

Effect of Super Heated Steam Treatment on Physical Property and Smoke Component of Burley Cut Tobacco

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과열증기처리에 의한 Burley종 각초의 물리성 및 연기성분 변화에 관한 연구

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ABSTRACT : This study was carried out to investigate the effects of super heated steam on the physical and chemical changes of burley cut tobacco. Total sugar, total alkaloid, ether extracts, crude ash, total nitrogen and pH for leaf chemical constituents were analyzed. Filling power and fineness index for physical properties, and carbonyl compounds, phenol compounds, ammonia, pH, hydrogen cyanide nicotine, carbon monoxide, total volatile base and tar for smoke components were also analyzed. The cut tobacco treated with super heated steam showed significant decrease in total sugar and total alkaloid. The filling power of the sample treated with the super heated steam system was increased abruptly when heated at over 250°C. The fineness index showed similar tendency to that of common toast method. Super heated steam treatment slightly decreased carbonyl compounds, phenol compounds, hydrogen cyanide, nicotine, carbon monoxide and tar in the tobacco. Especially the decrease of ammonia was the most remarkable. The pH of smoke was a little different compared with that in the common toast. The sensory test results showed that, compared to the common conveyer moving system, the tobacco treated with super heated steam system brought out more roasted flavor, lowered impact, irritation, and sting, further improved aftertaste, and lowered bitterness. The super heated steam treatment method used in the studies is expected to give better filling power, mild taste and toasted odor of tobacco than that of the common method for toast treatment.

Key words : burley tobacco, super heated steam, expansion, toast.

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In burley tobacco processing methods, the heat treatment is known to have an important effect on chemical properties. Generally burley tobacco has lower sugar contents and higher contents of nicotine and nitrogen compounds than flue-cured tobacco. High contents of the irritants cause the smokers to sense more bitter taste and irritant than the flue-cured tobacco. Thus the tobacco researchers are trying to reduce the nitrogen compound and irritants, and develop good flavor and aroma through addition of sugar and reaction at high temperature condition.

Various studies have been carried out in order to improve the quality of low grade flue-cured and burley tobacco(Myung et al., 1989 ; Saburo et al., 1972). One example is study on weakening the bitter taste and improving chemical properties of low grade tobacco through water treatment.

Most studies are concentrated on reduction of the abhorrent component producing impact, irritation, sting and aftertaste through toasting the low grade flue-cured tobacco which has low sugar content and off-flavor. Studies on not only the heat treatment but addition of the sugar and organic acid are also attracting the attention of the researchers. However, this becomes a problem because the current conveyer belt system has difficulty in controlling the quality and has low speed (Chiba et al., 1971 ; Matskura et al., 1983 ; Matsukura et al., 1986) as well.

Thus, this study was carried out to investigate the differences between the toasting which use conveyer belt system, short time heat treatment using super heated steam(Arno, 1982 ; Charles et al., 1981 ; Francis et al., 1983. ; Fredric et al., 1988 ; Gus et al., 1984 ; Ronald et al., 1986 ; Werner, 1989). The irritant and harmful components in main stream and the filling power of cut tobacco were compared between the two methods.

Materials and Methods

Leaf (grade 1, 3) and cutter (grade 1, 3) of Korean

burley tobacco (1993, 1994 crop) were used for this experiment. Sugar(5%), licorice (1.6%) and additives were added to the sample and conditioned for 24 hours. After conditioning, the cut tobacco samples(cut width : 0.9mm) were treated by super heated steam for 3~7 seconds in the superheated steam system shown in Figure 1. The filling power of the treated sample was measured. The cigarette sample was prepared by rolling the treated sample in cigarette paper and the cigarette was 84mm long(tip length : 30mm, total ventilation : less than 5%) and sorted under the conditions : pressure drop of 130 ± 10 mm H₂O, and cigarette net weight : toasted sample cigarette was 740 ± 20 mg, 230℃ super steam treated sample cigarette was 680 ± 20 mg, 280℃ super steam treated sample cigarette was 615 ± 20 mg. The sorted cigarettes were smoked using one channel smoking machine (Heiner Borgwaldt Co. RM 20/CS) and total particulate matter(TPM) was determined. Nicotine was measured according to the CORESTA distillation method, CO was analyzed with CO analyzer(Heinr Borgwaldt Co. CO8), and CO₂ was analyzed with CO₂ analyzer(Simens Co. Ultramat 21P). Five selected cigarettes were smoked at CORESTA standard

