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Polycyclic aromatic hydrocarbons, carbon monoxide, "tar", and nicotine in the mainstream smoke aerosol of the narghile water pipe

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Abstract

A smoking machine protocol and yields for "tar", nicotine, PAH, and CO are presented for the standard 171-puff steady periodic smoking regimen proposed by Shihadeh et al. [Shihadeh, A., Antonios, C., Azar, S., in press. A portable, low-resistance puff topography instrument for pulsating, high flow smoking devices. Behavior Research Instruments, Methods, and Computers; Shihadeh, A., Azar, S., Antonios, C., Haddad, A., 2004. Towards a topographical model of narghile water-pipe café smoking: A pilot study in a high socioeconomic status neighborhood of Beirut, Lebanon. Pharmacology Biochemistry and Behavior 79(1)]. Results show that smokers are likely exposed to more "tar" and nicotine than previously thought, and that pyronsynthesized PAH are present in the "tar" despite the low temperatures characteristic of the tobacco in narghile smoking. With a smoking regimen consisting of 171 puffs each of 0.53 l volume and 2.6 s duration with a 17 s interpuff interval, the following results were obtained for a single smoking session of 10 g of *mo'assel* tobacco paste with 1.5 quick-lighting charcoal disks applied to the narghile head: 2.94 mg nicotine, 802 mg "tar", 145 mg CO, and relative to the smoke of a single cigarette, greater quantities of chrysene, phenanthrene, and fluoranthene. Anthracene and pyrene were also identified but not quantified. The results indicate that narghile smoke likely contains an abundance of several of the chemicals thought to be causal factors in the elevated incidence of cancer, cardiovascular disease and addiction in cigarette smokers.

1. Introduction

Studies on the chemical composition, toxicity, and carcinogenicity of cigarette smoke generated using a smoking machine are widely used to predict and understand health effects of smoking, and to compare effects of varied tobacco blends, delivery methods, and puffing behavior. They complement in-vivo and epidemiological studies of smoking and have contributed significantly to a better understanding of cigarette smoke toxicity and carcinogenicity (Hoffmann et al., 2001) and to generating the evidence needed for anti-tobacco policies and action. More than 4800 compounds, including 69 carcinogens, have been identified in cigarette smoking machine studies that span a period of more than 40 years (Hoffmann et al., 2001). In contrast, we have been able to locate only four studies (Rakower and Fatal, 1962; Hoffman et al., 1963; Sajid et al., 1993; Shihadeh, 2003) of the chemistry of narghile smoke in the open English-language literature, in which a comparatively small range of chemical compounds were investigated. In none of these studies are CO or PAH, two major toxic agents in tobacco smoke, quantified using relevant narghile smoking parameters.

This relative paucity in research on narghile smoke chemistry cannot be attributed to the insignificance of the topic. The narghile water-pipe is prevalent in Southwest Asia and North Africa, and in recent years has shown a sharp rise in popularity particularly among young people (Chaaya et al., 2004). National and local surveys in Kuwait (Memon et al., 2000), Egypt (Mo-

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hamed et al., 2003), Syria (Maziak et al, 2004), and Lebanon (Shediac-Rizkallah et al., 2002; Jabbour, 2003) have found that 20–70%, and 22–43% of the sampled populations has ever smoked or currently smokes the narghile, respectively. Anecdotal evidence in the form of newspaper reports (e.g. McNicoll, 2002; Barnes, 2003; Landphair, 2003; Edds, 2003; Gangloff, 2004) and "hookah bar" advertisements in college papers and on the internet suggest that water-pipe smoking is catching on in North America and Europe as well.

With a dearth of scientific studies, researchers, public health officials, and the general public have had little data to assess the potential hazards of water-pipe smoking. Even so, a widespread perception among smokers, and even physicians (Kandela, 1997), is that the water through which the smoke bubbles filters the toxic components, rendering the practice considerably less harmful than cigarette smoking.

