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Comparison of CO, PAH, Nicotine, and Aldehyde Emissions in Waterpipe Tobacco Smoke Generated Using Electrical and Charcoal Heating Methods

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Supporting Information

ABSTRACT: Waterpipe tobacco smoking (WTS) has been characterized as a global epidemic. Waterpipe smoke has been shown to contain and deliver significant doses of many of the toxicants known to cause cancer, respiratory, and cardiovascular diseases in cigarette smokers. It has also been shown that the charcoal used to heat the tobacco contributes most of the polycyclic aromatic hydrocarbons (PAHs) and carbon monoxide (CO) found in the smoke, two major causative agents in smoking-related lung cancer and heart disease, respectively. Possibly as a result of growing awareness of charcoal as a toxicant source, electrical heating elements (EHEs) are being marketed for waterpipe use as reduced harm charcoal substitutes. We measured thermal performance characteristics (tobacco burned, total aerosolized particulate matter) and toxicant emissions in WTS generated using three commercially available waterpipe EHEs and charcoal to examine the hypothesis that EHEs can function similarly to charcoal while presenting a reduced toxicant profile. Toxicants quantified included total particulate matter, nicotine, PAHs, CO, and volatile aldehydes delivered at the mouthpiece when the waterpipe was machine smoked using a standard protocol. We found that while EHEs involved an 80% reduction in total PAH and a 90% reduction in CO emissions, they also resulted in a several-fold increase in the potent respiratory toxicant acrolein. These mixed findings underscore the complexity of toxicant reduction by product manipulation and suggest that marketing EHEs as reduced harm products may be misleading.

■ INTRODUCTION

Waterpipe tobacco smoking (aka shisha, hooka, argileh) has become a global phenomenon.[1] Its popularity is reflected in the volume of online searches of waterpipe products, especially in the United States, where a relative increase of more than 60% was observed between 2004 and 2013.[2] Youth appear to be particularly prolific consumers of waterpipes. For example, in Canada, the peak age group prevalence ranges between 18 and 24.[3] Importantly, a large population of waterpipe smokers perceive it as less harmful than cigarettes.[4,5] This perception of lower harm is not supported by the available evidence; waterpipe tobacco smoke contains high concentrations of the toxicants in cigarette smoke and that are associated with various diseases, including cancer.[6−12] Several of these toxicants, measured using analytical lab studies, have also been found in the urine, breath, and blood of waterpipe smokers.[12,13]

Waterpipe tobacco smoking involves the use of burning charcoal as the heat source. It is placed on top of a tobacco preparation known as ma'ssel, an Arabic word for honey.[14] Ma'ssel is a mixture of tobacco, glycerin, water, and flavorants.[14] It began to be widely marketed in the 1990s[15] and is highly popular among users today. Some users smoke an unflavored tobacco commonly known as jurak, or ajami, which is prepared by partially soaking the tobacco in water. In this use configuration, charcoal is placed directly on the tobacco without any foil separation.[16]

By weight, typically more charcoal is consumed during a smoking session than is tobacco.[17] It has been shown previously that charcoal accounts for approximately 90% of the CO and carcinogenic PAHs emissions in WTS[6] and that levels of plasma nicotine, CO boost, heart rate, and exhaled benzene where significantly lower when waterpipes were smoked in a clinical laboratory using an electric heater in place of the charcoal.[18] In recent years, electrical heating elements (EHEs) have become commercially available, allowing users to smoke without charcoal. EHEs commonly allow the user to select from a range of electrical power inputs. Package labeling and online vendors advertise them as "toxicant free" and "carbon monoxide free". Online customer reviews indicate that while some customers are pleased with

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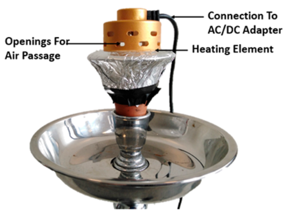
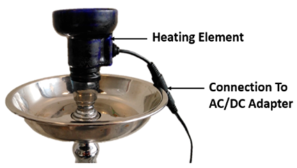
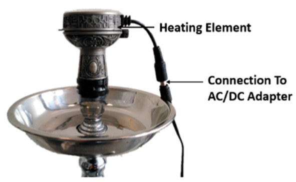
Table 1. Schematic Representation of the EHEs Used in This Study In Addition To Their Material Properties and Operation Range

