Effect of electric heating and ice added to the bowl on mainstream waterpipe semi-volatile furan and other toxicant yields – Supplementary Methods Section

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SUPPLEMENTARY METHODS

Menthol, Nicotine, Cotinine, PAHs, TSNAs

Seven semi-volatile organic compounds (SVOCs) were quantified in the mainstream TPM generated in each session: nicotine, cotinine, menthol, pyrene, benzo(a)pyrene, and the TSNAs, N-nitrosonornicotine (NNN) and 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanone (NNK). A filter was extracted with dichloromethane:acetonitrile (1:1) using sonication followed by an orbital shaker (150 rpm). The extract was split, internal standards were added, and one aliquot was solvent exchanged into 100 mM ammonium acetate and analyzed for NNN and NNK using liquid chromatography-tandem mass spectrometry (LC-MS/MS). The other aliquot was diluted and analyzed using gas chromatography/mass spectrometry (GC/MS) for the remaining SVOCs.

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Semi-Volatile Furans

Nine semi-volatile furans were quantified in the mainstream TPM/session: 5-(hydroxymethyl)-2-furaldehyde, 3-furan methanol, furfuryl alcohol, 2-furoic acid, 2-furaldehyde, 3-furaldehyde, 2-furyl methyl ketone, 5-methyl-2-furaldehyde, and methyl-2-furoate (see Table 2 for chemical abbreviations). One filter/session was extracted using a reverse-phase high performance liquid chromatography with diode array detection method developed for mainstream waterpipe tobacco smoke. Prior to analyzing the machine smoking samples for each waterpipe configuration, blank and TPM-loaded filters were fortified with known levels of the target furans and subjected to the method. Recoveries ranged from 99-106% for all target furans.

CO

Mainstream smoke from each puff of each session was diverted into a small ($0.413 m^3$) stainless steel (comprising 89% of the inner surface area) and glass (comprising 11% of the inner surface area) chamber. CO was quantified continuously in the chamber (0.1 Hz) using a CO analyzer (Model 48C, Thermal Environmental Instruments, Massachusetts, USA), that was calibrated with dynamic gas dilution system (Series 6100, Environics, Connecticut, USA). Total mass of mainstream CO was calculated from the area under the curve, the residual CO in the chamber at the end of each smoking session, and the ventilation rate of the chamber.

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SUPPLEMENTARY TABLES AND FIGURES

Table S-1. Two-stage puffing regimen derived from human smoking of a Research-Grade Waterpipe.[a,b]

Stage	Time (min)	Number of Puffs	Puff Duration (s)	Puff Volume (L)	Interpuff Interval (s)	Total Puff Duration (s)	Total Puff Volume (L)	Total Interpuff Intervall (s)
1	11.2	32	4.6	0.720	16.4	147.2	23.0	524.8
2	22.6	42	3.6	0.455	28.7	151.2	19.1	1,176.7
Total	**33.3**	**74**	**--**	**--**	**--**	**298.4**	**42.1**	**1,701.5**

Table S-2. Target chemicals.

Chemical Name	Abbreviation	CAS Number
Nicotine	Nicotine	54-11-5
Carbon Monoxide	CO	630-08-0
Menthol	Menthol	2216-51-5
Cotinine	Cotinine	486-56-6
Benzo(a)pyrene	BaP	50-32-8
Pyrene	Pyrene	129-00-0
N′-nitrosonornicotine	NNN	16543-55-8
4-(methylnitrosamino)-1-(3-pyridyl)-1-butanone	NNK	64091-91-4
5-(Hydroxymethyl)-2-furaldehyde)	HMF	67-47-0
3-Furanmethanol	3-FM	4412-91-3
Furfuryl Alcohol	FFA	98-00-0
2-Furoic Acid	2-FA	88-14-2
2-Furaldehyde	2F	98-01-1
3-Furaldehyde	3F	498-60-2
2-Furyl methyl ketone	2-FMK	1192-62-7
5-Methyl-2-furaldehyde	5-M-2-F	620-02-0
Methyl 2-furoate	MF	611-13-2