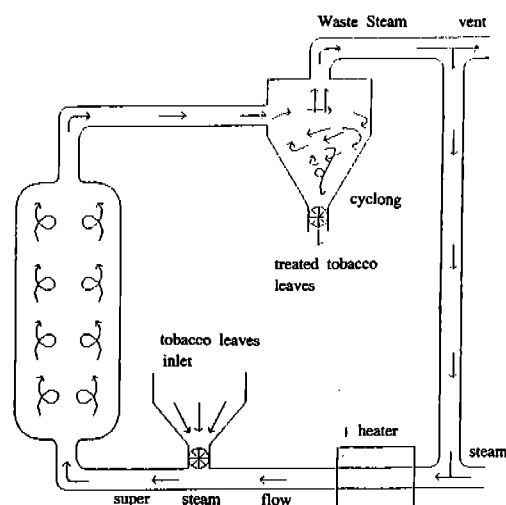


Fig. 1. Diagram of super heated steam treatment system of cut tobacco.

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condition for analyzing the aldehyde, phenol, hydrogen cyanide and ammonia.

Analysis of Aldehyde : The gas phase of smoke was collected using diameter 44 mm cambridge filter. The collected gas phase was passed through two gas washing bottles each filled with 250 ml of 1% p-nitrophenylhydrazine solution (p-nitrophenylhydrazine dissolved in 3 N-sulfuric acid) for 12 seconds. 24 hours later, the solution was extracted with 4 aliquots of 50 ml of diethyl ether and washed first with 50 ml of 3% sulfuric acid solution then with 50 ml of distilled water. The solution was dried over anhydrous magnesium sulfate. After filtering out the magnesium sulfate, 1 ml of normal hexadecan was added as the internal standard. The solution was concentrated using vacuum distillator and analyzed using HP 5890 Series II Gas Chromatography equipped with Supelco Co. SE - 54 Fused Silica Capillary Column (30m×0.25mm ID). Following conditions were applied : initial temp. : 150°C, initial time : 5 min., rate : 5°C/min, final temp. : 250°C, and final time : 30 min(Klimisch et al., 1976).

Analysis of Phenol : The particle phase of smoke was collected using diameter 44 mm cambridge filter. After separating the gas phase using the procedure described in the diagram below, the gas phase was esterified using BSTFA (N, O-bis(trimethylsilyl) trifluoro acetamide) and analyzed using HP 5890 Series II Gas Chromatography equipped with Supelco Co. SE - 54 Fused Silica Capillary Column (30m×0.25mm ID). Following conditions were applied : initial temp. : 40°C, initial time : 5 min., rate : 4°C/min, final temp. : 240°C, and final time : 30 min(Sakuma et al., 1984).

Analysis of Hydrogen Cyanide : The particle phase of smoke was collected using diameter 44 mm cambridge filter. The gas phase passed through cambridge filter was then sent through two gas washing bottles connected in series while cooling the

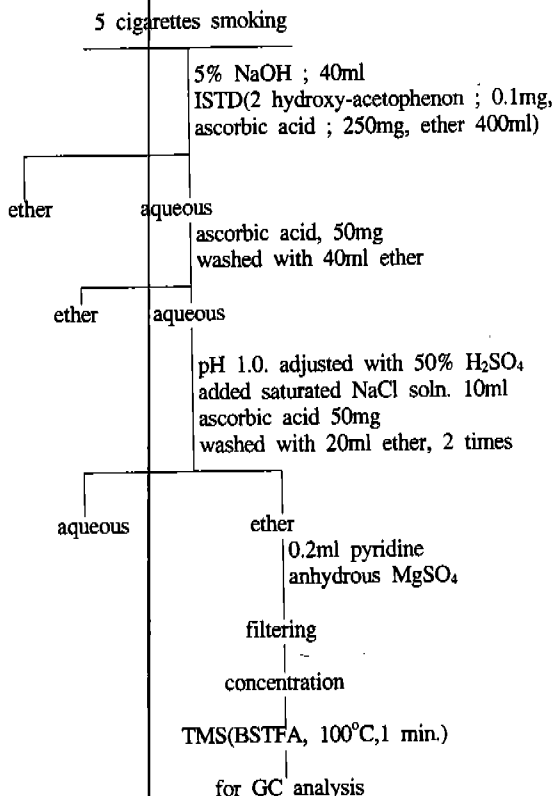


Fig. 2. Schematic procedure to analyze phenolic fraction.

