Journal of the Korean Society of
Tobacco Science Vol. 29, No. 2, 98~103 (2007)
Printed in Republic of Korea

A Comparison of Menthol Migration from Fillers to Filters and Mainstream Smoke in Leaf Tobaccos

Shin Baek*, Kun-Soo Kim, Jae-Jin Kwag and Si-Hyung Jo
KT&G Central Research Institute
(Received November 13, 2007)

ABSTRACT : This study compares menthol migration from fillers to filters and mainstream smoke in a type of leaf tobacco and according to the moisture content differences at the range of 11~15 %. The leaf tobacco used in this study consisted of Korea flue cured upper leaves B1O (KFUB1), A4OR (KFUA4), lower leaves C1L (KFLC1), CD4L (KFLCD4), burley upper leaves A3T (KBUA3), lower leaves D3W (KBLD3), Orient Basma I/III (OB), Orient Izmir B/G (OI), expanded tobacco (KET), and reconstituted sheet (KRC). Menthol migration to the filter and mainstream was measured under constant conditions for 80 days with intervals of 20 days. In the comparison between flue cured types, there were significant differences in the filter parts, as follows. KFUB1(34.4 %) KFUA4(37.4 %), KFLC1(43 %) and KFLCD4 were 55.7 %. In the comparision between other types of leaf tobaccos, KFUB1 was 34.4 % and KET was 52.6 % at filter parts. In the methol transfer to mainstream smoke was 16.5~24.2 %. The menthol migration to filters was measured based on the moisture content of 11~15 % for the KFUB1 after storing it for 80 days. The menthol migrations were 36~40 % at the moisture content of 11~15 %, respectively. The transfers to mainstream smoke were 12.8~15.8 %.

***Key words* : Transfer, migration, affinity, mainstream smoke**

In recent years, many cigarette companies have applied menthols using various methods. In particular, a cut tobacco addition, menthol applicator (Kaymich), and silver foil addition method have been widely used. It is important to conduct basic studies on menthol migration to filters and transfer to mainstream smoke, regardless of the methods used. The menthol used in cigarette industries presents specific characteristics compared to other compositions. Thus, a small amount of menthol was applied in general cigarettes to remove off-tastes and was used to present the characteristics of fresh and cool in menthol cigarettes. Because the menthol has an important position in cigarette industries as mentioned above, studies on menthol migration and transfer were performed (Wong *et al.*, 1988) in tobacco types and are currently being conducted. Menthol cigarettes have been produced by applying menthol to mixed leaf tobaccos based on these basic studies. Studies on the effects of applying a certain plasticizer, which be used to increase the hardness of filters in the menthol migration and transfer, such as diacetate, triethylglycol, and triacetin and of a certain humectant, such as propylene glycol and

*연락저자 : 305-805, 대전광역시 유성구 신성동 302 번지, KT&G 중앙연구원
*Corresponding author : *KT&G Central Research Institute, 302 Shinseong-dong, Yuseong-gu, Daejeon 305-805, Korea(phone : 82-42-866-5524; fax : 82-42-866-1949 ; e-mail : bs@ktng.com)*

glycerol, were performed. In addition, a radio labeling process was applied to configure the menthol molecular as ^{14}C in order to investigate the distribution of menthol after smoking. Studies on differences between filters (cellulose, paper) and effects of the menthol delivery for the total specimen were also performed. This paper compares the difference between the menthol migration to filters and the transfer to mainstream smoke according to the storage period by applying menthol to domestic tobacco types, such as flue-cured (4 types), burley (2 types), orient (2 types), expanded tobacco (1type), and reconstituted sheet (1type). In addition, this study investigates the difference between menthol migration and transfer after storing it up to 80 days and applying menthol to the domestic flue cured KFUB1, which has changed in its moisture contents as 11~15%.

MATERIALS AND METHODS

Cigarette manufacturing and selection conditions

The cigarette manufacturing was performed based on the encapsulated pressure drop(EPD) of 143 ± 3 mmH_2O due to the difference in densities and filling powers in tobacco types.

A type of synthetic menthol, which is produced by Symrise company, was applied by 0.8 % for each unit weight of cigarettes. Cellulose acetate was used as a filter, and the filter length was 24 mm. In addition, a type of non-ventilated product was used as tip paper, and 6 % of plasticizer was also used. Cigarettes were selected from the weight range presented in encapsulated pressure drop(EPD). The selected cigarette was stored at 25 ℃ and mesured according to the storage period.

