

# EFFECT OF CERTAIN LIQUID FILTER ADDITIVES ON MENTHOL DELIVERY

By JOHN G. CURRAN<sup>1</sup>

The objective of this project was to determine if liquid additives on a filter affect the rate of menthol migration from the tobacco to the filter and the amount of menthol delivered by the cigarette. The additives were applied to the filters by a brush applicator system, and the filters were attached to mentholated tobacco columns by a laboratory tipping apparatus. Respectively, the concentrations of the liquid additives, the percentages of menthol that migrated from tobacco to filter, and the amounts of menthol delivered after 6 wk of storage were: 5% triacetin, 24%, 0.38 mg; 8% triacetin, 32%, 0.36 mg; no additive, 10%, 0.32 mg; 5% triethylene glycol diacetate, 28%, 0.31 mg; 8% triethylene glycol diacetate, 34%, 0.27 mg; 8% triacetin and 6% glycerol, 31%, 0.27 mg; 8% triacetin and 10% glycerol, 31%, 0.27 mg; 8% triacetin and 6% 1,2-propanediol, 31%, 0.27 mg; 8% triacetin and 10% 1,2-propanediol, 42%, 0.22 mg. The application of plasticizer to the filter increased the rate of menthol migration from the tobacco to the filter; it also affected the amount of menthol delivered. No correlation was observed between the rate of menthol migration from the tobacco to the filter and the amount of menthol delivered. For example, cigarettes having filters bonded with 8% triacetin and 8% triethylene glycol diacetate had comparable rates of menthol migration but there was a significant difference between the amounts of menthol that they delivered. The most efficient menthol delivery was obtained from a cigarette having a filter bonded with 5% triacetin.

## INTRODUCTION

It is well documented (1, (2), (3) that menthol migrates from the tobacco column of a cigarette to its filter during storage in a cigarette pack. It is logical to assume that casings might affect the affinity of tobacco for menthol and that liquid additives on a cellulose acetate filter might affect its affinity for menthol. It has been reported (1) that the amount of menthol in a filter affects menthol delivery, since menthol retention by a filter decreases as the concentration of menthol in the filter increases. If the assumption is correct that liquid additives affect the affinity of a filter for menthol, then they might also affect menthol delivery.

The purpose of this work was to determine whether the addition of triacetin, triethylene glycol diacetate, glycerol, or 1, 2-propanediol to cellulose acetate filters affects either the affinity of a filter for menthol or the amount of menthol delivered by a cigarette.

## MATERIALS AND METHODS

### *Cigarettes*

All experimental mentholated filter cigarettes were constructed in our laboratory. The tobacco columns were 64 mm in length and were obtained from 100-mm men-

tholated cigarettes purchased in Kingsport, Tennessee. The filters were constructed from 3.3 den./fil, 39,000 total denier cellulose acetate tow. The liquid additives were applied to the tow with a brush applicator. The filter and tobacco column were attached with a laboratory tipping apparatus. The menthol content of these cigarettes was  $2.0 \pm 0.1$  mg.

### *Storage and Testing of Cigarettes*

Immediately after construction, the cigarettes were sealed in cigarette packs and stored for one, three, and six weeks. Each pack contained 16 cigarettes. Six cigarettes were used in the menthol distribution experiments. These cigarettes were separated into filter and tobacco segments before being analyzed. Ten cigarettes were used in the smoking experiments. They were smoked according to the Federal Trade Commission's specifications with a Phipps and Bird automated smoking machine. During the smoking of the different cigarettes, the number of puffs varied less than 3%. The smoke from five mentholated cigarettes was collected on a Cambridge filter pad; two pads were combined before being analyzed.

### *Analysis*

Ethanol was used to extract menthol from the six cellulose acetate filters, the six tobacco columns, and the two Cambridge filter pads. A known weight of internal standard (pentadecane) was added to each extract. The extracts were shaken for 2 hr before being analyzed by gas chromatography. The gas chromatographic unit was equipped with a single  $3\frac{1}{2}$ -ft  $\times$   $\frac{1}{8}$ -in. stainless steel column packed with Chromsorb W (60 to 80 mesh) coated with 10% w/w Castorwax. Operating conditions were as follows: column and detector temperature 165°C; injector temperature 250°C; helium and hydrogen flow 30 ml/min; air flow 360 ml/min.