bottles in dry ice acetone. The contents in gas washing bottle were transferred to a 500 ml graduated flask and filled to 500 ml mark with methyl alcohol. In a 50 ml volumetric flask, 5 ml of this solution and 5 ml of chloroamine T solution were added and filled to the mark with pyridine ester solution (1 ml diethyl acetone dicarboxylate, 100 ml pyridine, 500ml water). Within 9~15 min. after mixing the absorption was measured at 518 nm(Cephas et al., 1980).

Analysis of Ammonia : The particle phase of smoke was collected using diameter 44 mm cambridge filter. The gas phase passed through cambridge filter was then sent through two gas washing bottles connected in series and each filled with 50 ml of 0.1

M HCl. This solution was transferred to steam distillation apparatus and distilled after addition of 2 ml of 10 M NaOH solution. 10 ml of 0.1 M HCl solution was added to 100 ml volumetric flask and filled to the mark with the distillate. This solution was then transferred to a 250 ml beaker and 2 ml of 10 M NaOH was added while stirring. The electric potential (mV) was measured with ion meter (Sloan et al., 1976).

Analysis of Other chemical components : Other chemical components of tobacco leaves such as total nitrogen, total sugar, total volatile base, nicotine, etc. were investigated by book of analytical methods of tobacco & tobacco smoke, was established by Korea Ginseng & Tobacco Research Institute (Kim et al., 1991).

Results and Discussion

In the fast fluidization bed, the burley cut tobacco reacts with super heated steam for a short time. At this time, the temperature, flow rate, and turbulent flow formation play important role in heat transfer, chemical reaction, and puffing (Gus D. Keritsis, 1984). The super heated steam was used as the heat medium in order to prevent acidification or combustion due to contact with oxygen at high temperature. Also, the super heated steam has higher heat capacity than the air, and thus allows heat transfer during shorter period of time.

We suspect that at this time, the heat is transferred

even to the middle of the tobacco leaf tissue and the moisture in the tissue vaporizes, increasing the pressure within the tissue or the cell wall. As a result, the cell walls of palisad layer and spongy-like tissues swell and show puffing. This phenomenon can be observed in the microscope photograph in this paper and it was studied by Chang et al., (1972). However, some studies (Saburo et al., 1972) indicate that the sensory test of heat treated flue-cured tobacco shows presence of burnt smell and reduction of flavoring component. These results show that the super heated steam treatment is not adequate for puffing the flue-cured tobacco.

The addition of sugar compound and organic acid with heat treatment increase flavor and aroma of burley tobacco and remove dislike component. If the tobacco is heat treated with super heated steam for short period of time, then not only the filling power will increase but also the flavor and aroma will increase and dislike component can be reduced. Also, if fluidizing bed system of Fig. 1 is used, the disadvantage of conveyer system, irregular quality in low, medium, and high level product can be removed. The leaf component of the samples comparing the common conveyer toast system and the super heated steam system at various temperatures are as follows :

With increase in heat treatment temperature, amount of total alkaloid, total sugar, and total amino acid (Kim et al., 1985) content was decreased, and amount of ether extract, crude ash, and total nitrogen did not

Table 1. Chemical constitution of burley leaf (cutter 3) after the super heated steam treatment.

treatment condition	total alkaloid (%)	total sugar (%)	total amino acid (μ mol/100g)	ether ext. (%)	crude ash (%)	total nitrogen (%)	pH
toast*	1.98	2.6	1505	4.0	20.8	4.18	5.51
230°C**	1.74	2.1	1385	3.82	23.2	4.17	5.43
280°C**	1.61	2.1	1279	3.78	26.7	4.17	5.39

* heat treated at 150°C using conventional conveyer moving system of common toasting system.

** super heated steam treatment temperature.

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change much. The pH decreased somewhat with increase of the heat treatment temperature.

The filling power of the sample treated using the super super heated steam treatment system in Fig. 1 is shown in Table 2. With gradual increase in heat treatment temperature, up to temperature of 250°C, the filling power was relatively low ; however, past 250°C, the filling power increased abruptly.