Electric Heating Element	Tag	Manufacturer	Material	Resistance (Ω)	Power Range (W)
	EHE-1	"Ren Headstream" (China)	Ceramic	3	49.5-79.6
	EHE-2	"Ren Headstream" (China)	Ceramic	2.95	72.4-117.1
	EHE-3	"Hady" (China)	Metal	2.5	37.7-74.4

these products, others complain that not enough smoke is generated or that the tobacco is overheated.[19] Some online reviews indicate the perception that using EHEs results in a lower smoke toxicity compared to conventional charcoal heating.[20]

In theory, if EHEs are found to reduce exposure to harmful constituents in first- or second-hand waterpipe tobacco smoke, regulations could be devised which prohibit the use of charcoal when waterpipes are served (e.g., in waterpipe cafés) and which monitor statements of harm used to advertise and promote tobacco heating sources. In this study, we investigated the thermal performance of three commercially available EHEs and compared emissions of several major toxicants in WTS generated using EHEs to those generated using charcoal. We note that EHEs are not the same as so-called "e-hookah", which, like an electronic cigarette, employs an electrical heater to heat and vaporize a flavored nicotine-containing liquid.

■ EXPERIMENTAL PROCEDURES

This study was conducted in two phases. First, because the EHEs are powered by a variable power supply, the optimal operating power of each EHE needed to be determined. Second, WTS was generated using charcoal and each of the EHEs operating at its optimal power. Each condition was repeated in triplicate, and the WTS for each repeat was separately sampled and analyzed for total particulate matter (TPM), polycyclic aromatic hydrocarbons (PAHs), volatile aldehydes (VAs), nicotine, and carbon monoxide (CO).

EHE Devices. The electric heating elements used are shown in Table 1. EHE-1 is made of ceramic and is placed directly over a traditional waterpipe head in place of charcoal. EHE-2 is a ceramic head with a built-in electrical heater. Once powered, the entire head heats the tobacco from the bottom up, in contrast to charcoal, which heats the tobacco from the top down. EHE-3 is made of metal and is set up similarly to a traditional waterpipe. The electric heating element is located in a hinged cap that is placed on top of the head, directly above the tobacco.

Optimal Power Determination. Each of the EHEs allows the user to select from a range of electrical power output. For an apples–apples comparison, it was necessary to determine the "optimal" power at which each EHE would most closely mimic the performance of the charcoal. The selected performance metrics were the mass of tobacco consumed during a smoking session and the TPM emitted per unit tobacco consumed (or the "yield ratio"). The first metric indicates the total thermal energy delivered to the tobacco, while the second metric is a measure of the relative importance of thermal conduction heating versus convective heat transfer by heated air. The former mode of heat transfer emphasizes vaporization of the tobacco preparation through the top of the waterpipe head and into the environment between puffs, while the latter mode emphasizes vaporization during puffing. Vaporization during puffing results in a greater proportion of the vaporized components being carried into the puff bolus that reaches the mouthpiece. For example, a heating device that continuously heats the tobacco may result in a similar tobacco consumed outcome as charcoal while producing very little aerosol at the mouthpiece. A more detailed description of heat transfer phenomena in the waterpipe head can be found in Monzer et al.[6]

For each EHE, ten equally spaced power settings were investigated, spanning the minimum to the maximum power for each device, and the tobacco burned and TPM emitted were measured. For each EHE, the power setting resulting in the yield ratio closest to that of the charcoal condition was defined as the optimum.

Machine Smoking and Toxicant Sampling Protocol. The waterpipe was smoked using a digitally controlled automatic smoking machine operating under the reduced Beirut smoking protocol: 105 puffs, 530 mL puff volume, 17 s interpuff interval, and 2.6 s puff duration.[21,22] For each smoking session (charcoal or EHE), the bowl was filled with 850 mL of water, and an infiltration test was performed on the leather hose to ensure that the air infiltration was within the

