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Flavours and flavourings in waterpipe products: a comparison between tobacco, herbal molasses and steam stones

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► Additional supplemental material is published online only. To view, please visit the journal online (<http://dx.doi.org/10.1136/tobaccocontrol-2021-056955>).

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Received 30 July 2021

Accepted 17 February 2022

Published Online First

3 March 2022

Objectives Flavoured products are especially appealing to youth and contribute to the onset of waterpipe smoking and continued use of waterpipe tobacco. The goal of database and chemical analysis was to provide a clear overview of commonly used flavours and flavourings in tobacco and related waterpipe products, that is, herbal molasses and steam stones.

Methods In 2019, 249 waterpipe tobacco products were registered in the European Common Entry Gate by manufacturers to be marketed in The Netherlands. Flavour categories were assigned to the registered products based on their brand names and product descriptions. Nicotine and eleven 1111 flavourings were identified and quantified in waterpipe tobacco (n=8), herbal molasses (n=7) and steam stones (n=4) by extraction and gas chromatography–mass spectrometry (GC–MS) analysis.

Results Flavour categories could be assigned to 237 of 249 registered waterpipe tobacco products. Eight flavour main categories and 48 unique subcategories were identified and presented in a flavour wheel. All registered waterpipe tobacco products were flavoured, and the majority (78%) was fruit flavoured. Herbal molasses contained similar median flavouring levels, and steam stones contained lower median levels compared with waterpipe tobacco. Flavourings in waterpipe products were almost exclusively fruity and sweet, often in combination with menthol/mint flavourings.

Conclusions This study is the first to present a waterpipe tobacco flavour wheel, providing a quick overview of waterpipe tobacco flavours and thereby aiding communication among experts around the globe. GC–MS analysis revealed that the most prevalent flavourings are present in similar levels in herbal and tobacco waterpipe products. Banning flavourings in all waterpipe products would be a good strategy to reduce waterpipe smoking among youth.

INTRODUCTION

Waterpipe, also known as 'shisha' or 'hookah', smoking is most prevalent in the Middle East and has increased in popularity in Europe and the USA.^{1–3} Introduction of flavoured waterpipe tobacco strongly contributes to the increased popularity of waterpipe smoking, especially among youth.⁴ A misconception exists that waterpipe smoking is healthier than cigarette smoking due to water filtration of the smoke before inhalation. Also, the sweetened taste often wrongly leads to the assumption of using a safer product.^{5 6} Little research has been done on alternative waterpipe

products such as steam stones or herbal molasses. Steam stones are widely marketed waterpipe products and are often presented as less harmful than waterpipe tobacco.⁷ Steam stones are small porous mineral rocks soaked in a liquid, usually glycerol based, containing flavourings. They are available with and without added nicotine.⁸ Herbal molasses are flavoured plant-based products without tobacco and are also marketed as less harmful than waterpipe tobacco.⁹ However, earlier research on toxicants such as nicotine, carbon monoxide, polycyclic aromatic hydrocarbons and aldehydes has shown that herbal waterpipe products contain levels of all these toxicants similar to those in waterpipe tobacco, except for nicotine.¹⁰

Waterpipe products, with or without tobacco, are almost exclusively flavoured with predominantly fruity or sweet flavours. The attractiveness of flavours is a major reason to start and continue using waterpipe smoking.¹¹ Sweet flavours are especially appealing to youth and contribute to the popularity of waterpipe smoking among youth.^{4 6 12–16} Not only do flavours contribute to attractiveness, the flavouring ingredients, from now on referred to as 'flavourings', can be hazardous too.^{17–19} Flavoured waterpipe tobacco caused higher toxicity than unflavoured waterpipe tobacco in mice, when looking at different inflammatory and oxidative stress biomarkers.¹⁷ Also, flavourings found in waterpipe tobacco such as limonene, linalool and eugenol are known as being allergenic in human skin.¹⁸

Tobacco products, herbal products for smoking and products such as e-cigarettes and e-liquids, are covered by the Tobacco Products Directive 2014/40/EU (TPD).²⁰ Any of these products to be marketed in a EU member state, has to be registered in the European Common Entry Gate (EU-CEG; until 2015 a similar system, 'EMTOC').²¹ The amount of registered waterpipe tobacco products in the EU-CEG database in the Netherlands increased yearly, starting with 1 product in 2010 up to 249 products in 2019. Waterpipe products such as steam stones are not covered by the TPD and are therefore not registered in the EU-CEG database.