The cut tobacco treated with super heated steam showed spongy-like tissue(Fig. 2). The result indicates that the super heated steam heated was permeated to the innermost part of of the tissue, and make the tissue to be puffed well.

With the increase in storage period, the filling power was decreased(Table3). Chang C.S., etc reported that the increase in storage period alters plant tissue and increases denaturalization, decreasing the filling power.

As shown in Table 4., with increase in temperature, the fineness index was similar to common conveyer

Table 2. Filling power (cc/g) of burley (Burley21) cutter leaves mixture (1:1 Mixed cutter 1, 3 and leaf 1, 3) heat treated at various temperatures.

Treated temp.(oC)	filling power(cc/g)	increase (%)
toast*	6.08	-
150**	6.39	5.1
200**	6.57	8.1
250**	6.82	12.1
280**	7.49	23.2
320**	7.81	28.4

* heat treated at 150°C using conventional conveyermoving system

** super heated steam flow rate: 20m/sec moisture content before treatment: 20% treatment time: 3-7sec

Table 3. Filling power (cc/g) of burley(Br21) cutter 1. stored for various times after casing compared with toast method.

storing time	treated moisture content	treated condition	filling power(cc/g)	increase (%)
toast*(no storing)	30%	150(°C), 10min.	6.14	-
after drying (no storing)	20%	280(°C),super steam	8.21	33.7
after storing for 1 year	20%	280(°C),super steam	7.93	29.2
after storing for 2 years	20%	280(°C),super steam	7.69	25..3

* moisture content for filling power : 13%

Table 4. Fineness index of burley(Burley21) cutter 1. treated at various super steam temperature after casing. (moistuer content: 13%)

Treated temp.(°C)	toast	230°C	280°C
fineness index*	3.27	3.19	3.17

* moisture content for fineness index : 13%

belt moving system. This is a typical phenomenon of expended tobacco with swollen tissues. and the result is similar to puffing method using organic solvent.

With increase in treatment temperature, the amount of aldehyde(Table 5) compounds decreased steadily. It is likely that the change in the amount of aldehyde compounds is little because it is mainly produced during pyrolysis step of plant combustion.

Phenol compounds(Table 6) decreased steadily with increase of temperature. It is likely that since phenol

Table 5. Carbonyl compounds in the smoke (ug/cig.) of burley cutter grade 3 at various temperatures.

carbonyl compounds	treatment condition		
	toas	230°C	280°C
formaldehyde	25.9	23.6	20.5
acetaldehyde	692.5	618.2	561.8
acetone	87.3	78.4	69.1
propion aldehyde	19.6	18.0	16.5
acrolein	14.4	13.2	11.3

Table 6. Phenol compounds in the smoke (ug/cig) of burley cutter grade 3 at various temperatures.

phenol compounds	treatment condition		
	toast	230°C (SHST)*	280°C (SHST)*
phenol	25.8	23.1	20.9
catecol	170.7	151.9	138.9
O-cresol	14.3	12.7	11.2
p-catecol	16.3	14.7	12.9
hydroquinone	91.3	80.3	73.8
hydroxybenzoic acid	7.9	6.9	6.2

* SHST : Super heated steam treatment.

compounds are produced during pyrolysis process of plants, the change before and after the heat treatment is little.

As shown in Table 7, depending on heat treatment condition, NH₃ content was decreased with increase of temperature, and pH was lower than that of the toast. The hydrogen cyanide, nicotine, CO, CO₂, TPM and tar content, and puff No. decreased little. If sorted by same net weight, it is expected that due to same net weight, not only nicotine but also the total smoke component will show less decrease.

Mixed cutter 1, 3 and leaf 1, 3 of burley was treated with super heated steam at 280°C using fast fluidization bed. The following smoke flavor profile (Table 8) shows sensory test results of the super heated steam treatment sample and the toast sample treated with conventional conveyer system at 150°C.

The mixture of burley (Br. 21) cutter leaves (mixed cutter 1, 3, and leaf 1, 3) sample treated with 280°C super heated steam using fast fluidization bed system was compared to the sample heat treated at 150°C using conveyer moving system.

The result of sensory test results on table 8. show that the fast fluidization bed system brought to the tobacco more roasted flavor than that from conventional conveyer moving system, and lowered impact, irritation, and sting, improved aftertaste. It was also lowered in bitterness. The tobacco from fast fluidizing bed system at 280°C however, had some scorched flavor.