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Table 2. Thermal Performance, CO, Nicotine, PAH, and VA Yields [Mean (Standard Deviation)] for $N = 3$ Smoking Sessions According to the Reduced Beirut Smoking Protocol[a]

	charcoal	EHE-1	EHE-2	EHE-3
optimal power, W		79.6	77.3	50.2
thermal performance				
tobacco consumed, mg	4034.8 (260.9)	4149.3 (48.0)	4911.5 (473.5)	3848.6 (46.7)
TPM, mg	969.8 (169.2)	655.8 (83.2)	1080.6 (154.1)	850.1 (49.4)
yield ratio	0.24 (0.031)	0.16 (0.018)[b]	0.22(0.013)	0.22 (0.015)
toxicant yields per session				
nicotine, mg	5.4 (0.32)	4.43 (0.34)[b]	5.02 (0.64)	4.18 (0.26)[b]
carbon monoxide, mg	86.2 (4.6)	6.2 (0.7)[b]	4.7 (0.6)[b]	6.3 (0.4)[b]
PAHs, ng				
naphthalene[e]	98.5 (100.7)	14.2 (3.9)	18.8 (24.5)	46.7 (72.4)
acenaphthylene	ND	14.9 (12.9)	ND	13.3 (11.6)
acenaphthene	40.6 (30.5)	27.4 (9.2)	25.7 (8.1)	59.8 (7.0)
fluorene	79.3 (71.5)	10.5 (9.1)	NQ	6.0 (10.4)
phenanthrene	876.5 (328.5)	169.7 (30.0)	113.2 (34.7)	149.1 (28.3)
anthracene	148.6 (43.1)	24.0 (6.9)[b]	7.7 (8.2)[b]	48.7 (40.8)[b]
fluoranthene	707.5 (101.6)	140.4 (33.7)[b]	92.4 (24.5)[b]	94.7 (3.8)[b]
pyrene	611.2 (82.8)	93.4 (18.3)[b]	71.7 (26.5)[b]	73.10 (6.8)[b]
benzo[a]anthracene[e]	88.7 (25.3)	ND[b]	ND[b]	ND[b]
chrysene[e]	157.6 (26.3)	16.7 (2.1)[b]	49.7 (30.1)[b]	9.9 (8.6)[b]
benzo[k]fluoranthene[e]	47.3 (10.9)	ND[b]	ND[b]	ND[b]
benzo[b]fluoranthene[e]	ND	ND	ND	ND
benzo[a]pyrene[c]	98.8 (14.7)	18.9 (32.7)[b]	46.2 (17.2)[b]	37.8 (16.6)[b]
benzo[g,h,i]perylene	ND	ND	ND	ND
dibenz[a,h]anthracene[d]	ND	ND	ND	ND
indeno[1,2,3-cd]pyrene[e]	ND	ND	ND	ND
total PAHs	2954.5 (565.0)	530.1 (67.6)[b]	425.4 (108.5)[b]	539.0 (28.8)[b]
volatile aldehydes, μg				
formaldehyde[c]	12.2 (3.0)	20.0 (1.5)[b]	18.7 (4.3)	25.0 (7.1)
acetaldehyde[e]	579.8 (114.6)	1015.0 (71.5)[b]	761.6 (26.9)	1101.0 (79)[b]
acetone	429.3 (42.4)	385.0 (7.6)	376.8 (9.2)	494.0 (33.4)
acrolein	ND	14.1 (1.2)[b]	16.6 (1.3)[b]	14.4 (0.8)[b]
propionaldehyde	66.5 (17.6)	67.9 (3.7)	41.1 (3.1)	86.1 (17.9)
crotonaldehyde	73.7 (10.8)	ND[b]	ND[b]	ND[b]
methacrolein	89.8 (5.3)	88.6 (1.1)	92.3 (0.9)	98.6 (2.1)
butyraldehyde	68.8 (16.9)	67.5 (4.3)	63.7 (1.4)	94.1 (7.5)
benzaldehyde	ND	ND	ND	ND
valeraldehyde	4.3 (7.4)	ND	19.7 (4.8)[b]	76.8 (72.5)
glyoxal	8.7 (0.4)	10.2 (3.1)	8.7 (1.0)	11.0 (1.9)
methylglyoxal	157.1 (45.3)	187.4 (111.7)	277.1 (98.7)	336.1 (94.9)
total VAs	1490.3 (177.8)	1855.7 (177.9)	1676.3 (113.7)	2337 (183.4)[b]

[a]ND: Below detection limits. NQ: Below quantifiable limits. [b]Significant statistical difference compared to charcoal ($p < 0.05$). [c]Carcinogenic to humans according to IARC list of classifications, volumes 1−121 (2018). [d]Probably carcinogenic to humans according to IARC list of classifications, volumes 1−121 (2018). [e]Possibly carcinogenic to humans according to IARC list of classifications, volumes 1−121 (2018).

