



Machine-smoking studies of cigarette filter color to estimate tar yield by visual assessment and through the use of a colorimeter

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ABSTRACT

This paper explores using the intensity of the stain on the end of the filter (“filter color”) as a vehicle for estimating cigarette tar yield, both by instrument reading of the filter color and by visual comparison to a template. The correlation of machine-measured tar yield to filter color measured with a colorimeter was reasonably strong and was relatively unaffected by different puff volumes or different tobacco moistures. However, the correlation of filter color to machine-measured nicotine yield was affected by the moisture content of the cigarette. Filter color, as measured by a colorimeter, was generally comparable to filter extraction of either nicotine or solanesol in its correlation to machine-smoked tar yields. It was found that the color of the tar stain changes over time. Panelists could generally correctly order the filters from machine-smoked cigarettes by tar yield using the intensity of the tar stain. However, there was considerable variation in the panelist-to-panelist tar yield estimates. The wide person-to-person variation in tar yield estimates, and other factors discussed in the text could severely limit the usefulness and practicality of this approach for visually estimating the tar yield of machine-smoked cigarettes.

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1. Introduction

A standardized machine-smoking method for determining cigarette tar and nicotine yields was introduced in 1967 for comparing cigarette brand styles (Federal Register, 1967). As reported at its introduction, this method was not intended to reflect what any individual human smoker would receive when smoking a cigarette (FTC, 1967), and at the time of its introduction it had been long-recognized that there was a wide range of human smoking behaviors (Bradford et al., 1936 and Pillsbury, 1996).

Biomarker studies examining individual smoker urinary excretion of nicotine and its major metabolites confirm that, even among smokers smoking the same type of cigarette, there is a wide range of per cigarette nicotine intake (Byrd et al., 1995, 1998; Roethig et al., 2005; St. Charles et al., 2006).

Kozlowski (1981) proposed to use the intensity of the tar stain at the mouth-end of the cigarette filter to indicate the intensity with which the cigarette was smoked. The initial intent was to allow smokers to infer approximate tar and nicotine yield estimates from their cigarette and to assess whether the ventilation holes of the cigarette had been blocked while the cigarette was being puffed. It was expected that the technique might only provide ordinal scale information. However, Kozlowski reasoned that, even

with its imperfections, filter color could provide useful information to smokers (see also Kozlowski and Pillitteri, 1996).

Kozlowski et al. (1982) followed this initial proposal with a study to determine how well smokers could match cigarettes smoked with differing numbers of puffs to a reference set of commercial color swatches chosen to represent the filter colors at those different numbers of puffs. It was found that, after smoothing the data, the average of the panelists' ratings correlated well with the number of puffs taken.

Husset et al. (2002) proposed the use of a color chart similar to the one developed by Kozlowski to communicate tar yield to smokers and included example color charts. It was stated that the color chart allows the smoker to estimate the tar yield which is inhaled, though the values should only be taken as indicative because of the imprecision in the perception of colors. No data were provided to indicate how effectively smokers could use the color charts to estimate tar yields.

Rickert et al. (1994) discussed results that called into question the use of reference color swatches as a communication tool. They suggested relying on instrument measurements of the color of cigarette filters instead of relying on visual comparison to commercial color swatches. The filters were measured using reflectance spectrometry. This suggested the use of filter color as a laboratory tool rather than as a communication tool to smokers.

In later work, Rickert et al. (2004) found the correlations between the machine-generated smoke yields of several compounds and the filter color as measured by a spectrophotometer to

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generally be strong, though the regression equations relating filter color and constituent yield were not always the same for different cigarettes. It is likely that the strength of those correlations to filter color was a result of the correlation of the measured smoke constituents to tar yield. Strasser et al. (2006) and O'Connor et al. (2007) also examined filter color as a laboratory tool. O'Connor et al. (2007) concluded that filter color may be an inexpensive and reliable way to assess smoking topography in population-based studies, and reported that further validation studies were being conducted.

The objectives of this paper were to examine the two potential uses of filter color discussed above:

1. The use of instrument readings of the intensity of the stain on the filter, also referred to in this publication as “filter color,” as a laboratory tool for the estimation of the tar and nicotine yields of machine-smoked cigarettes. The relationships of an instrument reading of filter color to the machine-generated tar and nicotine yields of the cigarette were explored. This examination included a comparison to two other potential approaches to estimating cigarette tar and nicotine yields: nicotine and solanesol extraction from the filter (Watson et al., 2004; St. Charles et al., 2006; Shepperd et al., 2006).

