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Effects of Microwave Treatment on Chemical Compositions of Tobacco Stems

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To illustrate the effects of microwave treatment on the quality character of tobacco stem, the changes of cell wall substances, conventional chemical compositions and aromatic components in tobacco stems were studied for K326 and Hongda. The contents of cell wall substances and most of conventional chemical compositions showed a decrease in different degree after microwave expansion, while the petroleum ether extract were greatly increased. Seventy six aromatic components consisting of 19 aldehydes, 23 ketones, 7 alcohols, 5 acids, 4 phenols, 6 esters, 10 heterocycles and 2 olefins were indentified by GC-MS analysis in tobacoo stems, compared to the non-expanded tobacco stem, the total content of aromatic components was increased from 243.35 μg/g to 255.67 μg/g.

Keywords: Tobacco stem, Microwave expansion, Chemical compositions.

INTRODUCTION

Tobacco, one of the world's leading cash crops, is grown extensively in the developing countries such as India, China, Brazil, *etc*., Tobacco stems, representing approximately 25 % of tobacco leaf output, are under utilized by-products in the tobacco industry after the tobacco leaves are harvested[1]. Unfortunately, present disposal of these stems has become a matter of public concern because of their potential environmental and human health impacts[2]. The exploitation of multi-purpose utilization technologies is particularly significant and indispensable[3-5].

Tobacco stem, not only has similar chemical compositions with tobacco leaves, but can also improve structure of cigarettes, reduce tar and harm, has always been important ingredient in tobacco group during cigarette design[6]. However, compared with tobacco leaf, the lower content of aroma components results in the deficiency and impurity of the fragrance, in addition, the intensive smell of green grass and wood has a negative impact on the quality of cigarette when smoking, thus wide applications of stem are limited[7].

The evolution of cigarette design has led to the development of novel tobacco processing techniques, including tobacco stem expansion processes[8]. The widely used method to prepare expanded shredded tobacco stems (ESS) is by means of high temperature and high pressure[9]. Recently, microwave technology was gradually applied to expand tobacco stems because of the advantages of fast heating, energy conservation and environmental protection[10]. Peng *et al.*[11] prepared the biomass material from stem granules *via* microwave radiation method and optimized the microwave processing parameters including the steam pressure, microwave power and drum rotation frequency by response surface methodology. In our previous studies, to improve the filling value and blending uniformity, microwave expansion and stem recutting process were developed. The results revealed that the cut stem had similar color with cut tobacco, the reduction of smell of green grass and offensive irritancy enhanced the quality of cigarette products.

K326 and Hongda were main cultivars in Yunnan tobacco area (China) and widely used in Yunnan cigarette brands. In this paper, based on the tobacco stems of K326 and Hongda, the changes of cell wall substances, conventional chemical compositions and aromatic components were further analyzed before and after microwave expansion to illustrate the effects of microwave expansion on the quality character, which will be helpful to offer a reference basis for the processing treatment and rational utilization.

EXPERIMENTAL

The flue-cured tobacco leaves with the degree of C3F were from K326 and Hongda in Zhaotong tobacco area (Yunnan province, China). The tobacco stem samples were obtained by removing the leaves and cutting the stems to the size of 3-4 cm.

Preparation of the expanded tobacco stems by microwave treatment: The samples were balanced to the moisture content of approximately 13 %, then were put in the equipment for microwave expansion. After expanding 70 s, the expansion

ratio of the samples reached 200 ± 20 %, the moisture content was no less than 4 % and the samples had no obvious carbonization phenomenon.

Measurement of cell wall substances: The contents of pectin, holocellulose, lignin and total content of cell wall substances were measured according to the methods by Chen *et al.*[12].

Measurement of conventional chemical compositions: Following the established methods, the conventional chemical compositions were detected by continuous flow analyzer (Skalar, Holland).

Measurement of aromatic chemical components: The tobacco stems and expanded tobacco stems were grinded to the particles with sieve of 60 mesh and balanced for 24 h in a constant ambience with the temperature and humidity of 22 °C and 60 % RH, respectively. The volatile components were extracted with dichloromethane for 2 h by simultaneous distillation and extraction. The extract was dried by anhydrous sodium sulfate and was concentrated to 1 mL, then 50 µL 0.1 mM benzyl acetate ethanol solutions were added. The sample was injected into the GC-MS system.

