Effects of pretreatment on supercritical CO2 extraction of aroma components from tobacco waste

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Abstract. The supercritical carbon dioxide (SCO_2) extraction of biomass for aroma components has the characteristics of high extraction efficiency, low pollution, and easy separation of the extraction products. Combined with appropriate raw material pretreatment methods, the physical and chemical properties of the raw materials can be changed to further optimize the extraction process. However, the effects of different pretreatment methods on the yield and composition of biomass SCO_2 extraction products are still unclear. Therefore, this research used tobacco waste residue (TWR) as raw material to explore the effects of different pretreatment methods (grinding, freeze-drying, torrefaction and acid-washing) on the composition of its SCO_2 extraction products. Research shows that compared with untreated tobacco waste residue, grinding effectively increases the yield of SCO_2 extraction products of TWR and the extraction product yield increased over 30%; the freeze-drying and torrefaction processes caused a large loss of the extractables, leading to a reduction in extraction efficiency, a decrease in the content of the pyridines in the extraction product, and an increase in the relative content of the hydrocarbons. Acid washing effectively changed the relative content of the hydrocarbons and the pyridines in the extract while having a low impact on the yield of the extraction product, directionally increasing the content of hydrocarbon aroma components.

Keywords: Supercritical Fluid Extraction, Pretreatment, Aroma Components.

1 Introduction

The high-value recycling of biomass resources is an important sector in achieving the recycling and utilization of waste resources.[1–4] TWR, as a by-product in cigarette production, still contain rich organic active substances and aroma-causing ingredients.[5–7] But due to the lack of effective recycling methods, it is often directly discarded as waste, which has an impact on environmental safety. Therefore, there is an

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urgent need to develop a feasible method for recycling tobacco waste to reduce its environmental pollution, while effectively recovering the active ingredients for recycling, increasing the added value of tobacco, and laying a solid foundation for the subsequent diversified development of the tobacco industry.[8–11]

Conventional methods for extracting aroma components from tobacco waste include steam distillation and solvent extraction.[12–14] The water vapor extraction method is a more traditional extraction method. It generates water vapor to pass through plants to achieve the effect of extracting aroma ingredients. Although this method is simple, easy and pollution-free, the extraction efficiency and extraction rate are very low, making it difficult to adapt. the needs of modern industrial production. In order to improve the extraction efficiency and amount of biomass aroma substances as much as possible, researchers have increasingly used organic solvents as extractants. Solvent extraction method uses organic solvents such as petroleum ether, n-hexane, etc. for extraction.[15–18] This method has simple and cheap equipment and fast extraction speed. Researchers have effectively extracted odorants such as sterols, chlorogenic acid from tobacco wastes using solvent extraction methods.[19] The efficiency is high, but the extracted plant essential oils have many impurities so they are difficult to use directly, and the large use of organic solvents poses serious challenges to the environment. The separation of organic solvents and extracts requires tedious process steps. New extraction improvement methods such as accelerated solvent extraction, Soxhlet extraction, and ultra sonic assisted extraction have also been applied to obtain organic active components from biomass.[18] However, currently these technologies have not completely changed the fundamental issues of the traditional methods mentioned above. Therefore, based on the above defects, conventional methods are currently limited in the recovery and extraction of tobacco waste.

As an emerging extraction method in recent decades, supercritical fluid extraction technology maintains the fluid in a supercritical state by controlling temperature and pressure to extract chemical components from the raw materials; when the fluid returns to normal temperature and pressure, the fluid can be separated from the extract. Supercritical fluid has received research and attention at home and abroad in recent years due to its strong extraction ability, high yield, no pollution, and easy separation of extraction products. Many researchers have investigated on the impact of supercritical fluid extraction process parameters on the extraction process. Tobacco oil and six kinds of common tobacco polyphenols were obtained by supercritical ultrasonic-assisted extraction with the yields of 67.5% and 2.51% respectively.[20] These studies have shown that the use of supercritical extraction technology can effectively extract the aroma components in tobacco waste and achieve high-value utilization of tobacco waste.[21,22] In order to improve the extraction process of tobacco waste, it may be a simple and feasible way to pretreat tobacco waste.

There are many different pretreatment ways has been used in the utilization of biomass for the different targets. Grinding is the most common used pretreatment, which can reduce the size and heterogeneity of biomass through mechanical cutting. It can form uniform particles and improve specific surface area of biomass particles. Freeze drying is a low-temperature drying method that can remove unbound water from biomass without heating, in order to avoid the problem of equipment pipeline freezing during supercritical extraction due to high water content. Torrefaction is a mild heat treatment method that heats biomass in an anaerobic low temperature range (200 ℃ - 300 ℃) to release small molecules of oxygen-containing volatile matter, improve the grindability of biomass particles, change surface structure, increase hydrogen carbon ratio, and increase calorific value. Acid washing can change the chemical structure of biomass by dissolving alkaline substances in it. However, the effects and mechanisms of different pretreatment methods on the components and efficiency of tobacco waste extractions are still unclear and need to be studied.

Therefore, this article conducts different pretreatments on tobacco waste, analyzes the effects of different pretreatment methods on the extraction of aroma components from tobacco waste, and elucidates the impact mechanisms of different pretreatment methods on the supercritical extraction process of tobacco waste, in order to provide a basis for targeted extraction of tobacco waste. Provide guidance on the aroma-causing ingredients at the end.