A ban on characterising flavours, other than menthol, had already been introduced for cigarettes and roll-your-own tobacco in the USA in 2009 and in the European Union (EU) in 2014.^{12 20 22} From 2020 on, menthol was included in this ban in EU and UK and this year (2021), the US Food and Drug Administration (FDA) proposed a ban on menthol



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To cite: Bakker-'t Hart IME, Bakker F, Pennings JLA, et al. *Tob Control* 2023;**32**:627–634.

as characterising flavour in combustible tobacco.²⁰ Recently, a proposal was made to ban all characterising flavours other than tobacco in e-cigarettes and refills in the Netherlands, in order to discourage young people from vaping/smoking other products and still provide a less harmful alternative for people that try to reduce tobacco smoking.²³ Currently, characterising flavours are still allowed in waterpipe products. The TPD (7.12), however, states that products exempted from the characterising flavour ban, such as waterpipe products, may be included '*if there is a substantial change of circumstances as established in a Commission report*'.²⁰

In this study, data on Dutch EU-CEG registered waterpipe products (March 2019) and their ingredients were used as a primary source for information on waterpipe flavours and flavourings. Waterpipe tobacco product names and descriptions were used for flavour categorisation and subsequent design of a flavour wheel. The flavour wheel presented herein will aid communication among experts. We also provide an overview of the percentages of registered flavour main and subcategories.

It is difficult to obtain information on flavour categories for herbal molasses and steam stones, since information on these products is not present in EU-CEG data. Furthermore, compositions of these products, such as flavourings, are not mentioned on product labels. However, chemical analysis on a selection of waterpipe products, including non-tobacco containing products, provides us with some information on the presence of flavourings and nicotine. Waterpipe products were selected in such a way that the most common main flavour and subflavour categories were covered. Eleven flavourings and nicotine were identified and semiquantitatively determined in these products by gas chromatography–mass spectrometry (GC–MS). All collected information in this study on flavours and flavourings in waterpipe products provides a clear overview on flavourings added and actually present and can be used as a starting point for future risk assessments, regulation and policy.

METHODS

Data analysis and assignment of flavour categories to waterpipe tobacco products registered in the Dutch EU-CEG

Data processing and analysis was performed by the MS workstation (V7.0.2, Agilent technologies) and the statistical software program R (V3.6.0) and Excel as described previously.^{24 25} Waterpipe tobacco products in the Dutch EU-CEG database on March 2019 (n=249) were assigned to flavour categories based on their product descriptions and/or brand names and, where needed, additional internet searches. The first mentioned flavour in the brand name or product description was considered a primary flavour and any additional flavours were considered secondary flavours. In total, 48 unique primary flavours were identified, forming the subcategories in the flavour wheel. Subcategories with similar properties (such as different types of fruit) were grouped into eight main categories, similar to the e-liquid flavour and cigar/cigarillo flavour wheels.^{26 27} The category 'other flavours' was included for potentially used flavours (in the future or in other countries) that are not inferred from the registered waterpipe tobacco products in the Dutch EU-CEG database.

Selection of flavourings for chemical identification and semiquantification in waterpipe products

A total of 378 unique ingredients (based on name and/or CAS number) was identified in 249 waterpipe tobacco products. Ingredients were sorted based on prevalence in number of products. The 10 most frequently added flavourings were selected for chemical analysis. The following frequently added ingredients were not

considered flavourings or considered unsuitable for the chemical analytical method, since they are composed of multiple chemical substances: solvents/carriers (n=5; glycerol, 1,2-propylene glycol, water, ethyl alcohol and triacetin), sugars (n=4; high fructose corn syrup, caramel, sucrose and glucose), food preservatives/anticaking agents (n=3; potassium sorbate, citric acid and synthetic amorphous silica), dyes (n=1; FD&C Red) and absent CAS no (n=1; 'unspecified flavourings'). Anethole was not part of the top 10 most frequently added flavourings but added to the target list because of the high quantities registered in EU-CEG and earlier research.²⁸ Eleven flavourings and nicotine were identified and semiquantitatively determined by GC–MS.