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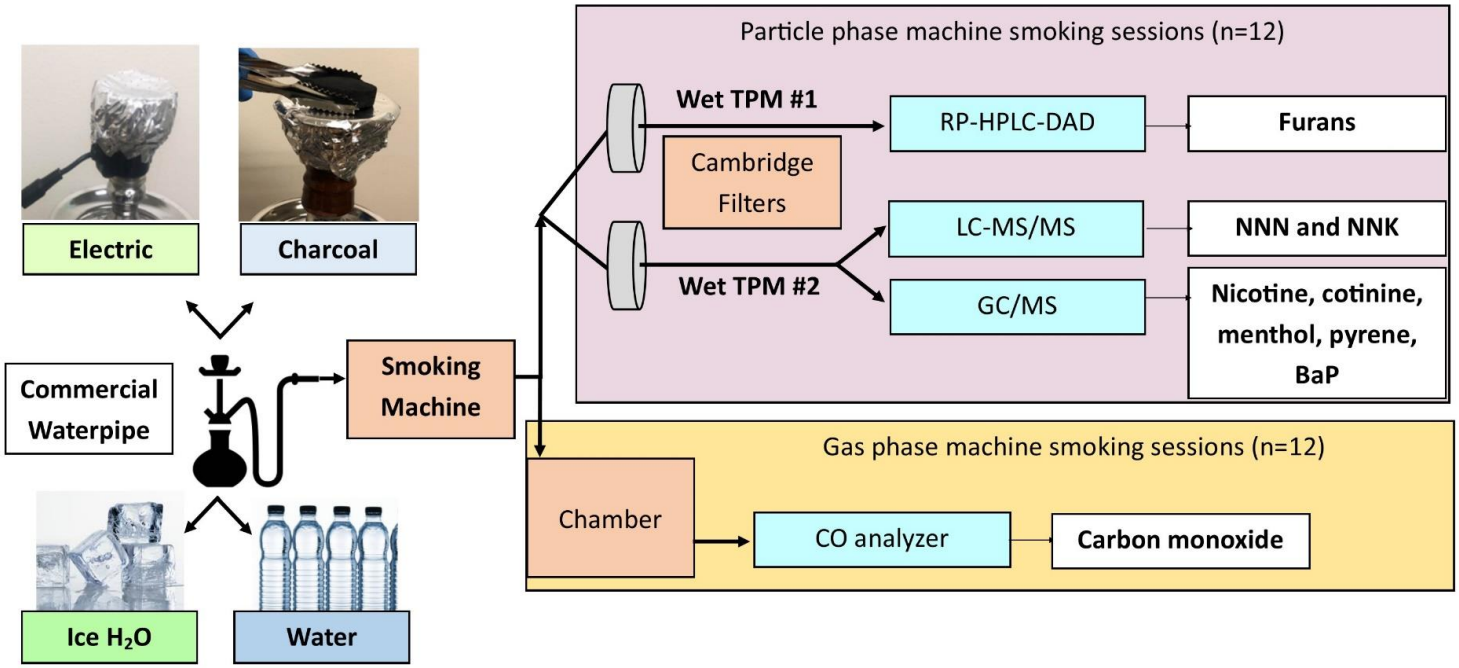

Figure S-1. Schematic describing mainstream waterpipe tobacco smoke sample generation, collection, and analysis.

