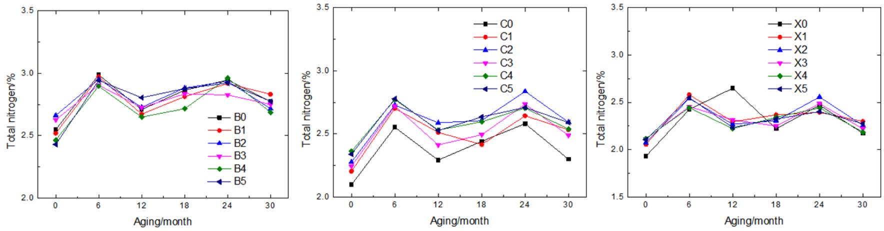

Fig. 4. Changes in Total Nitrogen Content in Upper, Middle, and Lower Positions at Different Aging Times

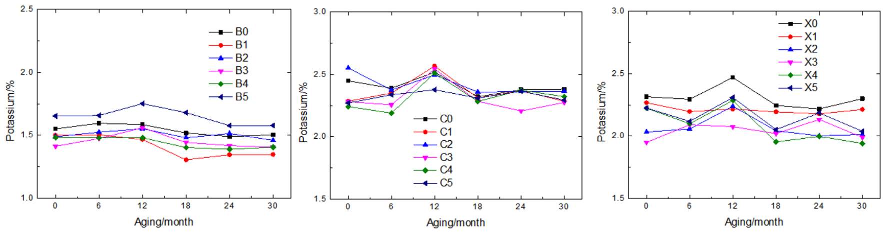

Fig. 5. Changes in Potassium Content in Upper, Middle, and Lower Positions at Different Aging Times

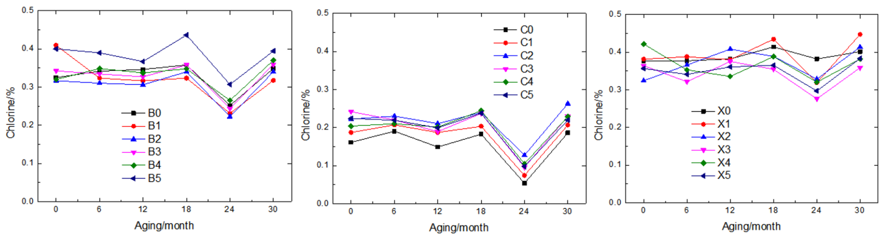

Fig. 6. Changes in Chlorine Content in Upper, Middle, and Lower Positions at Different Aging Times

3.2 Effects of Key Parameters of Threshing and Redrying on Conventional Chemical Components

To investigate the effects of key parameters of threshing and redrying (a1: temperature of tobacco leaves at the exit of the first moistening process, a2-a5: temperatures of the first, second, third, and fourth zones in the redrying drying area; same below) on six conventional chemical components (reducing sugar (RS), total sugar (TS), total plant alkaloids (TA), total nitrogen (TN), potassium (K), and chlorine (Cl)), one-way ANOVA was conducted on the reducing sugar, total sugar, total plant alkaloids, total nitrogen, potassium, and chlorine of tobacco leaves from different positions at the same alcoholization time (all data passed the single-sample non-parametric K-S test). The significance of differences was determined based on the P-value results using the Bonferroni method. To ensure sufficient data for statistical analysis, data from different positions with $P > 0.05$ according to the Bonferroni method were selected. This approach can be considered as excluding the influence of position on each conventional chemical component, making the key parameters of threshing and redrying the only variable.

Tables 4 to 9 show the simple correlation coefficients between the key parameters of threshing and redrying (a1 to a5) and the six conventional chemical components. It can be observed that, overall, reducing sugar is weakly positively correlated with a1 and a4, and the key parameters of threshing and redrying have a greater impact on reducing sugar during the aging period of 6-12 months. Specifically, a1 and a3 are moderately correlated with the aging time of 12 months, a2 is moderately correlated with the aging time of 6 months, a4 is moderately correlated with the aging time of 30 months, and a5 has the least impact on reducing sugar. This indicates that a1 and a4 can be used to positively regulate the content of reducing sugar, and this regulatory effect is more pronounced during the aging periods of 6-12 months and 30 months.

Overall, total sugar is weakly positively correlated with a1, a3, and a4, and the key parameters of threshing and redrying have a greater impact on total sugar during the aging period of 0-12 months. Specifically, a3 is moderately correlated with the aging time of 6-12 months, a4 is moderately correlated with the aging time of 0 months, and a2 has the least impact on total sugar. This indicates that a1, a3, and a4 can be used to positively regulate the content of total sugar, and this regulatory effect is more pronounced during the aging period of 0-12 months.

Overall, total plant alkaloids are weakly negatively correlated with a2 and weakly positively correlated with a5. The key parameters of threshing and redrying have a greater impact on total plant alkaloids during the aging periods of 0 and 24 months. Specifically, a2 is moderately correlated with the aging time of 0 months, and a5 is moderately correlated with the aging times of 0, 18, and 24 months. a4 has the least impact on total plant alkaloids. This indicates that a2 can be used to negatively regulate and a5 to positively regulate the content of total plant alkaloids, with this regulatory effect being more pronounced during the aging periods of 0, 18, and 24 months.