Waterpipe products and chemicals

Waterpipe products were selected in such a way that the most prevalent (main and sub) flavour categories, as identified in EU-CEG registered products, were covered. Eight different flavours of waterpipe tobacco (T1–8), seven flavours of herbal molasses (M1–7) and four flavours of (nicotine free) steam stones (S1–4) were purchased from commercial vendors in The Netherlands or the UK. For six (T3–T8) of the eight analysed waterpipe tobacco products, flavouring ingredients and their respective concentrations were specified in EU-CEG. Flavourings were registered as 'various flavours' for the other two waterpipe tobacco products (T1–2). Herbal products for smoking are covered by the TPD, requiring EU-CEG registration. Herbal molasses from this research were not registered in the Dutch EU-CEG. Two products only claimed to contain 'fibre materials', and it is unclear whether they are considered herbal products for smoking and would require registration. The other five products were obtained abroad (UK). Steam stones do not contain tobacco and are currently not covered by the TPD and Dutch tobacco law. Since no EU-CEG data were available on the investigated herbal molasses and steam stones and no ingredients were specified on their packaging materials, no information on flavouring content was available. Nicotine-containing steam stones were only found online after the steam stones of this manuscript were investigated and are therefore not part of this selection.

Ethanol absolute was obtained from Merck (Darmstadt, Germany). Standards of the flavourings were of analytical or food grade purity ($\geq 97\%$). Flavourings were purchased from Sigma-Aldrich (Zwijndrecht, The Netherlands) and ethyl acetate was purchased from Alfa Aesar (Kandel, Germany). Nicotine (purity $>99\%$) and the internal standard benzene-d₆ (purity 100%) were purchased from Acros Organics (Geel, Belgium) and Sigma-Aldrich, respectively.

Standard solutions

For qualification of the flavourings and nicotine, the standards were individually dissolved in ethanol (circa 2 mg/mL). A solution of the internal standard benzene-d₆ was prepared in ethanol (100.0 µg/L). All flavouring standards and nicotine were dissolved as a mixture in the internal standard solution in 10 different concentrations.

Product extraction of flavourings and nicotine

Of each waterpipe product (waterpipe tobacco (n=8), herbal molasses (n=7) and steam stones (n=4)), 3 g ($\pm 10\%$) were weighed in a 15 mL tube (in duplicate); 10 mL of ethanol containing 50 µg/mL of the internal standard was added, and the mixture was shaken on a PTR60 rotator (Grant instruments) for 60 min at room temperature. The steam stones were shaken in the liquid, in which they are dispersed, before extraction. This liquid has not been extracted and analysed separately. An aliquot

(500 µL) was taken out and centrifuged in an Eppendorf tube and passed through a 0.45 µm syringe filter. The filtrate was further diluted (10 and 100 times) with ethanol before GC–MS analysis.

GC–MS conditions and data analysis

An Agilent 7890B GC system coupled with an Agilent 240 ion trap mass spectrometer was used, equipped with a 7693 autosampler and a G4513A injector. GC–MS runs and data analysis were performed in the same manner as described before.²⁴

The 11 selected flavourings and nicotine were quantified (by quantifier ion of analytical standard) in waterpipe samples where the respective flavouring was positively identified (by qualifier ion of analytical standard). Concentrations of the flavourings in the duplicate runs were averaged for further analyses. Results were reported as semiquantitative since no spike recovery experiments were performed. Limits of detection (LODs) were calculated based on the calibration curve as $3 \times \text{SD/slope}$; limits of quantification were set as the lowest and highest point of the calibration curve <10 and >100 µg/mL.