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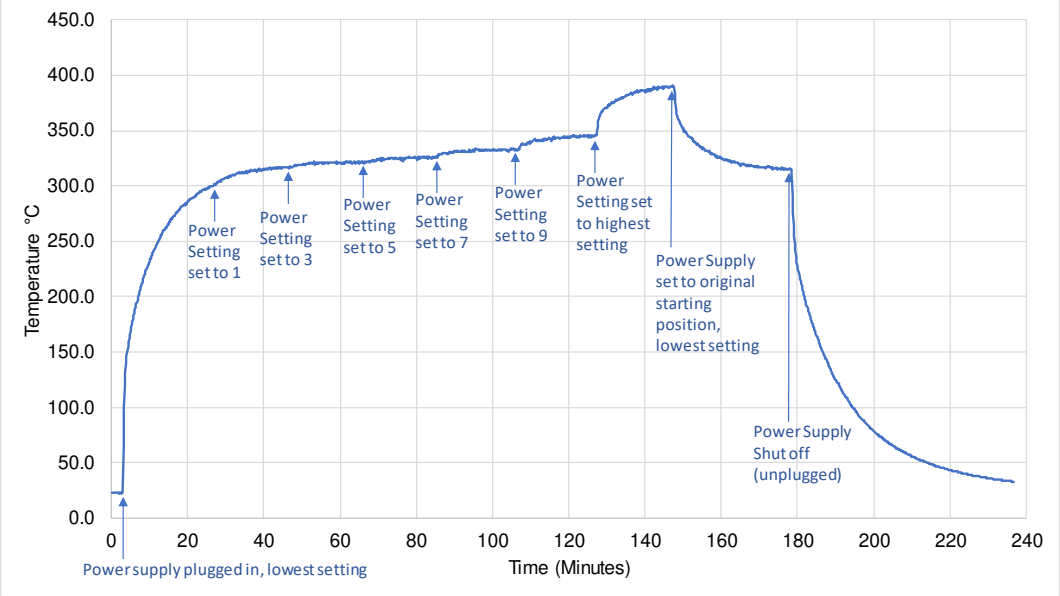

Figure S-2. Temperature of waterpipe electric head as a function of power supply setting. Power was selected using a thumbwheel that was numbered 1 – 9. Lowest setting was < 1, and highest setting was >9.

