MAINSTREAM SMOKE MENTHOL DELIVERY AFTER USING DIFFERENT FLAVOUR APPLICATION METHODS IN FILTER PRODUCTION

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Abstract

Menthol without a doubt is one the most popular flavours in the cigarette industry. It is applied to the filter, tobacco and even packaging. Indeed, menthol cigarette almost certainly has mentholated filter which has been enriched with the flavour one way or the other. To keep the customer satisfied with the favoured product the taste of mentholated cigarette has to be the same whenever customer picks up another pack. Hence, it's crucial to have a very stable amount of menthol in the filter right away from the moment of production to transportation and storage all the way to the final processing on the cigarette machine.

In this study we quantitatively analysed mainstream smoke of standardised tobacco column attached to standardised filter containing the same concentration of either encapsulated menthol, directly injected pure hot melt menthol crystals or menthol solution in propylene glycol. Smoking was done by ISO method using rotary Borgwaldt RM20D smoking machine and menthol analyses performed on Agilent 5973N GC-MS equipment.

This study also compared different packaging materials used for flavoured filter storage. We used multilayer composite polyamide/polyethylene (PAPE), pure polypropylene (PP) and pure polythethylene (PE) bags. Quantity of menthol in different filters was checked 24h post production. Then filters were kept in different packaging in standard warehouse conditions for 30 days and then menthol extracted for comparative analysis.

GC-MS conditions

The GC-MS analyses were performed on a Agilent Technologies 6890N GC system combined with an inert 5973 mass selective detector with interchangeable (EI), fast GC oven and a 7683 series injector - autosampler. Data handling and system operations were controlled by the Chemstation software. The injector temperature was set at 280 ˚C. The GC oven temperature program was as follows: 45˚C, ramped to 80C at 6˚C min-1, then ramped at 30˚C min-1 to 250˚C. The mass spectrometer was operated in the electron ionization (EI) mode at 70eV. The mass range was scanned from 50 to 250 m/z at full-scan mode.

Materials

1. Menthol 99,5% (Sigma Aldrich);
2. Anethol 99,5% (Sigma Aldrich);
3. Methanol 99% (Sigma Aldrich);
4. Propylene Glycol 99,9% (Algol Chemicals);
5. Filters 108/7,8/340/0CU with different menthol application.

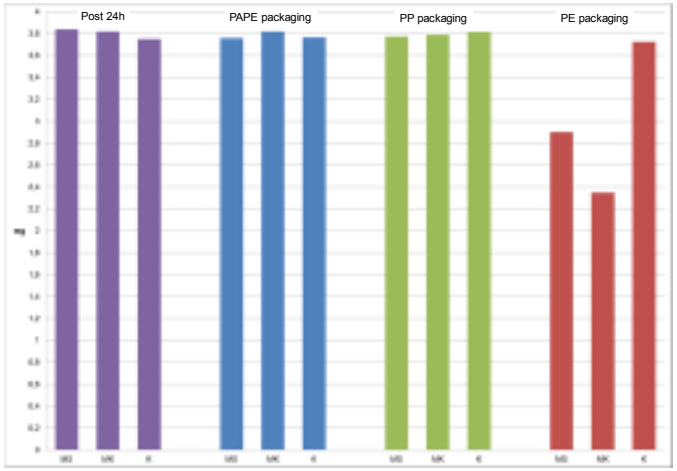
Experimental

20 filters of each type were produced using different menthol application methods: menthol solution with propylene glycol (MS), menthol crystals (MK) and capsules (K) keeping menthol dosing constant at 3.8mg per cigarette tip. 20 cigarettes of each type were manually made by taking Coresta Monitor 7 tobacco column and attaching mentholated filter tips which were pre-cured for 24h. Then these filter rods and cigarettes were kept in different packaging for 30 days in warehouse at 15-25C and RH 40-60%. Menthol concentration was determined at both intervals (24h post production and 30 day later) from filter rods and cigarettes kept in different packaging.