A GC-MS system consisting of an Agilent GC6890N/MS5973N with a fused capillary column coated with HP5-MS (30 m × 0.25 mm, 0.25 µm) was used to analyze the volatile extracts. The injector temperature was 260 °C. The flow rate of the carrier gas (helium) was 1 mL/min. The temperature was held 50 °C for 1 min, increased to 160 °C with the heating rate of 8 °C/min and held at 160 °C for 2 min, then increased to 260 °C with the heating rate of 2 °C/min and held for 15 min. The interface temperature was 280 °C with ion source temperature of 230 °C. Split ratio was 25:1.

RESULTS AND DISCUSSION

Analysis of cell wall substances: Table-1 showed the contents of the cell wall substances in expanded tobacco stems and non-expanded tobacco stems. Compared with K326, the contents of pectin, holocellulose, lignin and the total content of cell wall substances in Hongda were relatively high, after microwave expansion, all of the cell wall substances shown a decrease in different degree, but did not reach significant level. The decrease of lignin and holocellulose helped to reduce the wood gas and improve the smoothness of tobacco smoke.

Analysis of conventional chemical compositions: The changes of conventional chemical compositions for the tobacco stems before and after microwave expansion were shown in Table-2. In all conventional chemical compositions, only the content of nicotine in Hongda was significantly greater than that in K326. Compared with the non-expanded tobacco stems, protein, nicotine, total sugar, total volatile acid, total polyphenol, starch and potassium oxide decreased, the extent of reduction for Hongda was higher than K326. However, the contents of petroleum ether extract were greatly increased by 72.55 and 37.89 % for K326 and Hongda, respectively, which indicated that the macromolecules in tobacco stems were easy to decompose *via* microwave irradiation. The decomposition of the macromolecules was beneficial to increasement of aromatic chemical components, enrichment of tobacco flavor and enhancement of sensory quality for the expanded cut stems.

Analysis of aromatic chemical components: Table-3 showed the varieties and amounts of aromatic chemical components in the expanded tobacco stems and non-expanded tobacco stems. Seventy six volatile components consisting of 19 aldehydes, 23 ketones, 7 alcohols, 5 acids, 4 phenols, 6 esters, 10 heterocycles and 2 olefins were indentified by GC-MS analysis in tobacoo stems. The total content of volatile components was increased from 243.35 µg/g of non-expanded tobacco stems to 255.67 µg/g of the expanded tobacco stems. After microwave expansion, there was no significant differences ($P > 0.05$) for the contents of ketones and esters, while that of

TABLE-1
COMPARISION OF CELL WALL SUBSTANCES IN TOBACCO STEMS BEFORE AND AFTER MICROWAVE EXPANSION

Cell wall substances	K326			Hongda		
	Before expansion (%)	After expansion (%)	Change rate (%)	Before expansion (%)	After expansion (%)	Change rate (%)
Total content of cell wall substances	34.21 aA	33.70 aA	-1.49	36.92 aA	35.13 aA	-4.85
Pectin	6.43 aA	5.96 aA	-7.31	7.36 aA	6.77 aA	-8.02
Holocellulose	20.64 aA	20.01 aA	-3.05	22.39 aA	20.40 aA	-8.89
Lignin	6.62 aA	6.25 aA	-5.59	6.85 aA	6.18 aA	-9.78