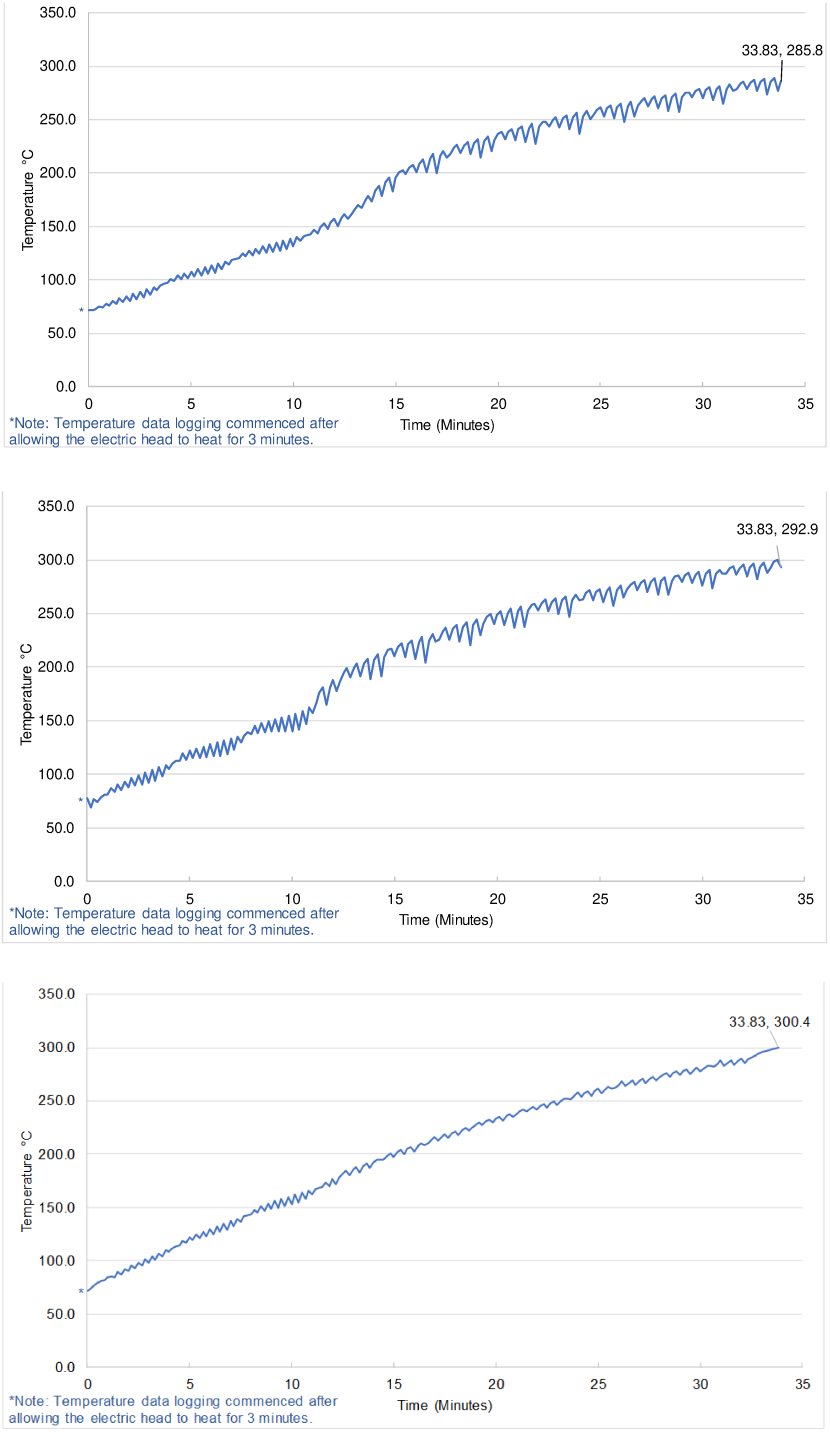

Figure S-3. Average temperature of waterpipe tobacco during machine smoking with electric heating (n=1 session per setting) and power supply at the following settings: lowest (< 1) [top], "3" [middle], "5" [bottom].

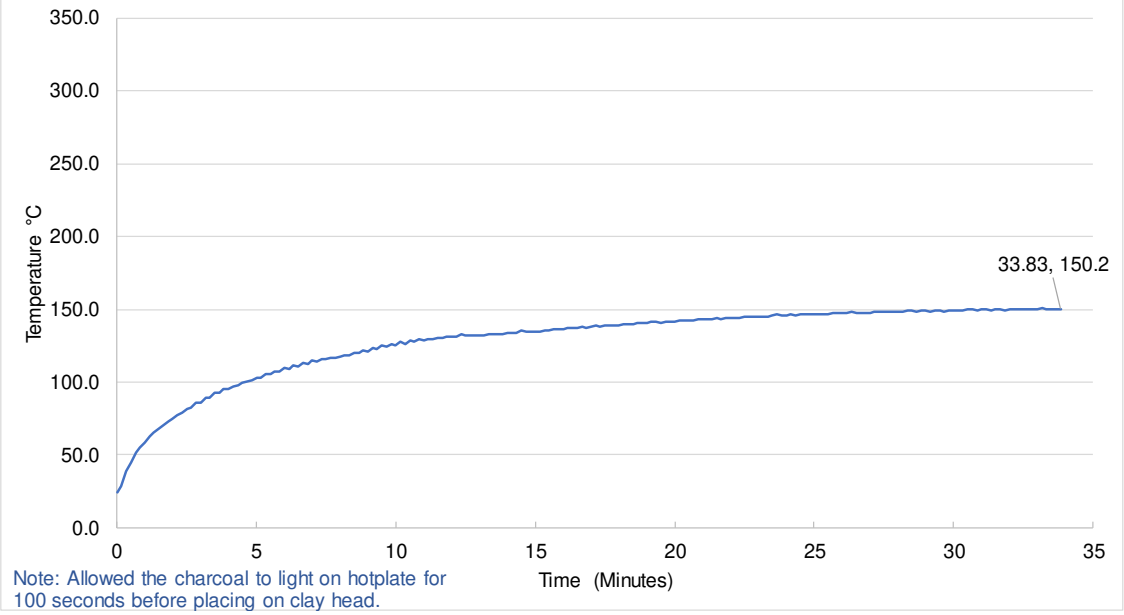

Figure S-4. Average temperature of waterpipe tobacco during machine smoking (n=3 replicate sessions) with charcoal heating.