2 Material and methods

2.1 Sample preparation

The tobacco waste raw material used in this study comes from Hubei China Tobacco Industrial Company. The raw materials were subjected to four pre-treatments: grinding, freeze-drying, torrefaction and acid-washing, and then supercritical carbon dioxide extraction experiments were conducted on tobacco with different pre-treatment methods and conditions to explore the effects of different processing methods on the aroma of tobacco essence. Influence of ingredient type and content. The four preprocessing methods are as follows:

Grinding: Use a cutting machine to grind the tobacco waste raw materials and sieve them into tobacco waste with three different particle sizes: 20-40 mesh; 40-60 mesh and above 60 mesh for subsequent extraction experiments.

Freeze-drying: The tobacco waste raw materials were freeze-dried in a freeze-drying machine, and three samples with freeze-drying times of 24 h, 36 h, and 48 h were prepared for subsequent extraction experiments.

Torrefaction: Place the tobacco waste in a high-temperature tube furnace for torrefaction. Use N_2 as the purge gas and purge for 20 minutes before the start of the experiment to eliminate the air in the reactor. The Torrefaction temperatures were set to 200 °C; 250 °C and 300 °C respectively. In each experiment, the sample was heated at a heating rate of 10°C/min to the target temperature and was kept for 1 h, and then naturally cooled to room temperature to obtain tobacco waste treated at different torrefaction temperatures for subsequent extraction experiments.

Acid-washing: Use hydrochloric acid (HCl), acetic acid (CH_3COOH) and phosphoric acid (H_3PO4) with a concentration of 10% to pickle the tobacco waste as it is. The acid-washing time is 4 hours, and then rinsed with deionized water 5 to 8 times respectively, and left to air dry at room temperature for subsequent extraction experiments.

2.2 Experimental procedures

Extraction experiments of aroma components were carried out on tobacco waste without pretreatment and different pretreatments. Supercritical carbon dioxide fluid extraction is used to enrich the aroma components in tobacco waste. Each extraction experiment used 100g of tobacco waste, the operating pressure of the extraction kettle is 30 MPa, the extraction temperature is 80 °C, and the extraction time is 1 h. Under these extraction conditions, a higher extraction yield of tobacco essence can be obtained. During each extraction process, 100 ml of absolute ethanol was injected as an entrainer, and the injection frequency was 15 Hz.

After each extraction experiment, the extraction product is collected from the separation kettle, which is tobacco essence. The yield of tobacco extract is calculated by weighing the mass difference before and after the extraction basket.

2.3 Analytical method

Gas chromatography-mass spectrometry. Gas chromatography-mass spectrometry (GC-MS) (7890A/5975C, Agilent) was used. Equipped with a capillary chromatography column (DB-5ms) (length 30 m; inner diameter 0.25 mm; film thickness 0.25 mm), tobacco extracts were analyzed to quantify the aroma components. Inject 1 μL sample into the injection port in a split-free manner, and the injection port is set to 250 °C.[23,24] Use purity > high-purity helium (99.999%, Wuhan Zhongxin Ruiyuan Gas Co., Ltd.) to maintain a constant flow rate of 1 μL/min. Pillar. Initially, set the temperature of the column to 40 °C for 3 min, then increase to 250 °C at a ramp rate of 5 °Cmin^{-1} and hold for 10 min; use a 1:1 split ratio to connect the mass spectrometer source and quadrupole. The tube temperatures were set to 230 °C and 150 °C, respectively, and the mass spectral scan range was 15-500. After a 6.3 min solvent delay, mass spectra were acquired and the species of the sample were identified using the National Institute of Standards and Technology's mass spectral library.

Ultraviolet Fluorescence. Detection of aromatic compounds in tobacco extracts using ultraviolet (UV) fluorescence spectroscopy. The energy difference constant of the spectrometer is -2800 cm^{-1}. The slit width was set to 5 nm and the scanning speed was set to 120 nmmin^{-1}. The details of the testing process have been described in a previous study. [25,26]The wavelengths of the UV fluorescence spectrum can be used to approximate the degree of polymerization of aromatic rings: wavelengths less than 290 nm represent single-ring aromatics, while the range between 290 and 370 nm represents 2-3 ring aromatics, and wavelengths greater than 370 nm represent Corresponds to aromatic substances with more than three rings.

3 Results and Discussion

3.1 Effects of different pretreatments on the yield of aroma ingredients

Table 1. Extraction rates of aroma components from different pretreated tobacco wastes.