TABLE-2
COMPARISION OF CONVENTIONAL CHEMICAL COMPOSITIONS IN TOBACCO STEMS BEFORE AND AFTER MICROWAVE EXPANSION

Chemical compositions	K326			Hongda		
	Before expansion (%)	After expansion (%)	Change rate (%)	Before expansion (%)	After expansion (%)	Change rate (%)
Protein	5.26 aA	5.01 aA	-4.75	4.94 aA	3.83 aA	-22.47
Total nitrogen	1.82 aA	1.78 aA	-2.20	1.57 aA	1.57 aA	0
Nicotine	0.36 bA	0.33 aA	-8.33	0.81 aA	0.63 aA	-22.22
Total sugar	23.73 aA	23.24 aA	-2.06	21.60 aA	20.40 aA	-5.56
Reducing sugar	18.01 aA	17.44 aA	-3.16	16.28 aA	16.13 aA	-0.92
Total volatile base	0.03 aA	0.02 aA	-33.33	0.02 aA	0.02 aA	0
Total volatile acid	0.13 aA	0.10 aA	-23.08	0.19 aA	0.13 aA	-31.58
Total polyphenol	2.87 aA	1.77 aA	-38.33	3.01 aA	2.05 aA	-31.89
Starch	3.57 aA	3.14 aA	-12.04	3.45 aA	2.10 aA	-39.13
Petroleum ether extract	0.51 aA	0.88 aA	72.55	0.95 aA	1.31 aA	37.89
Water soluble chlorine	1.92 aA	1.92 aA	0	1.97 aA	1.25 aA	-36.55
Potassium oxide	6.17 aA	6.05 aA	-1.94	7.51 aA	7.13 aA	-5.06

TABAL-3
COMPARISION OF AROMATIC CHEMICAL COMPONENTS IN TOBACCO STEMS BEFORE AND AFTER MICROWAVE EXPANSION

Aromatic component	Before expansion (μg/g)	After expansion (μg/g)	Change rate (%)
Aldehydes	17.45	21.68	24.24
Ketones	28.40	29.95	5.46
Alcohols	34.40	28.10	-18.31
Organic acids	44.96	52.72	17.26
Phenolics	1.51	1.18	-21.85
Esters	47.93	46.80	-2.36
Heterocycles	4.08	5.16	26.47
Olefins	64.62	70.08	8.45

olefins was significantly increased by 8.45 % ($0.01 < P < 0.05$), in addition, aldehydes, alcohols, acids, phenols and heterocycles changed with the most significant difference ($P < 0.01$) by 24.24 %, -18.31 %, 17.26 %, -21.85 % and 26.47 %, respectively.

As indicated in Table-4, there was slightly different in the contents of aroma components in tobacco stems for K326 and Hongda before and after microwave expansion. In general,

TABAL-4
COMPARISION OF AROMATIC CHEMICAL COMPONENTS IN TOBACCO STEMS BEFORE AND AFTER MICROWAVE EXPANSION (μg/g)