https://godfreyphillipsinternational.com/shisha/shisha-tobacco/

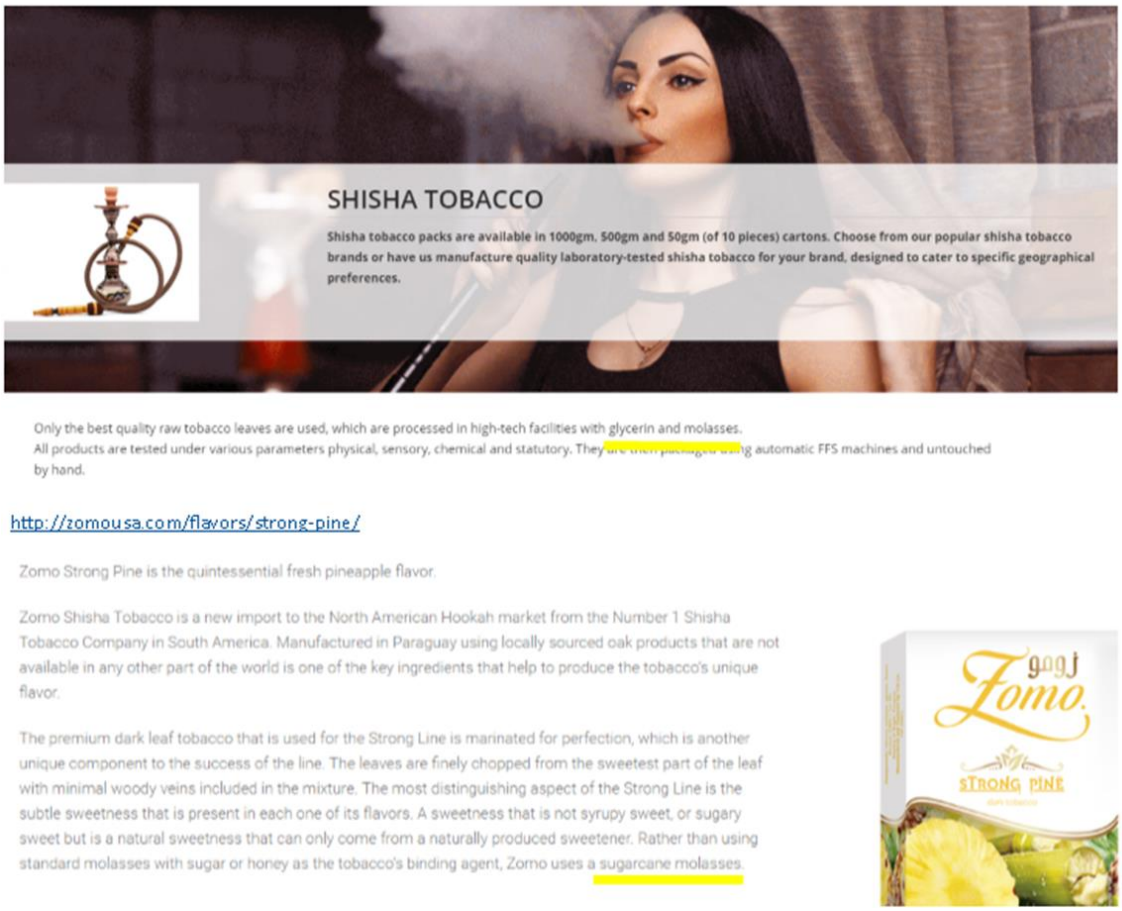

Figure S-5. Examples of two manufacturers' websites that describe the practice of adding glycerin and molasses to the tobacco; yellow underlines added for emphasis.

SUPPLEMENTARY REFERENCES

(a) Brinkman MC, Kim H, Gordon SM, et al. Design and validation of a research-grade waterpipe equipped with puff topography analyzer. *Nicotine Tob Res* 2016;18:785–93.
(b) Brinkman MC, Kim H, Buehler SS, Adetona AM, Gordon SM, Clark PI. Evidence of compensation among waterpipe smokers using harm reduction components. *Tob Control* 2018 Oct 30:tobaccocontrol-2018.