While it is tempting to do so because of the sheer volume of available cigarette smoke data, the water-pipe is so different from the cigarette that data on smoke composition and toxicity cannot be extrapolated from the later to the former. Apart from the obvious differences in smoke delivery, involving long passages and a water bubbler in the case of the narghile, the smoke aerosol generation process is also considerably different. Whereas the cigarette involves a self-sustaining combustion of roughly 1 g of dried and shredded tobacco in several puffs with volumes on the order of tens of ml, the argileh utilizes an external heat source (charcoal) to largely devolatalize typically 10–20 g of heavily flavored and hydrated tobacco paste (in the case of *mo'assel* tobacco; see Shihadeh (2003) for a description of narghile components and typology) with puff volumes an order of magnitude greater and with characteristic tobacco temperatures several hundreds of degrees Celsius lower. Thus there is a need for developing research methods and smoke composition data specific to the narghile water-pipe.

Our previous work (Shihadeh, 2003) on the mainstream narghile smoke chemistry showed that it contains significant amounts of "tar" and nicotine, and that even for the same total smoked volume, the results varied considerably depending on the machine puffing regimen used. We also found that while the "tar" of a single narghile smoking session was startlingly high typically two orders of magnitude greater than that produced from a single cigarette it was likely to have a different composition due to the much lower temperature of the tobacco in the narghile. We anticipated therefore that the proportion of pyrosynthesized 4- and 5-ring PAHs responsible for much of the carcinogenicity of "tar" should be considerably lower than for cigarettes. It was also found that approximately 5 g of charcoal were consumed in the course of a single smoking session, suggesting the possibility of large quantities of carbon monoxide being delivered to the smoker.

The current study follows up on these issues. The objectives were to (1) provide new data for "tar" and nicotine using an updated, and considerably more intense, puffing model which was derived from precise smoking topography measurements of 52 smokers in the field, (2) quantify the amount of CO delivered to the smoker, and (3) quantify PAH in the particulate phase so as to allow an informed interpretation of the high quantities of "tar" with respect to carcinogenic PAH compounds.

2. Materials and methods

2.1. Smoking machine

A first-generation digitally programmable smoking machine was developed for this study (see Fig. 1). The programmable inputs to the smoking machine include puff duration, flow rate, interpuff interval, and total number of puffs. The smoking machine relies on a high-flow vacuum pump which is modulated by an electronic proportional control valve. The control valve signal is generated using feedback control provided by a PC-based data acquisition and control (DAQ) system. The feedback is provided by an electronic mass flow meter whose output signal is constantly sampled and recorded in a look up table containing valve control voltages and the resulting flow rates. Prior to the first smoking session, a calibration program is run which increments the valve control voltage signal from zero to the maximum value, thus initializing the lookup table. Once a smoking session is started, the initial values in the table are dynamically updated as flow conditions change (e.g., as pressure drop across filters increases, or as filters are replaced). We have found that this control scheme provides less than 1% error in the session cumulative puff volume.

The smoke aerosol was split into two streams immediately downstream of the narghile hose outlet and each stream drawn through a single 47 mm Gelman type A/E glass fiber filter pad before being re-combined. Each pad was held in a transparent polycarbonate holder, also manufactured by Gelman. This two parallel-filter configuration required eight sets of filters (i.e. seven filter changes during each smoking session) to limit the particulate loading to circa 100 mg per filter. (ISO 4387:1991 specifies that up to 150 mg of tobacco smoke condensates may be collected on a 47 mm glass fiber filter pad.) A secondary filter was placed downstream of the 2-to-1 junction and weighed before and after each run to ensure that there was no breakthrough. We also experimented with single and quadruple parallel filter configurations (also with a total of 16 filters per smoking session to limit loading), and found that the two filter set up was most convenient to use given the on-line filter