This paper only examines the filter color of machine-smoked cigarettes; however, the intent of many researchers is to use filter color as a tool for estimating the tar, nicotine, and other constituent yields of a cigarette to a smoker (O'Connor et al., 2007). Smokers can smoke cigarettes with a variety of different puffing topographies and under a variety of different temperature and humidity conditions. It seems likely that the more consistent the correlation of filter color to cigarette tar and nicotine yields under different puffing regimens and cigarette moistures, the more likely it is to accurately estimate the tar and nicotine yields of a cigarette to a smoker. To determine the consistency of the correlation of filter color to machine-smoked tar and nicotine yields, different puff volumes and different cigarette moistures were examined to determine their impact on the relationship between filter color and machine-smoked tar and nicotine yields.

A study was also conducted to determine whether filter color changes over time. This was examined with a colorimeter study and with a panel study.

2. The use of filter color as a potential tool for visually estimating machine-smoked tar yield. This was evaluated in studies with adult smoker panelists estimating the tar yields of machine-smoked cigarettes using reference templates to represent the filter color corresponding to various machine-derived cigarette tar yields.

In examining whether filter color could be used by panelists to estimate machine-generated cigarette tar yields, another series of machine-smoking studies was conducted. In one study, panelists compared filter stains of test cigarettes to a series of actual filters from one cigarette brand style that had been machine-smoked to a range of tar yields using multiple machine-smoking conditions. This provided a best-case point of comparison since photographic or other representations of the filters are not expected to be as good as the filters themselves. Additional studies examined the use of reference templates that included pictures of filters from a single cigarette brand style that had been machine-smoked to a range of tar yields.

2. Methods

2.1. Smoking machines

The smoking machines used in these studies were linear smoking machines employing adjustable puff volumes, puff durations,

and puff intervals. Total particulate matter (TPM) was collected onto a 44 mm Cambridge Filter Pad (CFP). Unless noted otherwise, cigarettes and CFPs were acclimated under controlled environmental conditions ($60 \pm 3\%$ RH and $75 \pm 2^\circ\text{F}$) for at least 24 h before testing, and spent filters, when stored, were kept in a freezer (-5°C) in laminated foil bags.

The neoprene washer was removed from the Cambridge Filter pad adapter prior to smoking. A mark was made on the cigarette 9 mm from the end of the filter to indicate the proper insertion depth and the cigarettes were inserted to that depth. See the [Supplementary Appendix](#) for a discussion and illustration of some of the artificial patterns that can form on the end of the filter if the neoprene washer is in place or if the cigarette is inserted fully into the elbow base if the neoprene washer is removed.

2.2. Tar and nicotine yields

Tar and nicotine were determined according to ISO standards (ISO Standard 3308, 2000; ISO Standard 4387, 2000; ISO Standard 8454, 1995; ISO Standard 10315, 2000; ISO Standard 10362-1, 1999) with appropriate adjustment for the number of cigarettes smoked per replicate analysis. Nicotine and water were determined by gas chromatography (GC) from isopropanol extracts of TPM collected on the CFP. Tar was calculated by subtracting the weights of nicotine and water from the weight of TPM.

A set of standardized smoking conditions was defined in 1967 (Federal Register, 1967) employing a 35 cc puff of 2-s duration taken once per minute. Though known to not mimic the smoking characteristics of individual smokers, these conditions have been employed by cigarette manufacturers and others as a set of standardized smoking conditions for comparing the tar and nicotine yields of cigarettes. The development of this method is described in Pillsbury (1996). These smoking conditions will be referred to herein as the Cambridge Filter Method and abbreviated as “CFM.” Cigarettes that have an 11 mg tar yield (for example) under CFM conditions will be referred to as having an 11 mg CFM tar yield.

2.3. Filter extraction

Solanesol and nicotine from filters were determined from the 10-mm portions at the mouth-end of spent filters. Nicotine and solanesol extraction could not be conducted on the same cigarettes, and, therefore, when filter extraction was employed, the cigarette filters from some ports on the smoking machine were used for nicotine extraction and others were used for solanesol extraction.

Nicotine from spent filters was determined by GC using a modified version of Coresta (1989). Spent filters were extracted by rotation for 1 h in methanol containing carvone as an internal standard. The resulting extract was analyzed by GC using a capillary column.

Solanesol was extracted from the 10-mm portion of the mouth-end of spent filters by dissolving the filter segment with acetonitrile and analyzed using high performance liquid chromatography (HPLC) based on Coresta Recommended Method No. 52 (2002).

2.4. Colorimeter

A Hunter Laboratories ColorQuest XE Colorimeter was used to quantify the color of the filters used in this study. Colors were measured in reflectance mode using the 1976 CIE $L^*a^*b^*$ system (Wysszecki and Stiles, 2000), with D65 as the illuminant and a 10° standard observer angle. The parameter L^* represents lightness or darkness with $L^* = 100$ representing white and $L^* = 0$ representing black, positive a^* represents red, negative a^* represents green, positive b^* represents yellow, and negative b^* represents blue. The CIE $L^*a^*b^*$ system was chosen as a device-independent model to

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