accepted tolerance of 1.5−1.7 L/min at a mouthpiece flow rate of 12.2 L/min; see Saleh and Shihadeh[23] for details. Then, 10 g of flavored tobacco (Two Apples, Nakhle brand) was placed in the head and covered by aluminum foil perforated according to a predefined 18-hole pattern.[17] The head was then weighed and placed on the waterpipe body; electrical insulation tape was used to seal all joints in the waterpipe to avoid uncontrolled air leakage. For the charcoal condition, one 33 mm diameter quick lighting charcoal briquette (Three Kings Charcoal Co., Holland) was used in each session. The charcoal was lit and held in metal tongs for 1 min prior to placing it on the waterpipe head. For smoking sessions using electrical heating, the adjustable power supply was set to the optimal voltage for each EHE.

During each puff, the smoke exiting the mouthpiece was divided into two parallel flow streams, each passing through a 47 mm glass fiber filter (Gelman type A/E) where the particle phase of the smoke was trapped. Filters were replaced periodically during the smoking session to avoid overload.[21] The filters were weighed before and after sampling for TPM quantification and were stored in airtight containers in the dark at 3 °C until extraction for nicotine and PAH analyses. Sixteen PAH compounds (naphtalene, acenaphtylene, acenaphtene, fluorene, phenanthrene, pyrene, benzo[a]anthracene, chrysene benzo[k]fluoranthene, benzo[b]fluoranthene, benzo[a]-pyrene, benzo[g,h,i]perylene, dibenz[a,h]anthracene, and indeno-[1,2,3-cd]pyrene) were quantified using the method described by Jawad et al.[24] with an instrumental limit of detection (LOD) ranging from 1.35 to 1.5 ng and a limit of quantification (LOQ) ranging from 6 to 7.5 ng depending on the specific PAH. Nicotine concentration was determined by GC−MS following standard procedures presented in Talih et al.[25]

Downstream of the particulate filters, a portion of the flow was diverted through a DNPH-coated silica cartridge to sample and derivatize VA species for offline analysis[26] and into an inert sampling

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bag for offline CO quantification by electrochemical sensor (Bacharach Monoxor III).

VA emissions in the gas phase of the smoke were trapped on DNPH-coated H30 cartridges (Lp-DNPH, Supelco). After collection, these cartridges were eluted with 10 mL of ethanol/acetonitrile (90/10 ratio)[27] and delivered into amber vials. All samples were analyzed by high-performance liquid chromatography−mass spectrometry (HPLC−MS; LC/MSD Trap XCT, Agilent Technologies, Santa Clara, CA, United States) equipped with a photodiode array detector at $k = 360$ nm at a flow rate of 1 mL/min. The analytes were detected based on their retention times compared to calibration standards. Gradient elution on a reverse phase C-18 column (25 cm, 4.6 mm, 5 μm) was performed. The solvents used were (A) water/ACN/THF (6/3/1 v/v/v), (B) water/ACN (2/3 v/v), and (C) ACN. The elution profile varied linearly in time from pure A at $t = 0$ min to 25/75 A/B at $t = 20$ min and finally to pure C at $t = 35$ min. The instrumental LOD ranged from 0.001 to 0.006 μg, and the LOQ ranged from 0.005 to 0.019 μg depending on the specific VA.[28]

Statistical Analysis. Outcome variables, including thermal performance parameters and toxicants, were summarized as mean (standard deviation). Dependent (outcome) variables were compared based on heating source using one-way analysis of variance including posthoc pairwise comparisons (Bonferroni). $p < 0$ 0.05 was used to indicate statistical significance. SPSS version 24.0 (IBM, Armonk, NY) was used to perform the statistical analyses.

■ RESULTS

The thermal performance, CO, nicotine PAHs, and VAs yields of the three EHEs and charcoal are summarized in Table 2. The optimal power for the EHEs was found to range between 50 and 80 W. Yield ratio for EHE-2 and EHE-3 were not significantly different from charcoal when operated at powers of 77.3 and 50.2 W, respectively. On the other hand, at all powers tested, EHE-1 was incapable of reaching the yield ratio of charcoal. For EHE-1, the minimum difference with respect to charcoal was at the maximum power setting.

EHEs resulted in approximately a 90% reduction in CO and an 80% reduction in total PAH emissions relative to the charcoal condition. Nicotine yields were approximately 20% lower for EHE-1 and EHE-3, while EHE-2 was not significantly different than the charcoal condition.