Pretreatments	Conditions	Yields
No pretreatment	-	11.70
	20-40 mesh	14.08
Grinding	40-60 mesh	14.83
	≥60 mesh	15.60
	24 h	3.28
Freeze Drying	36 h	2.34
	48 h	3.67
	200 °C	1.65
Torrefaction	250 °C	1.50
	300 °C	1.50
	10%HCl	9.07
Acid Washing	10%H_3PO_4	8.62
	10%CH_3COOH	10.24

The yields of tobacco essence from different pretreated tobacco wastes are shown in Table 1. Generally speaking, the extraction yield of raw tobacco waste increases after grinding, and the tobacco essence obtained after extraction from freeze-dried, baked, and pickled tobacco waste is less than that of untreated tobacco waste. As the particle size of ground tobacco waste decreases, the extraction yield of tobacco essence shows an upward trend. The extract yield of tobacco waste after freeze-drying and Torrefaction has dropped significantly. The former is mainly due to the lack of water, which reduces the extraction efficiency of supercritical carbon dioxide for certain substances, so the extraction effect is not increased; the latter is mainly due to Since many small molecular volatiles are lost during the torrefaction process, the content of aroma-causing ingredients that can be extracted during the subsequent extraction process is greatly reduced. The extract yield of tobacco waste after acid-washing treatment dropped slightly, mainly due to the loss of nicotine and other alkaline substances in the tobacco during the acid-washing process, resulting in a decrease in the extract yield.

3.2 Effects of different pretreatments on the composition of aroma ingredients

The distribution of aroma components in tobacco extracts with different grinding particle sizes is shown in Fig. 1 (a). Pyridines and hydrocarbons are the main aroma-causing ingredients in the essence; small-molecule acids and alcohol ethers account for a considerable proportion, while esters and phenols are present in smaller amounts. Grinding has different effects on different types of aroma-causing ingredients in tobacco essence: the relative proportions of acids, hydrocarbons and phenolic aroma-causing ingredients in tobacco essence increase, while the relative content of alcohol ethers and pyridines decreases. Trend; Some aldehydes and ketones, such as furanones and pyrone compounds, are related to the burnt sweet aroma of flue-cured tobacco and are almost undetectable in the essence. The relative content of pyridine substances in the extract is basically unchanged when the grinding particle size is 40 mesh. When the grinding particle size is greater than 40 mesh, the relative content of pyridine substances decreases significantly. The relative contents of hydrocarbons, acids and phenols gradually increase as the grinding particle size increases. However, since phenolic substances account for a small proportion, the change in relative content is small. It is worth noting that the relative content of alcohol ethers in the essence of ground tobacco is less than that of untreated tobacco, but as the grinding particle size decreases, the content of alcohol ethers shows an upward trend, which may This is because although the heat during the grinding process decomposes some small-molecule alcohol ethers, as the particle size decreases, the contact between carbon dioxide and the raw materials during the extraction process is better, and the penetration efficiency is higher, resulting in the essence. The content of alcohol ether substances has rebounded.

The distribution of aroma components of tobacco extract after freeze-drying at different times is shown in Fig. 1 (b). After freeze-drying, the relative contents of acids, phenols and hydrocarbons in the tobacco extract increased; the relative contents of alcohol ethers and pyridines showed a downward trend. After freeze-drying, the relative content of pyridine aroma-causing ingredients such as nicotine decreased significantly. As the freeze-drying time increased, the relative content of pyridine substances increased slightly. The relative content of hydrocarbons nearly doubled. The relative content of acidic substances increases with the increase of freeze-drying time, indicating that the removal of moisture is beneficial to the extraction of acidic substances from tobacco waste. As an important category of tobacco aroma components, phenolic substances have a low relative content in the tobacco essence, but the relative content in the tobacco essence after freeze-drying has nearly doubled. Similar to the grinding treatment, the relative content of alcohol ether substances after freeze-drying is lower than the corresponding content of substances in untreated tobacco extract, but as the freeze-drying time increases, the relative content of alcohol ether substances increases, which shows that A certain amount of moisture is conducive to the extraction process of alcohol ethers. When the moisture content is low, the pores in the tobacco increase, and the extraction efficiency increases with the decrease in moisture content, thus causing a small increase in the relative content of alcohol ethers.

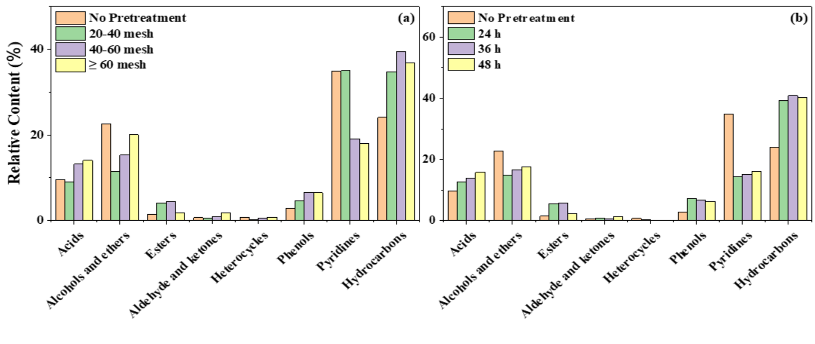

Fig. 1. Distribution of aroma components in tobacco extracts.(a) with different particle sizes, (b) with different freeze-drying time.