## RESULTS AND DISCUSSION

### *Migration of Menthol in a Cigarette*

The effect of triacetin and triethylene glycol diacetate on the affinity of a cellulose acetate filter for menthol during storage in a cigarette pack is shown in **Figure 1**. The percentage of menthol on all of the filters increased with storage. Filters bonded with 8% triethylene glycol diacetate contained the highest percentage of menthol, while filters with 0% plasticizer contained the lowest percentage of menthol. The percentage of menthol migrating from the tobacco to the filter is a measure of the affinity of the filter for menthol. The order of plasticizer

<sup>1</sup> Research Laboratories, Tennessee Eastern Company, Division of Eastman Kodak Company, Kingsport, Tennessee 37601. Contribution received February 11, 1975; U.S. Pat. No. 3,917,000.

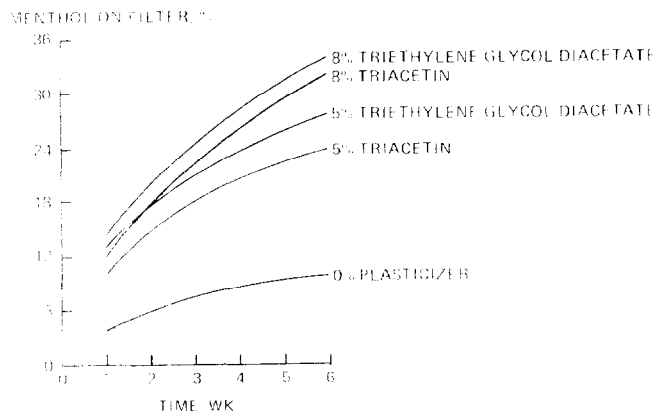
affinity for menthol is 8% triethylene glycol diacetate >8% triacetin >5% triethylene glycol diacetate >5% triacetin >0% plasticizer. These results show that cellulose acetate filters without plasticizer have a lower affinity for menthol than filters bonded with either triacetin or triethylene glycol diacetate; that filters bonded with triethylene glycol diacetate have a lower affinity for menthol than filters bonded with triethylene glycol diacetate; that the affinity of filters for menthol increases as plasticizer concentration increases. The migration rate of menthol from the tobacco to the filter also increases as plasticizer concentration increases.

The effect of glycerol and 1,2-propanediol on the affinity of a triacetin (8%) bonded cellulose acetate filter for menthol is shown in **Figure 2**. The percentage of menthol on all the filters increased with storage. Filters treated with 1,2-propanediol contained a higher percentage of menthol than filters with either glycerol or 0% additive. There was no difference in the percentage of menthol on filters treated with 6 and 10% 1,2-propanediol. This was also true for filters treated with 6 and 10% glycerol. These results show that treating triacetin-bonded cellulose acetate filters with glycerol does not alter their affinity for menthol; that treating the cellulose acetate filters with 1,2-propanediol increases their affinity for menthol; that the affinity of filters treated with glycerol or 1,2-propanediol remained constant as the additive concentration increased from 6 to 10%.