Benzo[a]pyrene and total PAH yields were significantly lower for all three EHEs compared to charcoal. Benzo[a]-pyrene is listed as a Class 1 carcinogen by the International Agency for Research on Cancer (IARC 2018).

Acrolein, a highly reactive irritant thought to be responsible for nearly all the noncancer respiratory disease risk associated with cigarette smoking,[29] was orders of magnitude greater for all EHEs relative to charcoal (see Supplementary Figure S1). Furthermore, none of the 12 quantified VA species was significantly greater for charcoal than for any of the EHEs; whenever a significant difference occurred, EHE emissions of VAs were always greater. Total VAs were not significantly different for EHE-1 and EHE-2 relative to charcoal, while a significant increase of 57% in total VA yield was observed for EHE-3. While total VA for EHE-1 and -2 was not significantly different than charcoal, it is possible that a greater number of samples would have resulted in significant differences. Indeed, an independent 2-factor analysis comparing EHEs to charcoal showed that total VAs were significantly higher ($p < 0.05$).

Results from the posthoc analysis showed no significant difference between PAHs and nicotine yields among three EHEs, while VAs and CO yields were significantly different between EHE-2 and EHE-3.

■ DISCUSSION

This study aimed at assessing the thermal performance of three commercially available waterpipe electric heating elements and quantifying their toxicant emissions relative to those with conventional charcoal. Limitations of this study include the use of a single flavor and brand of tobacco (i.e., there was no variation in the concentration of components present in the tobacco) and a single type of charcoal. Another limitation is that EHE performance was evaluated strictly by analytical laboratory criteria rather than user experience.

Two of the three tested electric heating elements (EHE-2 and EHE-3) were capable of attaining thermal performance characteristics similar to those of charcoal. Their yield ratios of 0.22 were the same as those reported by Monzer et al.[6] for an experimental heating element that was carefully designed to mimic spatial and temporal heating characteristics of charcoal. For all EHEs, the average nicotine yield was similar to that of charcoal. Thus, in terms of amount of tobacco burned, the density of the generated aerosol, and the nicotine yield, commercially available EHEs appear capable of providing charcoal-like performance.

Author: Also consistent with Monzer et al.[6] and Brinkman et al.,[18] replacing charcoal with an EHE greatly reduced CO and PAH emissions in the mainstream aerosol. On the other hand, EHEs resulted in an increase in VA emissions, including acrolein, the primary causative agent in noncancer respiratory disease in cigarette smokers. The elevated VA emissions from EHEs may be intrinsic to the constant power output of these devices. That is, during and between puffs, the electric power is constantly on. As a result, conduction heat transfer between the EHE and the tobacco may result in greater heating between puffs and higher tobacco temperatures in the direct vicinity of the heating surface, where charring occurs.[6] These higher temperatures would, in turn, result in greater thermal degradation of the tobacco and vegetable glycerin (VG) making up the ma'ssel and greater production of VA species. Thermal degradation of propylene glycol and VG by electrical heating has been widely reported in studies of electronic cigarettes.[30−32] In contrast, charcoal combustion is a function of ventilation rate; whenever a puff is executed, the charcoal visibly glows red, and a bolus of hot air and combustion products is drawn through the tobacco to generate the aerosol. Heating between puffs is relatively modulated by the slower combustion rate. Another hypothesis for the lower VA yields when waterpipes were smoked using charcoal is that constituents (e.g., free radicals) produced by the burning charcoal may have reacted with the VAs and resulted in their destruction.

In conclusion, EHEs can result in similar quantities of aerosolized particulate matter and nicotine as charcoal. The combustion-related toxicants CO and PAH are greatly reduced when EHEs substitute charcoal. On the other hand, this electrical heating modality can greatly increase emissions of acrolein, a major causative agent in noncancer respiratory disease. These mixed findings underscore the complexity of toxicant reduction by tobacco product design manipulation and suggest that marketing EHEs as reduced harm products may be misleading.

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■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.chemrestox.9b00045.

Representative HPLC chromatograms of VA-DNP hydrazones derivatized from DNPH and VA in the smoke aerosol of waterpipes smoked using the three EHEs and charcoal (PDF)

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Notes

The authors declare no competing financial interest.

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■ ABBREVIATIONS

WTS, waterpipe tobacco smoking; PAHs, polycyclic aromatic hydrocarbons; CO, carbon monoxide; EHEs, electrical heating elements; TPM, total particulate matter; VAs, volatile aldehydes; VG, vegetable glycerin.

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