The distribution of aroma components of tobacco extract after torrefaction at different temperatures is shown in Fig. 2 (a). After curing, the relative content of hydrocarbon substances in the tobacco extract increased significantly, while the relative content of other substances decreased, with the largest decrease being pyridine substances. The relative content of pyridine substances in the tobacco extract after curing is reduced by more than 80% compared to that of untreated tobacco essence; and as the Torrefaction temperature increases, the content becomes lower, indicating that the pyridine substances in the tobacco such as nicotine during Torrefaction After decomposition occurs, it is released from the tobacco in the form of volatile matter. Since the molecular weight of alcohol ethers is usually small, they volatilize in the form of gas or directly heated during the Torrefaction process, resulting in a decrease in their relative content in subsequent tobacco extracts. The content of phenolic substances increased significantly, indicating that some phenolic substances were generated through polymerization during the Torrefaction process, which increased the relative content of phenolic substances in tobacco extract. Changes in the relative content of hydrocarbon substances are mainly caused by changes in the relative content of other substances and the removal of carboxyl groups by acidic substances.

The distribution of aroma components of different types of tobacco extracts after acid-washing treatment is shown in Fig. 2 (b). The content of pyridine substances in tobacco after different types of acid-washing treatments is almost zero. This is mainly due to the neutralization reaction between pyridine nicotine substances and acids, resulting in almost no pyridine substances available for extraction during extraction. Since the tobacco raw materials have been pickled, the content of acid substances in the extract has increased significantly. Among them, tobacco treated with acetic acid has the highest acid content in the extract. This may cause acetic acid itself to act as an organic acid during the extraction process. Compared with other acid-washing liquids, it is easier to be extracted together, resulting in a significant increase in the content of acids in the extract. After acid-washing treatment, the relative content of alcohol ethers in the extract decreased, while the relative content of esters, phenols and hydrocarbons increased significantly. Different types of acids have different effects on different types of substances. Phosphoric acid has the most obvious increase in hydrocarbon substances; hydrochloric acid has the most obvious increase in phenolic substances. After acid-washing, the relative content of alcohol ethers decreases slightly. The relative content of alcohol ethers is relatively less affected by acetic acid and can be retained as much as possible in the extract.

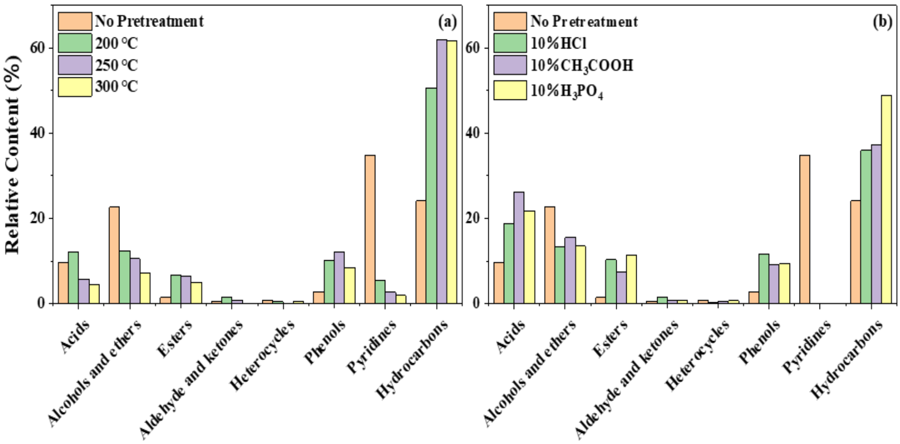

Fig. 2. Distribution of aroma components in tobacco extracts.(a) with different torrefaction temperature, (b) with different kinds of acid.

As shown in Fig. 3 (a), the carbon number distribution of different components shows that the carbon number distribution of the main aroma-causing components in the essence is above C11. After grinding, the average carbon number of aroma-causing components in tobacco extracts above 40 mesh increases, indicating that the contact area between carbon dioxide and tobacco particles is increased after grinding, which is conducive to the extraction of some macromolecule aroma-causing components, so in the subsequent extraction The relative content in the substance increases. In the freeze-dried tobacco extract, the relative content of C7-C10 substances significantly decreased, and the relative content of C11 and above substances increased significantly. Fig. 3 (b) shows that the presence of a little moisture is beneficial to the extraction process of low carbon number substances, such as low-order alcohol ethers and pyridines.

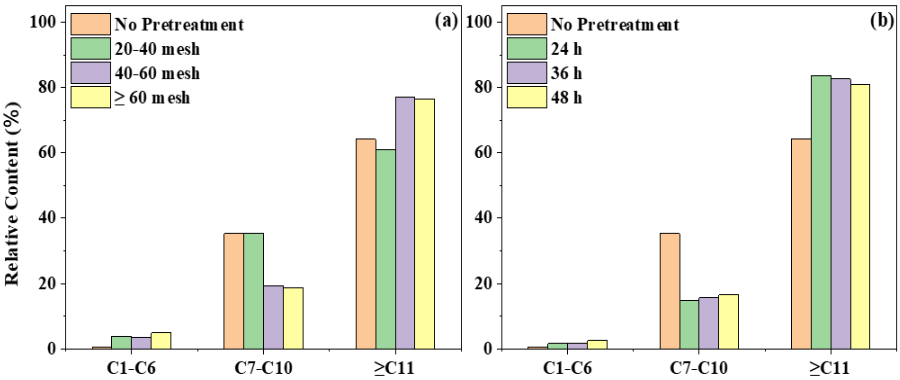

Fig. 3. Carbon number distribution in tobacco extracts. (a) with different particle sizes, (b) with different freeze-drying time.