RESULTS

Flavour categorisation of Dutch marketed waterpipe tobacco products

Flavour categories could be assigned for 237 (95%) out of 249 waterpipe tobacco products registered in the EU-CEG database

(March 2019). For the remaining 12 products, only general brand names were registered, with no additional product name or description, making it impossible to assign a flavour category. In total, 8 main flavour categories and 48 subcategories were defined.

Based on the 237 categorised products, a flavour wheel was designed, which is shown in figure 1. The flavour wheel also contains an ‘other flavours’ section for waterpipe tobacco flavours that might arise in the (near) future and cannot be placed in one of the other categories.

A visualisation of the percentage of each flavour category (except for ‘other flavours’, since no products from EU-CEG belong to this category) can be found in figure 2. The main category ‘fruit’ (n=185) covered 78% of the EU-CEG registered and categorised products (n=237).

Common (added to more than 10 registered waterpipe products) subcategorised flavours were: blueberry, apple, strawberry, grape, chewing gum, watermelon, lemon and mint. For 117 products, secondary flavour(s) categories were assigned. Of these products, most were assigned to ‘mint’ (72; 62%), as the only secondary flavour category (n=38) or in combination with other secondary flavours (n=34) (data not shown).

Ingredients of waterpipe tobacco in Dutch EU-CEG

In the EU-CEG data for the 249 waterpipe tobacco products, 378 unique ingredient descriptions were found. Ingredient