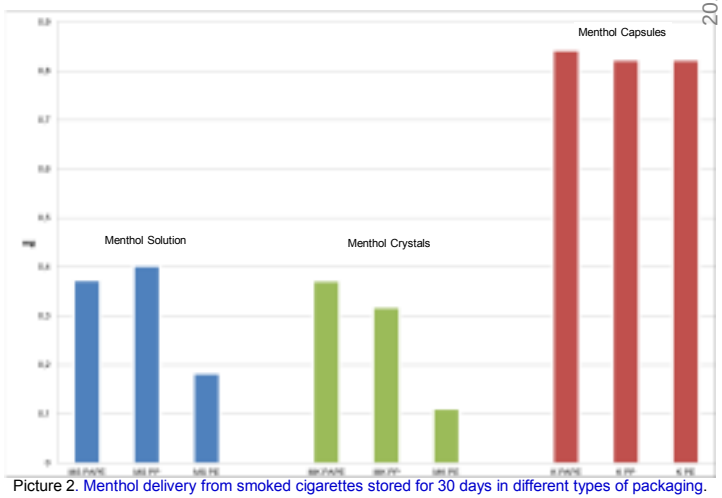
Cigarettes were smoked under ISO conditions. Capsules in menthol cigarettes were crushed just before placement in the smoking machine holder. Menthol delivery to the smoker was estimated by quantifying menthol trapped on the Cambridge filter. No menthol was found in the gas phase. For menthol extraction we used methanol with anethol as internal standard.

For each menthol application method smoked and unsmoked cigarette butts were also sliced longitudinally and chopped into 10mm pieces. Menthol was extracted using methanol with internal standard.

Each type of protective bag was 70-80 µm in thickness.



Picture 1. Menthol concentration in unsmoked filter tips kept in different packaging for 30 days



Picture 2. Menthol delivery from smoked cigarettes stored for 30 days in different types of packaging.

Results

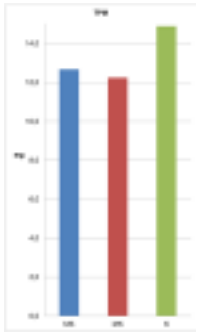
The menthol analysis doesn't show any significant changes in flavour concentration in polyamide/polyethylene (PAPE) versus polypropylene bags (PP). The major changes in menthol concentration were observed in polyethylene bags (PE): amount of menthol decreased by 24% in filters with menthol solution (MS) and by staggering 44% in filters with menthol crystals (MK) compared to the measurements 24h post production. Storage in PE bags didn't affect menthol concentration in capsule filters because PE has good water barrier properties and do not let the capsule to dry. On the other hand, PE had much worse barrier properties for gas permeability. The analysis showed that PE bags are not suitable for any long term storage of menthol filters produced by direct flavour injection independent of whether the menthol is in propylene glycol solution or in pure crystal form (Picture 1).

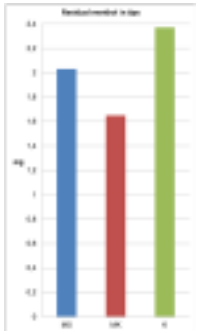
Menthol delivery from smoked cigarettes showed similar results. The cigarettes which were stored in PE bags had much lower delivery of flavour. Menthol solution and menthol crystals gave almost the same results in the PAPE and PP bags with concentration of flavour remaining very stable after 30 days of storage (Picture 2).

Menthol delivery in capsule cigarettes was stable independent of bags used for storage. In addition, flavour delivery was two times higher (for the same initial loading) compared to MS and MK cigarettes. We believe that during long storage directly applied menthol slowly migrates along the acetate tow fibers and spreads all over the filter, plug wrap paper and tobacco column with some menthol loss thus occurring even in the side stream smoke. Capsule menthol load, on the other hand, remains safely locked and gets released from crushed capsule just before smoking thus giving intense flavour boost directly to the cigarette consumer.

Another interesting and repeated observation was 16% higher TPM in capsule cigarettes versus MS and MK versions (Picture 3). This could be due to filter squeezing to crush the capsule resulting in the regular architecture of acetate tow filaments in the filter being significantly distorted.

Finally, residual menthol in smoked filter tips showed higher level of flavour in capsule filter butts despite already observed high delivery levels (Picture 4).


Picture 3. TPM of MS, MK and K cigarettes.


Picture 4. Residual menthol in smoked cigarette butts.

Conclusions

Polyethylene bags are not suitable for menthol filter storage. Polypropylene and composite polyethylene/polyamide films have very comparable barrier properties but we prefer the latter due to it's superior mechanical strength and durability during transportation.

Cigarettes with menthol capsule give more than double menthol delivery compared to other direct flavour application methods for the same initial menthol loading.

Direct injection application methods give deliveries of just about 10% from primary menthol load.

Capsule cigarettes give higher TPM.