Analysis of migration

The instrument used in this study was a Hewlett Packard 5890 gas chromatography(GC). The GC analyzing condition was that the injector and detector temperatures were to 230 ℃and 250 ℃, respectively, and the GC oven temperature were increased from 50 ℃ to 230 ℃with the intervals of 3 ℃/min. A DB-WAX (60 m × 0.32 mm i.d.) was used as a column. Each sample was also classified as either a filter or a filler according to the storage period, and was putted into 250 mL triangle flasks. Then, 100 mL of the anethole internal standard(1 g of a 0.5 mg/mL ethanol solution) was added. In addition, these samples were shaken at 150 rpm for 3 hours and were left for 1 day. Then, measurements were taken.

Analysis of transfer

Menthol was obtained using a CORESTA TPM trap method with a smoking machine (Heinr Borgwaldt RM20). The trapped cambridge filter was inserted into a 250 ml triangle flask. Then, 100 mL of anethol internal standard(1g of a 0.5 mg/mL ethanol solution)ISTD was added tothe flask. In addition, the sample was shaken at 150 rpm for 3 hours and was leftfor 1 day. Then, measurements were performed using the GC.

RESULTS AND DISCUSSION

The analysis results for 80 days storage can be summarized as shown in Table 1.

The menthol, which was storaged for 80 days in all tobacco types, was continuously migrated as a filter. In the comparison between the KFUB1 and the KFLC1, the KFLC1 presented a high migration ratio of about 8.6 %. The migration ratio presented high values for the lower grade in the upper leaf. Differences in the migration ratio in the lower leaf presented a higher value of about 3.0 % more than that of the upper leaf (Fig. 1).

For the migration ratio of KFUB1 after 80 days, the filler and filter presented 65.6 %, 34.4 %

Table 1. Ratios of the menthol migration in tobacco types for the storage period (unit: %)

Tob. types	Grades	Storage period (days)							
		20		40		60		80	
		Filler	Filter	Filler	Filter	Filler	Filter	Filler	Filter
KF	U B1O	77.7	22.3	71.8	28.2	68.1	31.9	65.6	34.4
	U A4OR	75.3	24.7	69.1	30.9	65.4	34.6	62.6	37.4
	L C1L	71.7	28.3	63.8	36.2	59.7	40.3	57.0	43.0
	L CD4L	65.8	34.2	56.6	43.4	45.6	50.4	44.3	55.7
KBC	U A3T	81.7	18.3	69.1	30.9	63.2	36.8	57.4	42.6
	L D3W	74.6	25.4	63.6	36.4	57.3	42.7	51.7	48.3
O	B	82.8	17.2	76.4	23.6	70.5	29.5	64.2	35.8
	I	81.6	18.4	74.5	25.5	70.2	29.8	63.6	36.4
KRC	SHEET	56.7	43.3	49.6	50.4	45.6	54.4	41.6	58.4
KET		60.4	39.6	54.7	45.3	51.1	48.9	47.4	52.6

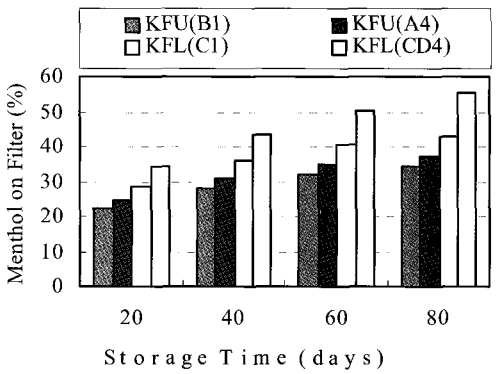

Fig. 1. Menthol migration of flue-cured tobaccos.

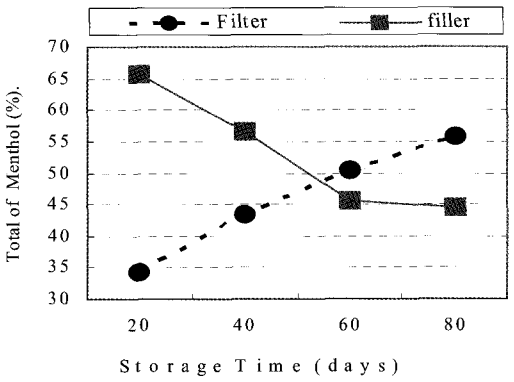

Fig. 3. Menthol distribution in the KRC after 80 days.

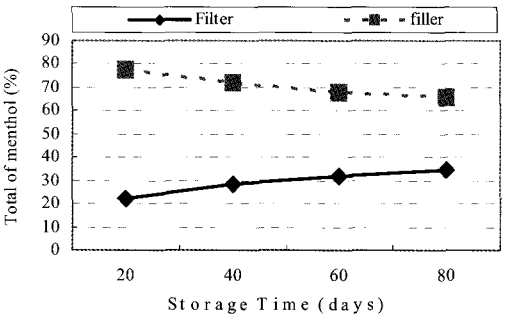

Fig. 2. Menthol distribution in the KFUB1 after 80 days.