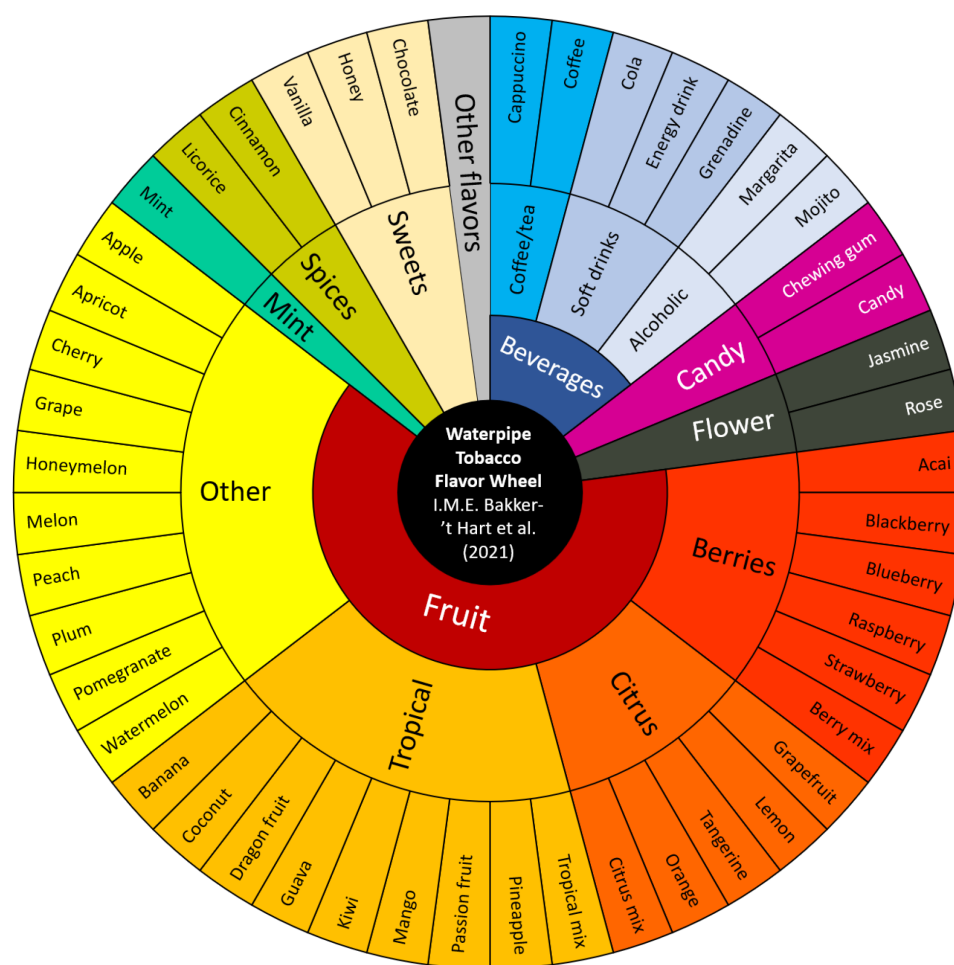


Figure 1 Flavor wheel of waterpipe tobacco products registered in EU-CEG (March 2019, The Netherlands). The inner ring shows the 8 main categories, where beverages and fruit can be further subdivided into another three or four categories, respectively. The outer ring is composed of the 48 identified sub-flavor-categories.