As shown in Fig. 4 (a), the distribution of the number of carbon atoms shows that the decomposition and volatilization of small molecular substances occurs during torrefaction, resulting in a significant decrease in the relative content of low carbon number substances in subsequent tobacco extracts and an increase in the average carbon number. For the acid-washing tobacco waste, as it shown in Fig. 4 (b), due to the loss of pyridine substances, the average carbon number of substances in tobacco extract has increased significantly, and most of them are long-chain substances above C11.

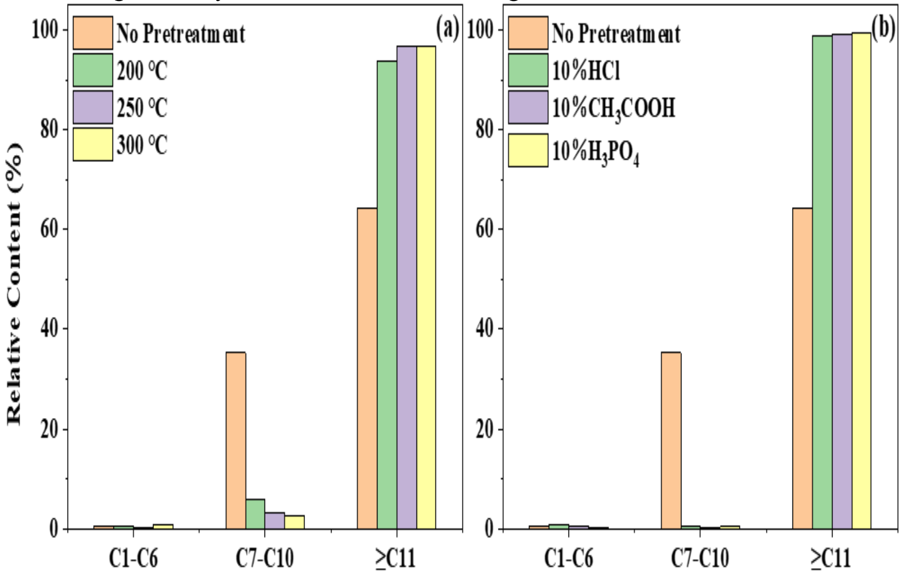

Fig. 4. Carbon number distribution in tobacco extracts. with different torrefaction temperature, (b) with different kinds of acid.

3.3 Effects of different pretreatments on the content of main aroma components

The distribution of the main aroma components in SCO_2 extracts of different pretreated tobacco wastes is shown in Fig.5. The contents of nicotine, stearic acid, linolenic acid, neophytediene, cembratrienol and hexadecanoic acid these six main aroma components in different pretreated tobacco extracts were analyzed.

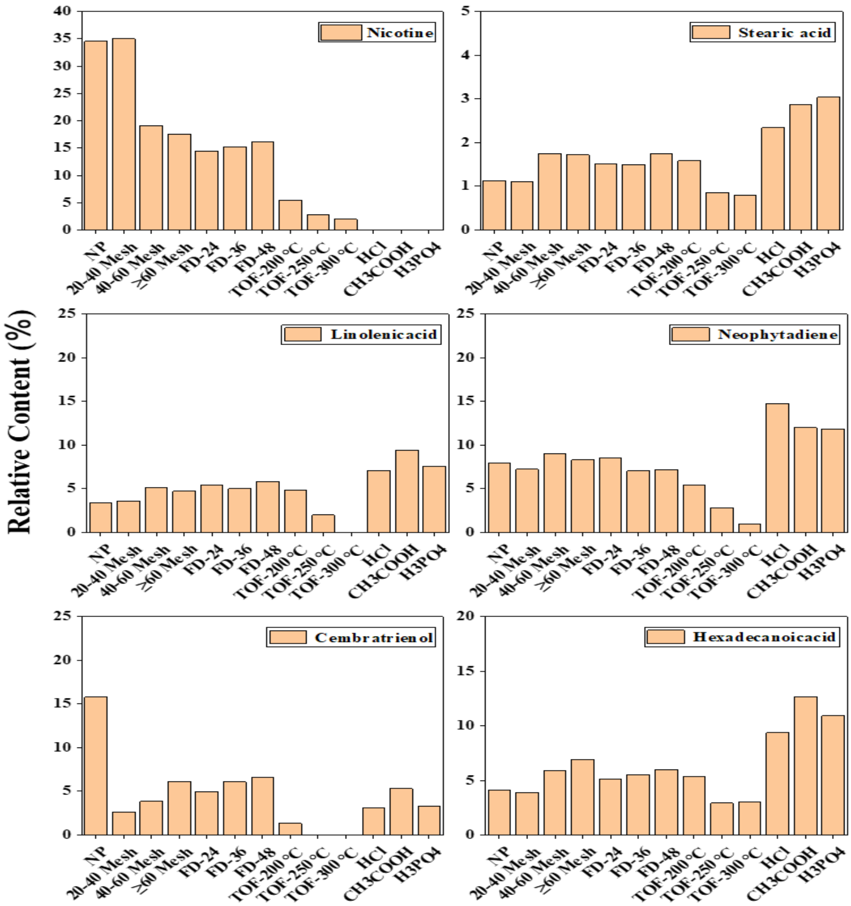

Fig. 5. Distribution of the main aroma components content in different pretreatments. "NP"= "No Pretreatment", "FD"= "Freezing-Cold", "TOF"= "Torrefaction".