(Fig. 2), Filler and Filter of KRC presented 41.6 %, 58.4 % (Fig. 3). For the migration ratio of the burley after 80 days, the lower leaf (48.3 %) presented a 5.7 % higher value than that of the upper leaf (42.6 %). In the comparison between the Orient Basma (I/III) and the Izmir (B/G), there was a slight difference between the two cases (35.8 % and 36.4 %), and the migration ratio was relatively lower than other tobacco types. In the comparison between the KRC and the KET, the migration was conducted for filters exceeding 50 % (58.4 % and 52.6 %)

where the KRC presented 5 % more migration than that of the KET.

Based on the results previously presented, the migration from filler to filters can be noted as KFLCD4 > KFLC1 > KFUA4 > KFUB1 in flue-cured leaves. In the case of the burley, orient, and KRC and KET, the migration orders can be noted as KBLD3 > KBUA3, Basma (I/III) = Izmir (B/G), and KRC > KET, respectively. In general, the migration to filters showed the order of KRC > KET > KB > KF > O (Fig. 4).

These results were due to the characteristics of tobacco leaves. The high migration ratio was due to the low affinity between the leaf and the menthol in which the menthol was unable to deeply penetrated the structure of leaves. In addition, it is evident that the interaction force between the leaf and the menthol was weak due to the poor interaction between the structure or composition of leaves and the menthol.

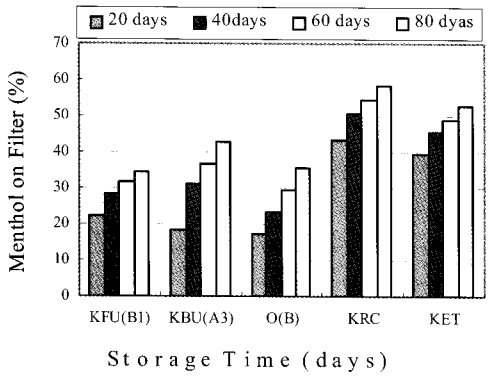

Fig. 4. Menthol migration of leaf tobacco types

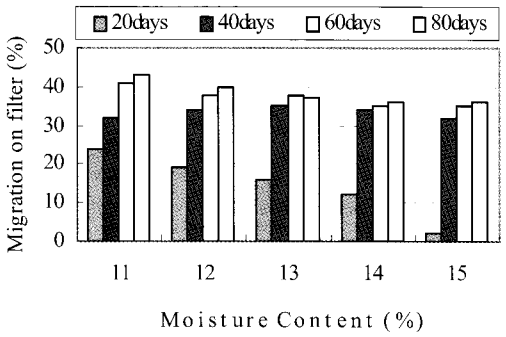

Fig. 5. Menthol migration according to the moisture content for the storage period.

In the amount of the menthol migration according to the moisture content, there were differences at the range of 11~15 % in the early period of 20 days, but the migration presented a similar tendency after 40 days.

It is evident that this similar tendency can be presented by decreasing the relative interaction according to the increase in the storage period even though the migration to filters was small due to the withdrawal of the menthol while the moisture content of fillers was at a high level after 20 days.

Transfer to mainstream smoke

Table 2 presents the menthol transfer to mainstream smoke according to the storage period.

The menthol transfer to mainstream smokes after 80 days was increased. The transferred amount of menthol was increased in proportio to the added amount of menthol.

Table 2. Menthol transfer to mainstream smokes by the storage period (unit: %)

Tob. types	Grade	Storage period (days)			
		20	40	60	80
KF	U B1O	16.2	16.8	17.7	18.9
KB	U A3T	15.5	15.9	16.2	16.5
O	B	15.2	16.8	18.1	19.2
KRC	SHEET	18.2	20.7	22.4	24.2
KET		13.6	16.4	18.2	20.6

In the comparison between the KFUB1 and the KBUA3 after 80 days, the menthol transfer to mainstream smokes of the KFUB1 (18.9 %) presented a higher value than that of the KBUA3 (16.5 %). In the comparison between the KFUB1 and the OB, the menthol transfers were 16.2 % and 15.2 %, respectively, in the early stage (after 20 days). However, the OB presented a slightly higher value (19.2 %) than that of the KFUB1(18.9%) after 80 days.

In the comparison between the KRC and the KET, the menthol transfers to mainstream smokes were 18.2 % and 13.6 %, respectively, in the early stage.