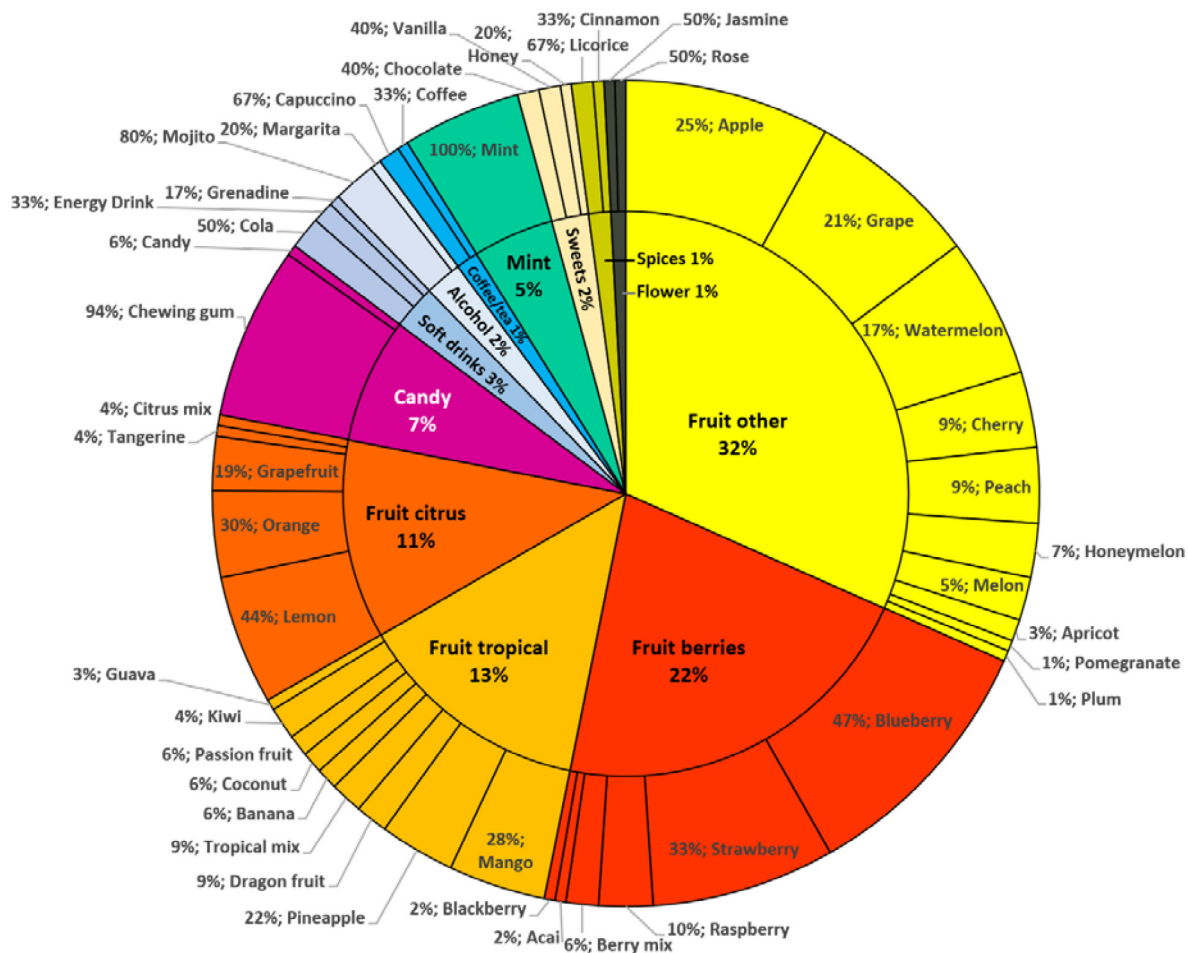


Figure 2 Percentages of the main- and subcategories of flavors based on 237 categorized EU-CEG registered waterpipe tobacco products (March 2019, the Netherlands). Main flavor categories (inner circle) are presented as a percentage of all products and flavor subcategories (outer ring) as a percentage of its main category.

descriptions such as ‘various flavours’ or ‘apple juice’ cover multiple chemical ingredients. The frequency of use of each ingredient varied from 0.4% (1/249) for, for example, pentyl acetate, to 100% (249/249) for glycerol. The 25 most frequently added ingredients are shown in online supplemental table S1. A selection of 10 frequently added flavourings for chemical analysis was acquired after exclusion of non-flavourings and flavourings that are a mixture of chemicals (entries 1–10: [table 1](#)), and anethole was added because of the registered high quantities (entry 11: [table 1](#)). Flavour descriptions, smoke taste and hazard statements (GHS codes) are listed in [table 1](#) for the selected 11 flavourings.

Twenty-five registered flavourings added to waterpipe tobacco in the highest quantity mg/g (including mixtures such as extracts and oils) are shown in online supplemental table S2).

GC–MS analysis for the identification and semiquantification of flavourings and nicotine in waterpipe products

Flavourings were identified and quantified by GC–MS after extraction of waterpipe tobacco, herbal molasses and steam stones.

Semiquantified flavour ingredients and nicotine (mg/g) in all waterpipe tobacco samples (T1–8), herbal molasses (M1–7) and steam stones (S1–4) are displayed in [table 2](#).

Only tobacco products were found to contain nicotine and no nicotine was detected in herbal molasses and steam stones

([table 2](#)). The nicotine levels in waterpipe tobacco ranged between 0.5 and 1.4 mg/g.

On average, herbal molasses contained similar levels (<0.03–7.14 mg/g, median concentration: 0.38 mg/g) of flavourings as waterpipe tobacco (<0.03–8.27 mg/g, median concentration: 0.275 mg/g). Steam stones contained lower levels (<0.03–5.62 mg/g, median concentration: 0.06 mg/g) of flavourings than tobacco and herbal molasses.

DISCUSSION

Flavours and frequently added flavourings of Dutch EU-CEG registered waterpipe tobacco

For 237 of the 249 Dutch EU-CEG registered waterpipe tobacco products, flavour categories could be assigned based on the product name or description. For the remaining 12 non-categorised products, four or more flavourings were registered in EU-CEG, indicating that these products are indeed flavoured. None of the categorised products could be categorised as ‘tobacco’ flavour. It is unclear if plain tobacco is used by waterpipe smokers in the Netherlands. Two studies from the USA indicate tobacco flavour does not seem appealing for (initiating) waterpipe smoking. The first study, the 2013–2014 Population Assessment of Tobacco and Health, showed that the first smoked waterpipe tobacco was flavoured in 89% of the cases.²⁹ The second study, a survey (from 2018), revealed that

Table 1 Ten frequently added flavouring ingredients in waterpipe tobacco registered in EU-CEG (March 2019, the Netherlands) and anethole.