In general, the relative content of nicotine, compared with untreated tobacco waste, is lower in the extracts of pretreated tobacco waste. The extracts of tobacco waste which were grinding and freeze-drying still contain high content of nicotine, which were about 15 percent in the extracts. But the nicotine in torrefied and acid-washing tobacco waste extracts reduce is low. This is mainly due to the poor thermal stability of nicotine, which undergoes decomposition reactions during the torrefaction process. The tobacco waste after torrefaction almost no longer contains nicotine, so this component is almost nonexistent in subsequent extracts. [20] Nicotine can still be detected in the extract of tobacco treated at a lower torrefaction temperature (200 °C). As the torrefaction temperature increases, nicotine is basically completely decomposed and cannot be detected in the extract. Pretreatment can effectively increase the stearic acid content in tobacco waste extracts, but its content was still at a low level. The effects of pretreatment on linolenic acid and cembratrienol had similar trends. The four types pretreatments will significantly reduce the content of cembratrienol, among which torrefaction has the greatest impact. At higher torrefaction temperatures, the cembratrienol was destroyed and almost do not exist in the extracts. [19] For linolenic acid, acid-washing and torrefaction pretreatment can effectively affect their content in tobacco waste. Torrefaction also leads to the decarboxylation reaction of oxygen-containing acids to generate carbon dioxide, resulting in a decrease in linolenic acid content. The acid-washing process provides protection for your daughter in an acidic environment, while reducing the nicotine content in tobacco waste, thereby increasing the relative content of linolenic acid. The acid-washing treatment effectively increased the relative content of neophytodiene and hexadecanoic in their tobacco waste extracts. This was mainly due to the destruction of nicotine during the acid-washing, resulting in an increase in the relative content of neophytodiene and hexadecanoic. Therefore, acid washing treatment can be used as a targeted control method for the content of neophytene and hexadecanoic in the extract.

3.4 Effects of different pretreatments on aromatic aroma components

The effects of different pretreatment methods on the content of aromatic substances in tobacco essence are shown in the Fig.6. Physical pretreatment has little impact on the types and contents of aromatic substances in tobacco essence, while chemical pretreatment has a significant impact on the types and contents of aromatic substances in tobacco essence. After grinding, the aromatic aroma components in the tobacco essence are mainly 2-3 ring aromatic substances, and the proportion of single-ring aromatic substances is relatively balanced. In the tobacco essence extracted from tobacco waste with different particle sizes, the content of aromatic substances changes little. As the tobacco particle size decreases, the content of aromatic substances increases slightly. This is mainly due to the decrease in particle size. The specific surface area of supercritical carbon dioxide increases, the contact between supercritical carbon dioxide and raw materials is more complete, and the extraction is more complete, so the content of aromatic substances in the essence increases slightly.

The freeze-drying process shows a promoting effect on the extraction of different types of aromatic substances. For monocyclic aromatic substances, freeze-drying promotes the extraction process, and the corresponding content in the essence increases significantly, but the change in content is not significant at different freeze-drying times; for polycyclic aromatic substances, freeze-drying can also Promote the extraction process and improve the extraction rate. As the freeze-drying time increases, the extraction rate of polycyclic aromatic substances increases, and the content of polycyclic aromatic substances in the extract increases slightly.

Torrefaction pretreatment has a great impact on the composition of tobacco extract. As shown in the Fig.7, the aromatic components of tobacco processed at a lower torrefaction temperature are similar to those of untreated tobacco. They are still dominated by 2-3 ring aromatic substances, and the content of single-ring aromatic substances is relatively low. [21] The low-temperature torrefaction treatment slightly increased the content of aromatic substances in the extract. This may be because the release of small molecule volatiles during the Torrefaction process changes the pore structure of the tobacco raw materials. On the one hand, it reduces the relative content of tobacco nonaromatic substances. On the other hand, it increases the specific surface area of the extracted raw materials and improves the extraction efficiency. Therefore, Essence contains a higher proportion of aromatic substances. When the torrefaction temperature increases, the release of volatiles is enhanced, polycyclic aromatic substances in tobacco are cracked and volatilized, and the content of single-cyclic aromatic substances is significantly increased. Therefore, the relative content of single-cyclic aromatic substances in the extract is increased. Combined with the analysis results of GC-MS, it is speculated that this process is mainly caused by the decomposition reaction of pyridine substances such as nicotine during torrefaction and conversion into monocyclic aromatic substances. Therefore, the content of polycyclic aromatic substances in tobacco can be semi-quantitatively transformed by adjusting the torrefaction processing conditions to meet the production needs of additives for cigarettes with different flavors. [17]