In addition, the KRC presented 3.6 % higher value than that of the KET after 80 days.

In general, the KRC presented the highest value, and the menthol transfers of the KET, OB, KFUB1, and KBUA3 were 20.6 %, 19.2 %, 18.9 %, and 16.5 %. These results can be noted as KRC > KFUB1 > KBUA3 > OB > KET for the early stage (after 20 days) and KRC > KET > KFUB1 = O > KBUA3 for the time elapsed (after 80 days). Fig. 6 highlights these relationships.

The KET and KRC presented high increasing ratios, 6.0 % and 7.0 %, compared to other leaves.

The increase in the menthol transfer to mainstream smoke after 80 days was due to the fact that the menthol, which has a high affinity to a type of plasticizer, migrated to filters and

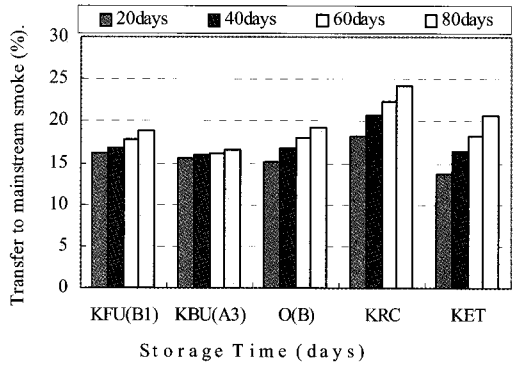

Fig. 6. Smoke menthol transfer to different tobacco types for the time elapsed.

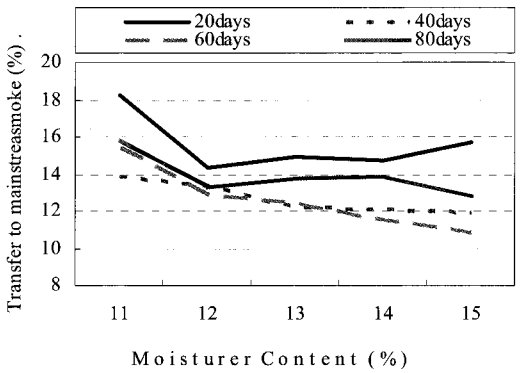

Fig. 7. Menthol transfer ratios to mainstream smoke by moisture and storage period.

Table 3. Smoke menthol transfer to different moisture content for the time elapsed

Mos. Con.(%)	Part	Storage period (days)			
		20	40	60	80
11	Cambridge filter	0.81	0.7	0.71	0.72
	Filter	1.05	1.45	1.47	1.51
	Tran. ratio(%)	18.2	13.9	15.4	15.8
12	Tran. ratio(%)	14.3	13.4	12.9	13.3
13	Tran. ratio(%)	14.9	12.2	12.4	13.8
14	Tran. ratio(%)	14.7	12.1	11.5	13.9
15	Tran. ratio(%)	15.7	11.9	10.8	12.8

was eluted as smoke. It can be expected that this increase continued because the extra amount of menthod being accommodated in the CA(cellulose acetate) filter will be emitted as smoke in the long period of storage. However, the transfer to mainstream smoke was increased or decreased due to the level of the elution from the CA.

By applying menthol after configuring the moisture content of the KFUB1 as 11~15 %, the menthol transfer to mainstream smoke according to the period was measured after storing it for 80 days. The lower moisture content (11 %) presented a more efficient transfer to mainstream smoke. In general, the transfer ratio was decreased according to the increase in the storage period.

If the interaction between the moisture and the menthol was small, the as same as the menthol transfer to filters, the migration ratio was increased. However, if the moisture content was high, and the storage period significantly lengthen, the interaction between the moisture and the menthol was increased, or the menthol transfer was decreased due to the interaction between the moisture and the plasticizer included in filters.

CONCLUSIONS

The menthol migration from filler to filters can be noted as KFLCD4 > KFLC1 > KFUA4 > KFUB1 in flue-cured leaves. In the case of the burley, orient, and KRC and KET, the migration orders can be noted as KBLD3 > KBUA3, Basma (I/III) = Izmir (B/G), and KRC > KET, respectively. In general, the migration to filters showed the order of KRC > KET > KB > KF > O. The menthol transfers of mainstream smoke of the KET, OB, KFUB1, and KBUA3 leaf tobaccos were 20.6 %, 19.2 %, 18.9 %, and 16.5 %. These results can be noted as KRC > KFUB1 > KBUA3 > OB > KET for the early stage (after 20 days) and KRC > KET > KFUB1 = O > KBUA3 for the time elapsed (after 80 days).

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