Aromatic component	K326		Hongda	
	Before expansion	After expansion	Before expansion	After expansion
Aldehydes				
3-Methyl butyraldehyde	0.60aA	1.05aA	0.43aA	0.94aA
2-Methyl butyraldehyde	0.32aA	0.67aA	0.25aA	0.56aA
3-Methyl-2-butenal	0.06aA	0.05aA	0.08aA	0.08aA
Caproaldehyde	0.13aA	0.12aA	0.16aA	0.14aA
Furfural	2.79aA	3.60aA	2.27aA	2.91bA
2-Pyridinecarboxaldehyde	0.09aA	0.12aA	0.09aA	0.12aA
Benzaldehyde	0.16aA	0.23aA	0.12aA	0.12aA
5-Methylfurfural	0.18abA	0.46bA	0.12bA	0.24bA
2,4-Heptandienal A	0.12aA	0.03aA	0.22aA	0.06aA
2,4-Heptandienal B	0.21aA	0.10aA	0.22aA	0.11aA
1H-Pyrrole-2-carbaldehyde	0.11aA	0.10bB	0.07aA	0.08bB
4-Pyridinecarboxaldehyde	0.07aA	0.08bA	0.09aA	0.10bA
Phenylacetaldehyde	3.33aA	4.09aA	2.77aA	3.69aA
Nonanal	0.13aA	0.09aA	0.18aA	0.10aA
5-Methyl-1H-pyrrole-2-carbaldehyde	0.18aA	0.20aA	0.09aA	0.12aA
2,6-Nonadienal	0.06aA	0.04aA	0.07aA	0.04aA
2-Phenylpropenal	0.17aA	0.17aA	0.15aA	0.12aA
Safranal	0.08aA	0.06aA	0.10aA	0.05aA
γ-Undecanolactone	0.62aA	0.44aA	0.56aA	0.40aA
Ketones				
Acetone	0.26bA	0.16bA	0.29bA	0.23bA
2-Methyltetrahydrofuran-3-one	0.27aA	0.56bB	0.16aA	0.47bB
2-Cyclopentene-1,4-dione	0.79aA	1.20bA	0.51aA	0.88bA
2-Furyl methyl ketone	0.22aA	0.74aA	0.12aA	0.40aA
Methyl heptenone	0.25aA	0.19aA	0.30aA	0.24aA
3-Methylcyclopentane-1,2-dione	0.12aA	0.13aA	0.15aA	0.16aA
1-(1H-pyrrol-2-yl)-ethanone	1.01aA	1.03bA	0.73aA	1.09bA
1-(3-Pyridyl)-ethanone	0.04aA	0.03aA	0.06aA	0.03aA
6-Methyl-2-heptanone	0.10aA	0.07aA	0.13aA	0.10aA
Pulegone	0.05aA	0.09bA	0.04aA	0.06bA
Solanone	5.29aA	5.57aA	6.35aA	6.04aA
β-Damascenone	0.80aA	0.64aA	0.64aA	0.74aA
β-Damascone	0.15aA	0.16aA	0.11aA	0.13aA
Geranylacetone	0.29aA	0.25aA	0.36aA	0.28aA
β-Ionone	0.64aA	0.47aA	0.69aA	0.57aA
Megastigmatrienone A	0.20aA	0.22aA	0.20aA	0.23aA
Megastigmatrienone B	0.54aA	0.54aA	0.58aA	0.64aA
Megastigmatrienone C	0.08aA	0.09aA	0.10aA	0.11aA
Megastigmatrienone D	0.58aA	0.58aA	0.79aA	0.80aA
2,3,6-Trimethyl-1,4-naphthalenedione	0.08aA	0.07aA	0.15aA	0.08aA
Solanascone	0.67aA	0.58aA	0.53aA	0.34aA
Farnesylacetone A	1.02aA	0.94aA	1.24aA	1.35aA
Farnesylacetone B	0.33aA	0.32bA	0.39aA	0.35bA
Alcohols				
Furfuryl alcohol	0.32aA	0.53bB	0.28aA	0.51bB
Benzyl alcohol	0.16aA	0.29bB	0.12aA	0.69bB
Linalool	0.05aA	0.03aA	0.06aA	0.06aA
Phenethyl alcohol	0.86aA	0.33bB	1.05aA	0.77abAB
Thunbergol	5.78aA	3.17aA	3.46aA	2.38aA
Phytol	4.00aA	3.64aA	4.45aA	4.02aA
Cembratriene-diol	5.62aA	4.72aA	8.19aA	6.96aA
Organic acids				
2-Methylbutyric acid	0.07aA	0.07bA	0.11aA	0.12bA
2-Furoic acid	0.23aA	0.37aA	0.20aA	0.45aA
Myristic acid	0.14aA	0.15aA	0.20aA	0.10aA
Pentadecanoic acid	0.52aA	0.61aA	0.52aA	0.39aA
Palmitic acid	20.45aA	26.80bA	22.52aA	23.66bA
Phenolics				
Phenol	0.11aA	0.06aA	0.15aA	0.09aA
2-Methoxyphenol	0.03bB	0.02cC	0.04bB	0.03bBC
2-Methoxy-4-vinylphenol	0.42aA	0.34aA	0.76aA	0.64aA
Esters				
Butyrolactone	0.14aA	0.10bA	0.13aA	0.13bA
Dihydroactinidiolide	0.24aA	0.23aA	0.25aA	0.26aA
Dibutyl phthalate	0.88aA	1.47aA	0.99aA	0.95aA
Methyl palmitate	2.22aA	2.45aA	3.20aA	3.95aA
Ethyl palmitate	4.14aA	3.79bA	5.27aA	4.78bA
Methyl linolenate	12.33aA	11.64aA	18.14aA	17.05aA
Heterocycles				
Pyridine	0.41aA	0.50aA	0.53aA	0.62aA
2-Pentylfuran	0.23aA	0.16aA	0.25aA	0.21aA
2-Acetyl-3,4,5,6-tetrahydropyridine	0.09aA	0.32aA	0.07aA	0.33aA
Polyolpyran	0.03aA	0.17aA	0.01aA	0.10aA
2-Acetyl-1,4,5,6-tetrahydropyridine	0.16aA	0.32aA	0.15aA	0.49aA
2,3-Dihydrobenzofuran	0.04aA	0.03aA	0.06aA	0.04aA
Benzothiazole	0.24aA	0.32aA	0.21aA	0.20aA
Thianaphthene	0.06aA	0.06aA	0.07aA	0.06aA
Indole	0.17aA	0.17bB	0.17aA	0.14bB
3-(1-Methylethyl)(1H) pyrazol-[3,4-b]pyrazine	0.26aA	0.21aA	0.29aA	0.22aA
2,3-Bipyridyl	0.29aA	0.26aA	0.29aA	0.23aA
Olefins				
Neophytadiene	34.37aA	34.26aA	29.51aA	35.09aA
Anthracene	0.32aA	0.40aA	0.42aA	0.33aA