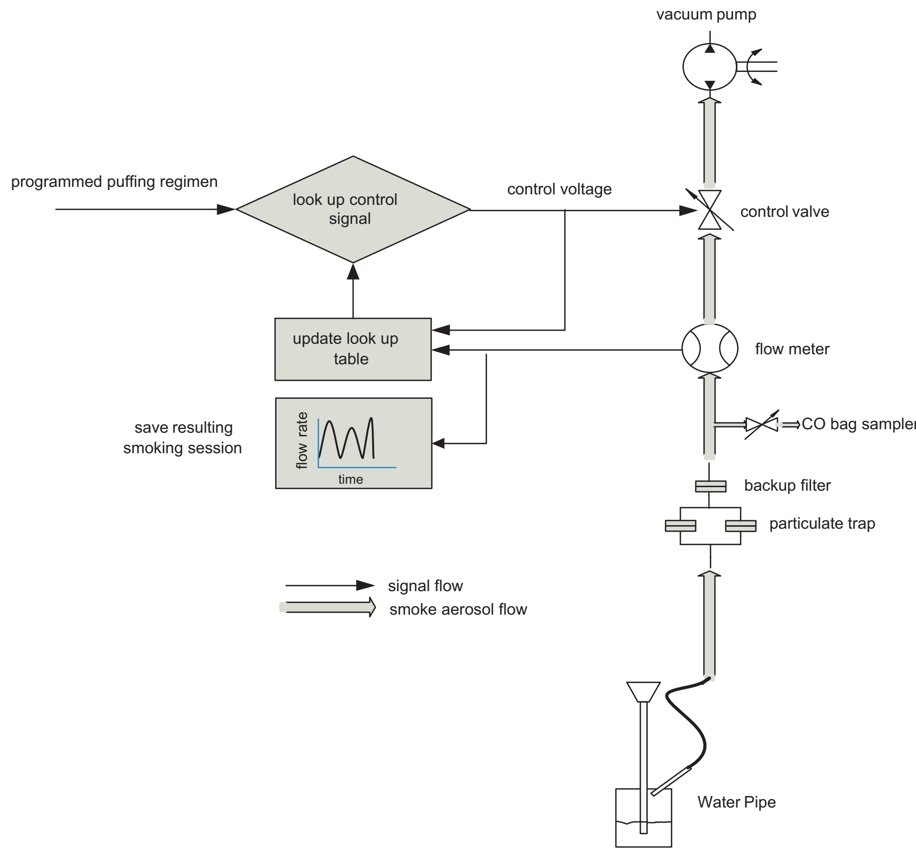

Fig. 1. Schematic of the digital smoking machine.

changes during a smoking run. Filter holders were equipped with quick-release polypropylene fittings to help ensure that the operator could change the filters in the span of the 17 s interpuff interval.

To limit evaporative losses when the filters were removed from the smoking machine, the downstream fitting of each filter holder had a spring-loaded automatic shutoff valve mechanism that immediately closed when the holder was removed from the machine. The upstream side was simply manually sealed with a rubber end cap immediately upon removal. We did not fit an automatic shutoff valve on the upstream side as this would likely have caused particle transport losses in the narrow passages of the valve.

For CO determination a fraction (circa 9% vol) of the smoke aerosol flow was sampled from the main flow smoke path through a critical orifice by a miniature sealed diaphragm pump that exhausted into a 10 l tedlar grab sample bag (SKC, Inc. #232-08). The pump was activated during each puff by the DAQ system via a digital solid state relay.

2.2. Machine smoking protocol

Except for the changes to the smoking regimen, filter replacement schedule, and coal application method discussed below, all other procedures given in Shihadeh (2003) were followed, covering aluminum foil preparation, bowl water changes, tobacco type, quantity, storage, and homogenization, and narghile preparation.

2.2.1. Smoking regimen

A smoking topography study of 52 volunteer smokers in a popular café in the Hamra neighborhood of Beirut was undertaken to determine realistic smoking parameters for the smoking machine study. The study made use of an electronic smoking topography instrument to record narghile flow rate as a function of time. Based on time-segmented analyses of the recorded smoking sessions, we derived a steady periodic smoking model of the ''average'' smoking session, consisting of 171 puffs, each of 0.53 l volume and 2.6 s duration. The interpuff interval was 17 s. The smoking topography instrument and the 52 smoker pilot study are further described in Shihadeh et al., in press, 2004, respectively.