Table 7. Smoke component of burley cutter (grade 3) after treatment of toast or super heated steam.

Treatment Condition	NH ₃ (ug/cig.)	pH*	HCN (ug/cig.)	nicotine (ug/cig.)	CO (ug/cig.)	CO ₂ (ug/cig.)	TPM (ug/cig.)	tar (ug/cig.)	puff No.
toast	40.5	6.3	223	1.18	17.7	42.0	14.5	13.4	6.6
230°C	19.4	6.5	199	1.07	15.8	37.8	13.2	11.1	6.2
280°C	8.1	6.6	176	0.96	14.1	34.0	11.8	10.8	6.0

* Blank pH : 9.7 (with cambridge filter)

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Toasted



Super heated steam treated

Fig. 3. Scanning electron micrograph of conventional toasted and super heated steam treated cut tobacco for the comparison of expansion.

Table 8. Comparison of smoke flavor profile between samples toasted with conventional toast and super heated steam.

Category	Intensity Scale									
	Low		Medium				High			
	1	2	3	4	5	6	7	8	9	10
Amplitude						○	△			
Irritation					○			△		
Sting				○			△			
Aftertaste					○			△		
Bitterness					○				△	
Roasted				△		○				

△ : toast sample by current conveyer system

○ : super heated steam treated sample at 280°C using fluidizing bed

Conclusion

1. The content of total sugar, total alkaloids, total amino acids were decreased significantly with increase of super heated steam temperature in the burley cut tobacco. The content of total nitrogen, and ether extract also decreased but to a lesser extent.
2. The cut tobacco treated with super heated steam was showed greatly increased filling power at over 250°C. After various storing time, the puffing was compared. With decrease in storing time, the filling power due to heat treatment increased.
3. Using electron microscope, the puffing of the tissues

before and after the treatment was observed and compared. Super heated steam treatment played a beneficial role on the swelling and puffing.

4. Among the smoke contents, the decrease in ammonia was great whereas carbonyl compounds, phenol compounds, hydrogen cyanide, nicotine, CO, TPM, and tar decreased considerably. Relatively high decrease in ammonia delivery is believed to be due to not only the net weight decrease effect but high temperature treatment effect. Control, sample treated at 230°C, and sample treated at 280°C showed relatively low decrease in in-smoke delivery, which is because the cigarette were sorted by different net weight. If sorted by same net weight, it is expected that due to same net weight, not only nicotine but also the total smoke component will show less decrease.
5. The sensory test result shows that compared to heat treatment at 150°C for 10 min. using conveyer method as in the current method, the super heated steam treatment lowers irritation, bitterness, impact, and sting, improves aftertaste, and brings out richer roasted flavor.

요 약

본 연구는 당류를 가한 버어리종 각초를 현재의 toast방법과 과열증기를 이용한 순간 고온열처리방법으로 처리한 후 이화학적 특성변화와 키크미를 검토하였다. 각각의 방법으로 처리한 각초에 대하여 화학성분으로는 전당, 전알칼로이드, 에테르 추출물, 조회분, 전질소, 전아미노산, pH등을 분석하였고, 물리성은 부풀성, 부스러짐지수를 측정하였다. 각각의 방법으로 처리된 각초로 제조한 쉐련의 주류연 연기성분중 carbonyl 화합물, phenol류, 암모니아, HCN, nicotine, CO, CO₂, TPM, Tar등의 변화를 분석하였다.

현행 토스트방법인 열풍 과 conveyer 벨트에 의한 열처리보다 fluidizing bed을 이용한 순간 과열증기 열처리방법으로 처리한 각초는 전당과 전알칼로이드가 온도의 증가에 따라 감소되었다. 팽승성은 과

열증기온도가 250℃ 이상일때 급격히 증가하였으며 부스러짐 지수는 현행 toast방법으로 처리한 시료와 유사하였다. 잎 조직의 팽화현상을 전자현미경을 사용하여 관찰 비교한 결과 현행 토스트방법과 conveyer 벨트에 의한 열처리보다 fluidizing bed을 이용한 순간 과열증기 열처리방법으로 처리한 각초의 스폰지층이 현저하게 부풀었음을 확인하였다. 연기성분중에서는 암모니아가 크게 감소하였고, carbonyl 화합물, phenol류, HCN, nicotine, CO, TPM, tar등이 전반으로 감소하였다. 켈련의 시기에 현행 토스트 처리방법으로 처리한 각초로 제조한 켈련보다 과열증기로 처리한 켈련시료가 자극성이 현저히 감소되었으며 구수하며 뒷맛도 좋은 것으로 나타났다.