ketones such as solanone, β-damascenone, β-damascone, megastigmatrienone, farnesylacetone endowed the smoke freshness, sweet and characteristic aroma of mature tobacco. In case of the esters, methyl palmitate, ethyl palmitate and methyl linolenate had the flavor of fat and wax, made the smoke mellowness. Dihydroactinidiolide could eliminate irritation during smoking. Olefins such as neophytadiene improved taste and spice for cut stems. Phenolics had a negative effect on the taste for its acerbity, the obvious decline for the contents of phenol, 2-methoxyphenol and 2-methoxy-4-vinylphenol after microwave treatment was favorable to the promotion of quality for cut stems[13]. In case of the acids, palmitic acid was more abundant than other acids. The significant raise of acids such as 2-furoic acid and palmitic acid played an important role in the mellowness of taste, adjustment of pH value and reduction of irritation for smoke. Heterocycles can endow characteristic aroma for smoke, compared with other heterocycles, pyridine, 2-acetyl-3,4,5,6-tetrahydropyridine, polyol pyran, 2-acetyl-1,4,5,6-tetrahydropyridine were totally increased by 19.15, 306.25, 575 and 161.29 %, respectively. Thunbergol and cembratriene-diol were important aroma precursors in tobacco,

the higher contents favored the formation of aroma matter, the distinct descend for the contents of thunbergol and cembratriene-diol indicated that microwave treatment promoted the transformation for aroma compounds, in addition, the decline for phytol can alleviatespicy flavors for smoke. Compared with the non-expanded tobacco stems, the amounts of aldehydes such as 3-methyl butyraldehyde, 2-methyl butyraldehyde, furfural, 2-pyridinecarboxaldehyde, 5-methylfurfural and 5-methyl-1H-pyrrole-2-carbaldehyde were obviously increased in the expanded tobacco stems, it can featured smoke sweet, improve density of smoke and make smoke elegant.

Conclusion

The effects of microwave expansion on the cell wall substances, conventional chemical compositions and aromatic components in tobacco stems were investigated. It is found that the contents of pectin, holocellulose, lignin and the total content of cell wall substances in Hongda and K326 shown a decrease in different degree after microwave expansion. Compared with the non-expanded tobacco stem, protein, nicotine, total sugar, total volatile acid, total polyphenol, starch and potassium oxide decreased, while the contents of petroleum ether extract were greatly increased. Seventy six aromatic components consisting of 19 aldehydes, 23 ketones, 7 alcohols, 5 acids, 4 phenols, 6 esters, 10 heterocycles and 2 olefins were indentified by GC-MS analysis in tobacoo stems, the total content of volatile components was increased by 12.32 μg/g after microwave expansion. The results showed that the microwave treatment was beneficial to the decomposition of the macromolecules and increasement of aromatic chemical components, cut stems prepared by microwave expansion had better sensory quality.

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