2.2.2. Coal application

Because the new smoking regimen was considerably more intense than the previously used 100 puff regimen, we found that the previously sufficient single quick-light charcoal disk (Three Kings brand, Holland) was consumed well before the end of the smoking session, rendering the last 20 puffs nearly smoke-free. Smokers normally add coals during a smoking session to subjectively maintain the "strength" of the smoke. We performed several experiments with varying coal application schemes to identify one which gave realistic yet diminishing smoke yields toward the end of the smoking session, as was commonly observed in the field. To do so, we monitored the tobacco burn fraction in the head, the puff-resolved total particulate matter (TPM), and visually inspected the burned tobacco charge at the end of the session.

Fig. 1 shows typical TPM data collected for three coal application schedules involving 1, 1.5, and 2 charcoal disks. The 1.5 and 2 coal cases were begun with a single coal disk which was augmented at the 80th puff with an additional pre-lit half or whole coal disk. Half disks were made by running whole disks through a high-speed band saw. As shown, smoke production for the single coal case dropped precipitously after 100 puffs, whereas the 2-coal case over-produced in the second half of the session, leading to an excessively burned-out (i.e. entirely blackened) tobacco charge by the session's end. The 1.5 coal condition appeared to give a relatively consistent smoke production rate throughout the smoking session, while leaving a part of the tobacco charge relatively moist, as is normally the case with real smoking. To further tune the 1.5 coal procedure, the timing of the second coal application was moved from the 80th to the 105th puff, yielding somewhat lower tobacco burn fractions close to the median 46% burn fraction found in our previously reported pilot field study of 28 smokers (Shihadeh, 2003). Table 1 provides a summary of the TPM and tobacco burn fractions for the four variations. Condition C was used for the remainder of the study.

It should be noted that these quick-light charcoal disks are commonly used in narghile smoking and are invariably sold wherever narghile tobacco is sold. Smokers rely on them when convenience dictates, since the more traditional charcoal requires a small grill and longer lighting times. Nonetheless, we estimate that while self-lighting charcoal disks are used in an important fraction of narghile smoking sessions, the majority of narghile smoking, especially in restaurants and cafés, is done using the traditional charcoal, which is inherently heterogeneous in size and shape. In the interest of reproducing experiments and simplifying the procedures, we have used the standard quick-lighting charcoal disks.

Table 1
Effect of coal quantity and timing of second application on tobacco burned and TPM generated

Schedule	Coal disks	Second application puff number	Tobacco burned, g	TPM, g
A	1	N/A	3.78	1.15
B	1.5	80	4.90	1.64
C	1.5	105	4.66	1.38
D	2	80	5.08	1.92

Schedule C was used in this study.

2.2.3. Filter changes

As mentioned above, eight pairs of filters were used during each run to prevent filter overloading. The filter pairs were changed at 40, 60, 80, 95, 110, 125, 140, and 171 puffs, yielding an average loading of 90 mg TPM per filter.

2.3. Chemical analysis

Thirty-two replicate smoking sessions were conducted. For every smoking run, the weight of the loaded, foil-wrapped head was recorded before and after each smoking run, as were the filter holders and the coal disks. TPM was determined as the total weight increase of the 16 filter holder assemblies.

To determine water content, the 16 filter pads were combined in a 250 ml bottle and stirred for 20 min with 50 ml of ethanol. 5 ml of the resulting solution was then added to the reaction chamber of a modified KF apparatus (Aquametry II, Barnstead-Thermolyne). Using filter blanks with known quantities of water we found that this extraction procedure was quantitative to the accuracy of the KF instrument. Water content was determined in this fashion for five replicate smoking sessions.

To quantify nicotine, the 16 filter pads for each smoking session were combined and extracted in ethyl acetate and toluene and analyzed by GC-MS according to standard methods (Siegmund et al., 1999). Nicotine was determined in this manner for five replicate smoking sessions. "Tar" or nicotine-free dry particulate matter (NFDPM) was then calculated for the aggregate data by subtracting the average water content and the average nicotine from the average TPM found. Because the TPM and water content were found to be three orders of magnitude greater than the nicotine, the NFDPM was essentially equal to the DPM.