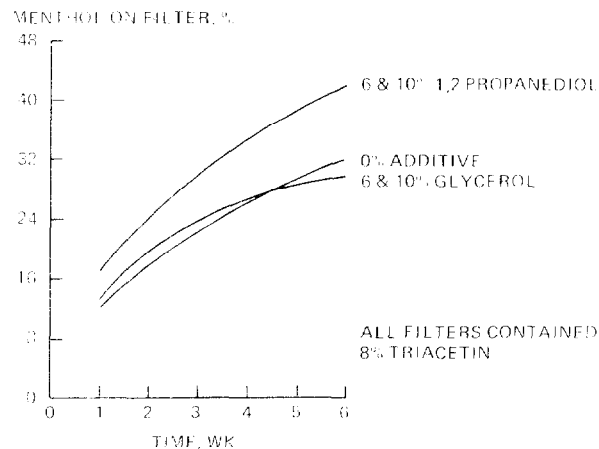
**Menthol Delivery**

The effect of triacetin and triethylene glycol diacetate on menthol delivery is shown in **Table 1**. These deliveries fluctuated with cigarette age. The average menthol deliveries are statistically different. As plasticizer concentration increased, menthol delivery decreased. The largest delivery was obtained from a cigarette with a filter bonded with 5% triacetin and the smallest delivery was obtained from a cigarette with a filter bonded with 8% triethylene glycol diacetate. Thus, both the type and amount of plasticizer significantly altered menthol delivery.

There was no correlation between the menthol delivery of these cigarettes and the affinity of their filters for menthol. Of the five filters, the one bonded with 8% triacetin had the second highest affinity for menthol, yet cigarettes with this filter had the second highest menthol delivery; nonbonded filters had the lowest affinity for menthol. However, two other cigarettes, whose filters



**Figure 1. Effect of plasticizer on menthol migration.**



**Figure 2. Effect of glycerol and 1,2-propanediol on menthol migration.**

had a greater affinity for menthol, delivered more menthol. One explanation for these results is that cigarette smoke changes the affinity of a filter for menthol.

The effect of glycerol and 1,2-propanediol on menthol delivery is shown in **Table 2**. The menthol delivery decreased with cigarette age, while the delivery of the control cigarette fluctuated with age. Therefore, the addition of glycerol and 1,2-propanediol to a filter reduces menthol delivery.

There was no correlation between the menthol delivery of these cigarettes and the filter's affinity for menthol. The affinity of a triacetin (8%) bonded cellulose acetate filter was not altered by the addition of glycerol, yet there was a 15% difference in the menthol delivery of these two filter cigarettes.

In conclusion, triacetin, triethylene glycol diacetate, and 1,2-propanediol affect the affinity of a cellulose acetate filter for menthol. The affinity of a cellulose acetate filter for menthol changes as cigarette smoke is presented to it. Glycerol and the above compounds also affect the amount of menthol delivered. Both type and concentration of plasticizer affect menthol delivery.

**Table 1. Effect of plasticizer on menthol delivery**

Plasticizer	Menthol delivered,* mg				X
	1 wk	3 wk	6 wk		
Control (0%)	0.35	0.34	0.32		0.34
Triacetin (5%)	0.37	0.41	0.38		0.39
Triacetin (8%)	0.34	0.37	0.36		0.36
Triethylene Glycol Diacetate (5%)	0.34	0.35	0.31		0.33
Triethylene Glycol Diacetate (8%)	0.32	0.32	0.27		0.30

\* Each value is the average of three runs.

**Table 2. Effect of glycerol and 1,2-propanediol on menthol delivery**

Additive	Menthol delivered,* mg				X
	1 wk	3 wk	6 wk		
Control (0%)	0.34	0.37	0.36		0.36
Glycerol (6%)	0.32	0.31	0.27		0.30
Glycerol (10%)	0.32	0.30	0.27		0.30
1,2-Propanediol (6%)	0.31	0.29	0.27		0.29
1,2-Propanediol (10%)	0.31	0.27	0.22		0.27

All filters contained 8% triacetin.  
\* Each value is the average of three runs.

**LITERATURE CITED**

1. Brozinski, M., Dolberg, U., and G. Lipp. Distribution of menthol in the tobacco, filter and smoke from menthol cigarettes. *Beitr. Tabakforsch.*, 6 (3):124-130. 1972.
2. Curran, J. G. Delivery of menthol from cigarettes containing either a mentholated filter or mentholated tobacco. *Tob. Sci.* 16:40-42. 1972.
3. Reihl, T. F., Shockley, L. L., and M. L. Reynolds. Menthol distribution and transfer. *Tob. Sci.* 17:10-11. 1973.