Overall, there is no correlation between total nitrogen and the key parameters of threshing and redrying, indicating that these parameters have difficulty influencing the content of total nitrogen. This is consistent with the study by Zhan Lei et al. [26], which found that while increasing redrying temperature leads to higher total sugar and reducing sugar, its effect on total nitrogen is relatively small.

Overall, potassium is weakly positively correlated with a2, suggesting that a2 can be used to positively regulate the content of potassium.

Overall, chlorine is weakly negatively correlated with a1 and a4, and the key parameters of threshing and redrying have a greater impact on chlorine during the aging period of 0-6 months. Specifically, a1 is moderately correlated with the aging time of 0 months, while a2 and a5 have the least impact on total chlorine. This indicates that a1 and a4 can be used to negatively regulate chlorine, and this regulatory effect is primarily manifested during the threshing and redrying stage.

Table 4. Simple Correlation Coefficients between Key Parameters of Threshing and Redrying and Reducing Sugar

Correlation Coefficient (Month)	a1	a2	a3	a4	a5
RS (0)	0.29	-0.13	-0.11	0.17	0.29
RS (6)	0.26	-0.42	-0.21	0.12	0.02
RS (12)	0.34	-0.26	0.40	0.33	0.01
RS (18)	0.31	-0.18	0.05	-0.08	0.21
RS (24)	0.07	-0.11	0.18	0.33	0.01
RS (30)	0.20	-0.07	-0.15	0.43	0.07

Table 5. Simple Correlation Coefficients between Key Parameters of Threshing and Redrying and Total Sugar

Correlation Coefficient (Month)	a1	a2	a3	a4	a5
TS (0)	0.27	0.02	-0.08	0.59	-0.36
TS (6)	0.15	-0.18	0.40	0.35	-0.10
TS (12)	0.28	-0.29	0.44	0.25	0.02
TS (18)	0.24	-0.07	0.12	0.14	0.21
TS (24)	0.09	-0.06	0.23	0.30	0.00
TS (30)	0.22	0.00	-0.10	0.37	0.08

Table 6. Simple Correlation Coefficients between Key Parameters of Threshing and Redrying and Total Plant Alkaloids

Correlation Coefficient (Month)	a1	a2	a3	a4	a5
TA (0)	-0.05	-0.42	0.34	-0.04	0.47
TA (12)	0.28	-0.32	-0.02	-0.10	0.16
TA (18)	0.04	-0.20	0.19	0.04	0.41
TA (24)	0.18	-0.14	0.17	-0.03	0.53

Table 7. Simple Correlation Coefficients between Key Parameters of Threshing and Redrying and Total Nitrogen

Correlation Coefficient (Month)	a1	a2	a3	a4	a5
TN (12)	0.16	-0.06	-0.17	-0.10	-0.09

Table 8. Simple Correlation Coefficients between Key Parameters of Threshing and Redrying and Potassium

Correlation Coefficient (Month)	a1	a2	a3	a4	a5
K (0)	0.00	0.33	-0.09	0.07	-0.13
K (12)	-0.17	0.27	0.13	0.16	-0.05

Table 9. Simple Correlation Coefficients between Key Parameters of Threshing and Redrying and Chlorine

Correlation Coefficient (Month)	a1	a2	a3	a4	a5
Cl (0)	-0.48	0.11	0.38	-0.36	-0.27
Cl (6)	-0.30	0.16	-0.10	-0.24	-0.21
Cl (12)	-0.08	-0.01	-0.12	0.01	0.01
Cl (18)	-0.27	0.06	-0.15	-0.31	-0.03

4 Conclusion

As the aging time (0-30 months) increases, the reducing sugar generally shows a fluctuating trend of first rising and then falling, with the highest content at 12 months of aging. The total sugar generally shows a pattern of first decreasing and then fluctuating slightly, reaching its maximum at 0 months and a lower level at 18 months of aging. There are no significant differences in total plant alkaloids ($P > 0.05$), indicating that aging time has little effect on total plant alkaloids. The total nitrogen generally shows a trend of first increasing, then decreasing, and then increasing and decreasing again, forming an "M" shape, with lower content between 12 and 18 months of aging. Potassium generally stabilizes initially, reaches its maximum at 12 months, and then decreases and stabilizes, with small changes overall. Chlorine generally remains stable initially, begins to decrease at 18 months of aging, reaches its minimum at 24 months, and then increases.

Reducing sugar is weakly positively correlated with a1 and a4, with this correlation being more pronounced at 6-12 months and 30 months of aging. Total sugar is weakly positively correlated with a1, a3, and a4, with this correlation being more pronounced at 0-12 months of aging. Total plant alkaloids are weakly negatively correlated with a2, with this correlation being more pronounced at 0, 18, and 24 months of aging. Total nitrogen shows no correlation with the key parameters of threshing and redrying. Potassium is weakly positively correlated with a2, and chlorine is weakly negatively correlated with a1 and a4, with these correlations mainly manifesting during the threshing and redrying stage.

(3) This study investigated the effects of key parameters of threshing and redrying on six conventional chemical components of tobacco leaves through one-way analysis of variance and correlation analysis. The study found that these key parameters have a certain regulatory effect on the content of reducing sugars, total sugars, total alkaloids, and chlorine, especially under specific aging times the regulatory effect is more pronounced. However, the impact of these parameters on the content of total nitrogen is not significant, which is consistent with existing research results. By optimizing the key parameters of threshing and redrying, the chemical components of tobacco leaves can be effectively regulated, thereby improving the quality and usability of the tobacco leaves.

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