To quantify PAH, the method described by Brunnemann et al. (1994) was adopted with some modifications. The 16 filter pads were combined and extracted using sonication in a solution of 10% dichloromethane in acetonitrile. The resulting solution was concentrated by evaporation, and cleaned by elution with 80:20 hexane dichloromethane mixture through a silica gel column treated with sodium sulphate. The mixture was then evaporated to dryness under nitrogen, and re-dissolved in acetonitrile. The acetonitrile solution was then analyzed by HPLC (Hewlett Packard, Model 1100) coupled to a diode-array UV detector. Chromatographic separation was achieved using a 25 cm × 4.6 mm C18 column, with a solvent program beginning with a 50% acetonitrile-water mixture for 3 min, followed by a 10 min linear ramp to 100% acetonitrile, and ending with an additional 25 min at this condition. PAH were identified by the recorded spectra of the UV detector, and confirmed by standards spiking. PAH were quantified using the standard addition method with a mixture of 13 PAH: anthracene, benzo(a)anthracene, benzo(a)-pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, bibenzo(a,h)antracene, fluoranthene, fluorine, indeno(1,2,3-cd)pyrene, phenanthrene, and pyrene. Quantifications were made in this manner for 10 replicate smoking sessions.

Carbon monoxide was quantified for each of five replicate smoking sessions using a calibrated electrochemical CO analyzer (Monoxor II, Bacharach Inc.) that was connected to the grab sample bag after the smoking session was terminated. A limited number of experiments were made with a non-dispersive infrared CO analyzer (Emission Systems Inc., Model 4001) to validate the measurement. Measured volume concentrations of CO were reported in units of mass by multiplying by the total drawn smoke volume and the density of the CO at ambient temperature and pressure. The initial dead volume between the sampling point and grab bag was negligible to the accuracy of the CO instrument, and was therefore excluded from analysis.

3. Results

3.1. TPM and tobacco consumed

The average TPM for the 32 replicate smoking sessions was 1.38 ± 0.26 g (mean ± standard deviation), while the average tobacco consumed was 4.7 ± 0.4 g. The wide range of tobacco consumed for the 32 replicate sessions probably reflects inherent variability in hand-packing the tobacco mixture in the narghile head, as well as differences in the burning history of the charcoal disk caused by the varying degrees of coal fracture, disintegration, and migration on the head which resulted from its "drumming" at the bubbling frequency.

Fig. 2 shows that TPM and tobacco consumed are linearly correlated. To account for variations across experiments, all chemical determinations were reported per g of TPM for the smoking session in question. The mean quantity of analyte per gram of TPM was then scaled by the mean TPM for the 32 replicate smoking sessions to infer the population-mean quantities for "tar", nicotine, CO, and selected PAH of the "average" smoking session (Fig. 3).

3.2. Moisture

Average water content determinations for five replicate smoking sessions was found to be 0.416 ± 0.019 g/g TPM. The mean TPM for these five smoking sessions was 1.45 ± 0.10 g.

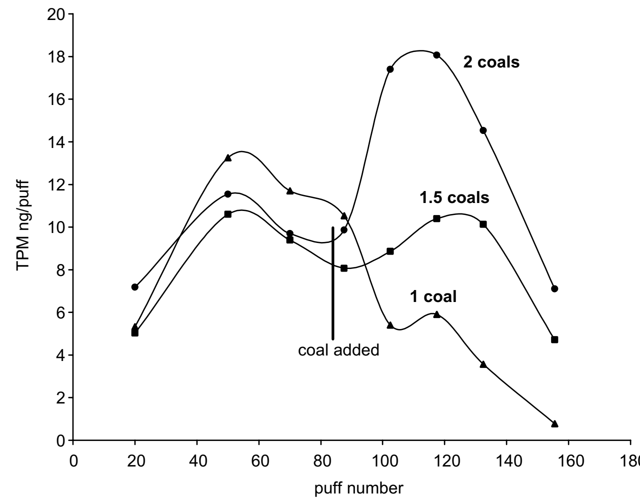

Fig. 2. Evolution of interval-average TPM with puff number for a variety of coal application schedules. All schedules started with a single coal disk; at the 80th puff, and additional half or whole coal was added. The 1.5 coal schedule can be seen to provide relatively uniform TPM production throughout the smoking session and was used in this study.