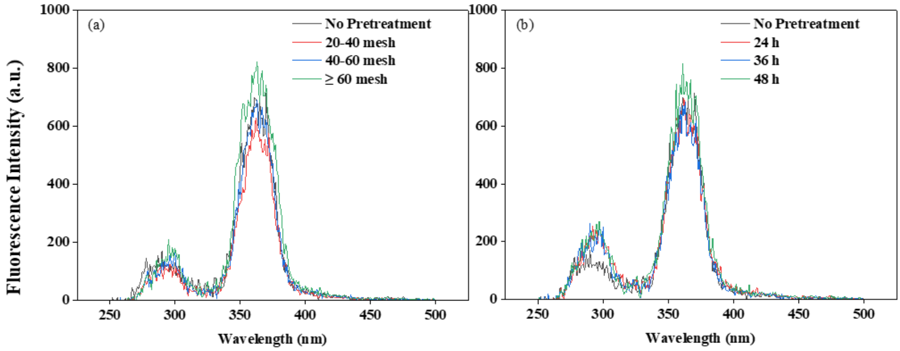

Fig. 6. Aromatics distribution in tobacco extracts. (a) with different particle sizes, (b) with different freeze-drying time.

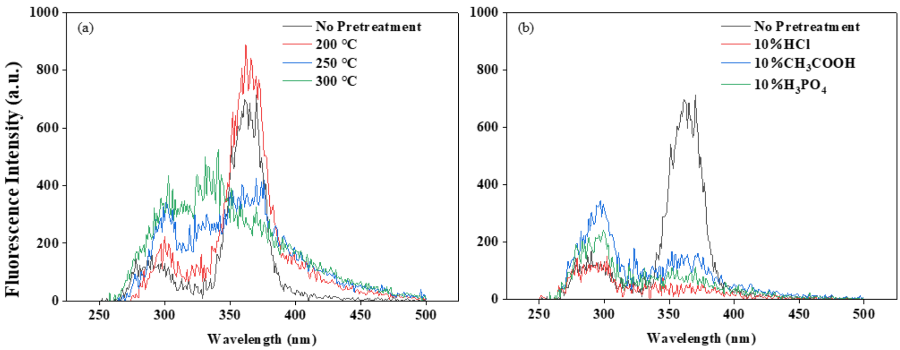

Fig. 7. Aromatics distribution in tobacco extracts. (a) with different torrefaction temperature, (b) with different kinds of acid.

Similar to the torrefaction treatment, the content and composition of aromatic substances in tobacco after acid treatment have changed significantly. Compared with untreated tobacco, the content of polycyclic aromatic substances in the extract of pickled tobacco is significantly reduced, while the content of monocyclic aromatic substances is significantly increased. This is also mainly due to the neutralization reaction of alkaloids such as nicotine during the acid-washing process, resulting in the conversion of polycyclic aromatic substances into monocyclic substances. Different types of acids have different effects on the content of aromatic substances in tobacco essence. Compared with the other two acids, acetic acid can retain the aromatic components in tobacco as much as possible and increase the content of aromatic components in the essence. Since hydrochloric acid is the most acidic, it has the greatest impact on the content of aromatic substances. The content of aromatic substances in its extract is lower than the content of corresponding substances in the other two pickled tobacco extracts.

4 Conclusion

This paper studies the effects of different pretreatment methods on the extraction of aroma components from tobacco waste by supercritical carbon dioxide. The results show that grinding, as a simple and easy pretreatment method, can effectively improve the extraction rate of tobacco waste supercritical extraction essence, and has little impact on the components of the essence. It is an effective pretreatment. means. Freeze drying and Torrefaction have a serious impact on the yield of the extract. Although Torrefaction can effectively change the component distribution of the aroma components in the extract, it is still not a suitable pretreatment method. Acid washing significantly changes the distribution of components in the tobacco extract with little impact on the yield of tobacco extract. It is a pretreatment method worth considering as a subsequent directional extraction of certain aroma-causing components.

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Reference

1. L. Ostervold, S.I. Perez Bakovic, J. Hestekin, L.F. Greenlee, Electrochemical biomass upgrading: degradation of glucose to lactic acid on a copper(II) electrode, RSC Adv 11, 31208–31218 (2021).
2. B. Wang, J. Lan, C. Bo, B. Gong, J. Ou, Adsorption of heavy metal onto biomass-derived activated carbon: review, RSC Adv 13, 4275–4302 (2023).
3. Y.P. Rago, F.-X. Collard, J.F. Görgens, D. Surroop, R. Mohee, Torrefaction of biomass and plastic from municipal solid waste streams and their blends: Evaluation of interactive effects, Fuel 277, 118089 (2020).
4. S. Park, S.J. Kim, K.C. Oh, L. Cho, Y.K. Jeon, D.H. Kim, Acid and alkali pretreatment of agro by-products: Evaluating torrefaction efficiency and dechlorination, Energy 283, 128548 (2023).