Reference

1. An, K. Y. (1990) Study on the pH of the particulate matter in cigarette smoke. *J. of the Korean Soc. of Tob. Sci.* 12 : 3-7
2. Arno, Weiss, Jöhn Ulrich (1985) *U.S. patent No. 4,523,598*. Process for improving the filling capacity of tobacco material.
3. Cephas, H. S. (1980) Coulometric determination of hydrogen cyanide in cigarette smoke. *Beit. Tabakforsch. Int.* 10 : 106-110
4. Chang, C. S., W. H. Johnson (1972) High temperature convection drying of tobacco during curing, II. effect of air temperature and velocity on heat and mass transfer coefficients. *Tob. Sci.* 16 : 61-64
5. Charales, H. Hibbits, Chester, Va. (1981) *U.S. patent No. 4,407,306*. Methode for expanding tobacco with steam at high temperature and velocity.
6. Chiba, S., S. Ikegami, H. Kobayashi, Y. Nishikada, T. Murang, S. Tanaka (1971) Roasting of shredded stem. *Jpn. Tob. Salt Public Corp. Res. Inst. Sci. Pap.* 113, 157-169
7. Francis, V. Burde, Midlothian, Rogen Z. Delta Bured(1983) *U.S. patent No. 4,414,987*. Process for increasing the filling power of tobacco lamina filler.
8. Fredric, L. Rickettz(1988) *U.S. patent No. 4,791,942*. Process and apparatus for the expansion of tobacco.
9. Geltart, D. (1986) *University of Bradford, U.K. Gas fluidization technology*(High velocity fluidized beds, fluid bed heat transfer)
10. Gus, D. Keritsis, Richmond, H. Howard Sun(1984) *U.S. patent No. 4,458,700*. Process for increasing the filling power of tobacco lamina filler having a low initial moisture content.
11. Kim, C. H. et al.(1991) *Analysis methods of tobacco & tobacco smoke*(established by Korea Ginseng & Tobacco Research Institute)
12. Kim, Y. H. (1985) Changes in the some ingredients of low grade tobacco leaves by heat treatment *J. of the Korean Soc. of Tob. Sci.* 7 : 49-55
13. Klimisch, Hi-J., H. Wernicke and K. Meibner (1976) Gaschromatographische bestimmung von isopren, acetaldehyd und acrolein aus der gasphase von cigaretten rauch. *Beit. Tabakforsch. Int.* 8 : 350-353
14. Matsukura, M., K. Takahashi, S. Ishiguro, H. Matsushita, N. Miyauchi (1983) Composition of semivolatiles from roasted tobacco. *Agr. Bio. Chem.* 47 : 2281-2285
15. Matsukura, M., S. Ishiguro (1986) Improvement to aroma and taste by adding roasted tobacco volatiles to cigarettes. *Agr. Bio. Chem.* 50 : 3101-3106
16. Myung, Pyung keun and Cheon Suk Kim (1989) The formation of pyrazine on toasting condition in burley and flue-cured tobacco leaves. *J. Pharm. Sci. (ChungNam National University)* 5 : 9-13
17. Ronald, D. (1986) *U.S. patent No. 4,625,736*. Method and apparatus for expanding tobacco with water.
18. Saburo, Ikegami (1972) Toasting of flue-cured tobacco. *Agr. Chem. Soc. Japan* 114 : 185-196
19. Sakuma, H., M. Kusama, K. Yamaguchi and S. Sugawara (1984) The distribution of cigarette smoke components between mainstream and side

stream smoke. *Beit. Tabakforsch. Int.* 12 : 251 – 258

20. Solon, C. H. and G. P. Moris(1976) Determination of unprotonated amonia in whole cigarette smoke. *Beit. Tabakforsch. Int.* 8 : 362 – 365
21. Werner, Hirsch(1989) *U.S. patent No. 4,844,101.* Apparatus for expanding comminuted tobacco meterial.