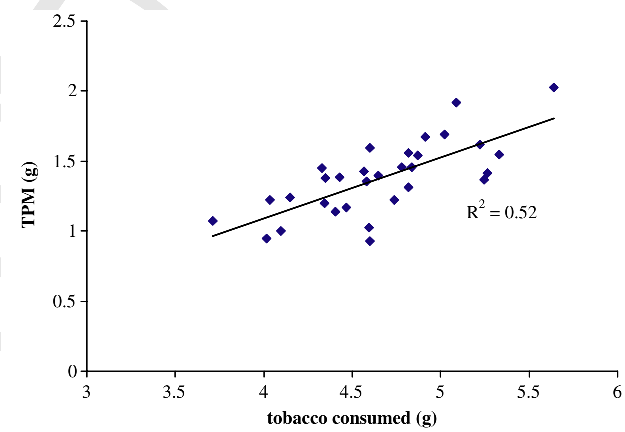

Fig. 3. TPM and tobacco consumed for 32 replicate smoking sessions consisting of 171 puffs of 0.53 l volume, 2.6 s duration, and 17 s interpuff interval.

3.3. Carbon monoxide

Determinations of carbon monoxide for five replicate smoking sessions yielded an average of 105 ± 4 g/g TPM. The mean TPM for these five smoking sessions was 1.36 ± 0.11 g.

3.4. Nicotine and "tar"

The nicotine determinations for five smoking sessions yielded an average of 2.15 ± 0.049 mg/g TPM. The TPM for these five sessions was 1.36 ± 0.21 g. Using this percentage and that previously found for the water content, the average "tar" for the 32 sessions was calculated to be 802 mg.

3.5. *PAH*

It was possible to positively identify chrysene, fluoranthene, anthracene, pyrene, and phenanthrene in the narghile smoke condensates. Of these, only signals corresponding to chrysene, fluoranthene, and phenanthrene were well-resolved and quantifiable. These compounds exhibited average recoveries of 32%, 64%, and 93%, respectively using the extraction and clean-up method described above. The chromatograms were heavily populated with peaks possibly resulting from the various flavorings of the *mo'assel* tobacco paste. Determinations for PAHs in ten replicate smoking sessions yielded 0.543 ± 0.151 μg/g TPM phenanthrene, 0.160 ± 0.053 μg/g TPM fluoranthene, and 0.081 ± 0.044 μg/g TPM chrysene. The mean TPM for these ten sessions was 1.36 ± 0.22 g.

Table 2
Substances found in argileh smoke for 171-puff smoking session. Arithmetic mean reported for 5 replicate machine smoking sessions (10 smoking session for PAH determinations). Previous results using 100, three-second puffs as well as cigarette smoke data are shown for comparison

	Current study[a]	Shihadeh, 2003[b]	Single cigarette
Tobacco consumed, g	4.7	3.0	
"Tar", mg	802	242	1–27 [c] (11.2) [d]
Nicotine, mg	2.96	2.25	0.1–2 [c] (0.77)[d]
CO, mg	143		1–22[c] (12.6)[d]
PAH			
Phenanthrene, μg	0.748		0.2–0.4[c]
Fluoranthene, μg	0.221		0.009–0.099[e]
Chrysene, μg	0.112		0.004–0.041[e]

[a] Ten grams of tobacco mixture used in arghileh head, 171 2.6-second puffs of 0.53 l volume each, spaced 30 s apart.
[b] Ten grams of tobacco mixture used in arghileh head, 100 three-second puffs of 0.3 l volume each, spaced 30 s apart.
[c] Reported ranges for commercial cigarettes, Jenkins et al., 2000.
[d] Arithmetic mean for 1294 domestic cigarette brands tested by FTC for 1998 (FTC, 2000).
[e] LGC, 2002.