5. R.K. Sharma, H. Kaur, M. Singh, M. Kumar, R. Sharma, G.C. Shah, P. Sharma, Chemical Composition and Antimicrobial Properties of Essential Oil Anaphalis triplinervis from Western Himalaya, Chem Nat Compd 55, 751–753(2019).
6. L. Wu, Z. He, Y. Wu, J. Liu, C. Li, J. Cao, B. Wang, S. Min, Evaluation of Aroma Components in Chinese Southwest Tobacco by Headspace Gas Chromatography-Mass Spectrometry, Asian J. Chem 25, 8853–8858 (2013).
7. S. Shukla, I. Marks, D. Church, S.-K. Chan, J.K. Pokorski, N.F. Steinmetz, Tobacco mosaic virus for the targeted delivery of drugs to cells expressing prostate-specific membrane antigen, RSC Adv 11, 20101–20108 (2021).
8. X. Li, F. Shu, C. He, S. Liu, N. Leksawasdi, Q. Wang, W. Qi, Md.A. Alam, Z. Yuan, Y. Gao, Preparation and investigation of highly selective solid acid catalysts with sodium lignosulfonate for hydrolysis of hemicellulose in corncob, RSC Adv 8, 10922–10929 (2018).
9. Z. Sun, D. Buwalda, K. Barta, Two-step catalytic conversion of lignocellulose to alkanes, RSC Adv 9, 23727–23734 (2019).
10. Y. Hu, J. Zhao, H. Li, X. Wang, P. Hou, C. Wang, A. Li, L. Chen, *In vivo* detection of salicylic acid in sunflower seedlings under salt stress, RSC Adv 8, 23404–23410 (2018).
11. C. Luo, L. Huang, Y. Chen, Z. Wang, H. Ren, H. Liu, Z. Liu, Effects of potassium additives on the combustion behavior of chrysanthemum biochar blended with graphite carbon as a heating source for heat-not-burn tobacco, RSC Adv 12, 3431–3436 (2022).
12. I.T. Stanisavljević, M.L. Lazić, V.B. Veljković, Ultrasonic extraction of oil from tobacco (Nicotiana tabacum L.) seeds, Ultrasonics Sonochemistry 14, 646–652 (2007).
13. S. Jokić, M. Molnar, M. Jakovljević, A.-M. Cikoš, S. Šafranko, M. Matić, M. Banožić, Ekstrakcija bioaktivnih spojeva iz duhanskog otpada primjenom eutektičkih otapala, Kem. ind. (Online) 69, 1–10 (2020).
14. Z. Guo, L. Wang, C. Song, L. Zhang, Extraction of nicotine from local tobacco using double-supported liquid membranes technique, Journal of Membrane Science 356, 105–109 (2010).
15. A. Demirbas, Conversion of Agricultural Residues to Fuel Products via Supercritical Fluid Extraction, Energy Sources 26, 1095–1103 (2004).
16. M. Ashraf-Khorassani, L.T. Taylor, N. Nazem, W.M. Coleman, Isolation of Tetra-acyl Sucrose Esters from Turkish Tobacco Using Supercritical Fluid CO_2 and Comparison with Conventional Solvent Extraction, J. Agric. Food Chem 53, 1866–1872 (2005).
17. X. Gu, J. Cai, X. Zhu, Q. Su, Dynamic ultrasound-assisted extraction of polyphenols in tobacco, J. Sep. Science 28, 2477–2481 (2005).
18. J. Shen, X. Shao, A comparison of accelerated solvent extraction, Soxhlet extraction, and ultrasonic-assisted extraction for analysis of terpenoids and sterols in tobacco, Anal Bioanal Chem 383, 1003–1008 (2005).
19. YJ Xu and X Lu, Analysis of main sterols in tobacco by gas chromatography-mass spectrometry with solvent extraction. CHINESE JOURNAL OF CHROMATOGRAPHY 24 (3), 1000-8713 (2006).
20. H Zhang and J Peng, Journal of Yunnan University. Supercritical - ultrasonic assisted extraction of oil and polyphenols from tobacco leaves. Natural Science 362,0258-7971 (2022).
21. Y. Wang, W. Gu, Study on supercritical fluid extraction of solanesol from industrial tobacco waste, The Journal of Supercritical Fluids 138,228–237 (2018).
22. G.J. Tita, A. Navarrete, Á. Martín, M.J. Cocero, Model assisted supercritical fluid extraction and fractionation of added-value products from tobacco scrap, The Journal of Supercritical Fluids 167, 105046 (2021).
23. Z. Xiong, J. Guo, W. Chaiwat, W. Deng, X. Hu, H. Han, Y. Chen, K. Xu, S. Su, S. Hu, Y. Wang, J. Xiang, Assessing the chemical composition of heavy components in bio-oils from

the pyrolysis of cellulose, hemicellulose and lignin at slow and fast heating rates, Fuel Processing Technology 199, 6299 (2020).
24. Q. He, W. Deng, K. Xu, L. Jiang, J. Xu, S. Su, X. Hu, Y. Wang, S. Hu, J. Xiang, Enhancing pyrolysis of automobile shredder residue through torrefaction: Impact on heavy components formation in oil, Fuel Processing Technology 252, 107964 (2023).
25. W. Deng, K. Xu, Z. Xiong, W. Chaiwat, X. Wang, S. Su, S. Hu, J. Qiu, Y. Wang, J. Xiang, Evolution of Aromatic Structures during the Low-Temperature Electrochemical Upgrading of Bio-oil, Energy Fuels 33, 11292–11301 (2019).
26. Z. Xiong, L. Jiang, J. Xu, Q. Li, W. Deng, S. Su, S. Hu, Y. Wang, J. Xiang, Effects of the Gas-/Liquid-Phase Interactions on the Evolution of Bio-oil during Its Thermal Treatment, Energy Fuels 34,8482–8492 (2020).