4. Discussion

Using a smoking model based on detailed smoking topography field measurements, new data have been generated on the composition of smoke from a narghile loaded with 10 g of *mo'assel* tobacco mixture, and fueled with 1.5 quick-lighting charcoal disks applied in such a manner as to give realistic aerosol production rates and tobacco burn fractions. As expected, the updated smoking model, which prescribes a more intensive smoking regimen than used in our earlier study, resulted in significantly higher quantities of nicotine and "tar". Further, PAHs and CO, which have not been previously reported for realistically generated narghile smoke aerosols, have been quantified. Limitations of the study include the potential that the coal type and application schedule is not representative of real smoking, and that few PAH compounds could be quantified with confidence.

The results are summarized in Table 2. In comparison to our previous study, the amount of tobacco consumed, the nicotine, and "tar" have increased substantially, affirming the importance of the smoking regimen when investigating the chemistry of tobacco smoke aerosols. While the nicotine produced in a smoking session is of similar magnitude to what would be found in a several cigarettes, the "tar" is one to two orders of magnitude greater, as is the CO. "Tar" is normally taken as an indication of the quantity of carcinogens present in the smoke of a cigarette (e.g. benzo(a)pyrene, BaP, scales linearly with cigarette smoke "tar"). However in the case of the narghile, the much lower tobacco temperatures involved (circa 450 °C versus 900 °C) imply that the "tar" composition should be skewed towards products of simple distillation rather than pyrolysis and combustion. Indeed, based on the figures given in Table 2, phenanthrene per mg "tar" is roughly 30 times greater in cigarette smoke than in narghile smoke, indicating that with respect to pyrosynthesized PAH, cigarette "tar" is more potent. The same may not be true for other carcinogenic compounds, such as tobacco specific nitrosamines, which are already present in the tobacco.

Notwithstanding the lower concentration per mg of tar, the three PAH quantified in the smoke, all 3- or 4-ring compounds, were found in quantities many times that of a single cigarette. Chrysene is a tumor initiator while fluoranthene and pyrene (identified but not quantified) are co-carcinogens (Surgeon General, 1979). The fact that 5-ring PAHs such as the notorious BaP were not detected in this study may be due to masking by co-eluting compounds in the complex narghile smoke matrix, or may indicate that they are present in quantities below detectable limits. Further development of the PAH quantification procedures are needed to firmly resolve this question, though it is generally accepted that BaP is present wherever combustion-originating PAH compounds are found. Furthermore, recent work on PAH formation from catechol pyrolysis has shown that BaP formation kinetics exhibit pseudo-first order Arrhenius parameters very close to those of chrysene (Ledesma et al., 2002), indicating that since chrysene is found in abundance, conditions in the narghile are favorable for the formation of BaP. We would thus caution against concluding that the absence of BaP and other carcinogenic 5-ring PAH in Table 2 means that they are absent from narghile smoke. Chrysene to BaP quantities in cigarette "tar" are typically 2–3:1. In addition, if the PAH are synthesized during the smoking session their presence strongly suggests that the precursor benzene exists in the vapor phase of the smoke as well.

The high CO reported in Table 2 is likely a result of the charcoal combustion. Carbon monoxide is considered a major causative agent in cardiovascular disease among smokers (Hoffmann et al., 1997). It is worth noting that the CO to nicotine ratio of narghile smoke is approximately 50:1, compared to 16:1 for cigarettes. Thus if narghile smokers titrate for nicotine as do some cigarette smokers, they can be exposed to significantly greater CO in the course of seeking nicotine satisfaction. The same is true for the PAHs; chrysene for example yields a 40 ng/mg nicotine ratio compared to 2–3 ng/mg for cigarette smoke. Thus smokers who switch from cigarettes to narghile smoking under the impression that the water filters the smoke may actually expose themselves to higher quantities of PAH and CO.

Taken together the limited data to date already indicate that narghile smoke likely contains an abundance of several of the toxicants that are thought to render cigarette smokers more prone to cancer, heart disease, and addiction.

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