	Flavouring agent	CAS no.	Prevalence (%)	Median concentration (mg/g)	Flavour description	Smoke taste	GHS code
1	Vanillin	121-33-5	59	0.31	Sweet, powerful, creamy, vanilla-like	Sweet vanilla; used widely in tobacco flavours at levels of 25 ppm up to add sweetness	H317, H319
2	Ethyl Vanillin	121-32-4	47	0.02	Intense, sweet, vanilla-like odour; creamy, vanilla taste	Sweet, strong vanilla notes; useful for adding sweetness over a wide range of levels	H302, H315, H319, H335, H412
3	Dihydrocoumarin	119-84-6	41	0.001	Very sweet, nut-like, hay-like, coumarin-tobacco like odour	Nutty, hay, honey, spicy; sweet coumarin-like	H302, H317
4	Ethyl Butyrate	105-54-4	40	1.24	Ethereal, fruity with buttery-pineapple-banana, ripe fruit and juicy notes	Sweet, fruity, winey, excellent in any fruit or wine flavour for tobacco	H226
5	Ethyl Acetate	141-78-6	34	0.49	Ethereal, sharp, wine-brandy-like	Weak fruity, chemical; not important as a tobacco flavour	H225, H319, H336
6	Ethyl-2-methylbutyrate	7452-79-1	31	0.54	Strong, green, fruity, apple with strawberry notes	Green apple, winey-fruity	H226
7	Maltol	118-71-8	27	0.22	Sweet, fruity, berry, strawberry, caramellic	Sweet, flue-cured; tends to fade somewhat on ageing; excellent tobacco flavour; used in caramel, nut, brown sugar maple and flue-cured flavours	H302
8	Isoamyl Acetate	123-92-2	25	1.10	Sweet, fruity, banana, pear odour and taste	Weak, sweet, banana	H226
9	Menthol	2216-51-5	25	0.76	String trigeminal cooling sensation with a slight mint note	No description available	H315, H319
10	Benzyl Alcohol	100-51-6	25	1.26	Faint, sweet, almond fruity, somewhat chemical	Very weak cherry-floral, smoothing; primarily used as a cosolvent for highly water insoluble materials such as sclareolide and anise oils	H302, H332
11	Anethole	4180-23-8	11	2.25	Sweet, herbaceous, anise (artificial Licorice) odour and taste	Sweet, anise	H317

This table contains the name of the flavour ingredient, the frequency of addition in the registered waterpipe tobacco products and median concentration per product. Flavour and smoke taste descriptions were collected from the Leffingwell database.²⁹ Flavour descriptions are based on the individual flavourings. The smoke taste is the taste of a flavouring in smoke observed after combustion of tobacco containing the flavouring. Hazard statements of the Globally Harmonized System of Classification and Labeling of Chemicals (GHS codes) were derived from PubChem. EU-CEG, European Common Entry Gate.

none of the questioned waterpipe smokers used tobacco flavour as the first smoked flavour.³⁰ Other studies showed that waterpipe tobacco with flavour and humectants were preferred over waterpipe tobacco without flavour and/or humectant among waterpipe smokers.^{13 31}

The categorised waterpipe tobacco products could be placed into seven main flavour categories: fruit (78%), candy (7%), beverages (6%), mint (5%), sweets (2%), spice (1%) and flower (1%). As the subcategories in this flavour wheel are fully based on EU-CEG data, they do not by definition represent each waterpipe tobacco flavour available. Therefore, we included an eight main flavour category, 'other flavours'. The high abundance of fruity flavours seems to match the demand and preference for these flavours. A recent focus group study in the UK revealed that certain users started waterpipe smoking because of fruity flavours and the introduction of new flavours.³² Absence of flavours would make it unlikely for a participant to initiate waterpipe smoking.^{32 33} Multiple reasons for waterpipe smokers to prefer fruity flavours are listed by Kowitt *et al*³³ such as: 'fruit flavor makes it like a candy' or 'fruit flavor makes it less harmful'. Ben Taleb *et al*³⁴ found enhanced subjective smoking measures of enjoyment and satisfaction by participants that smoked flavoured tobacco compared with non-flavoured tobacco.

The most prevalent subcategories (present in at least 10 registered waterpipe tobacco products) we found were six fruit flavours (blueberry, apple, strawberry, grape, watermelon and lemon), chewing gum and mint. Ben Taleb *et al* asked participants from the USA for their preferred flavours, which resulted in mixed fruits, apple, mint and grape. These results showed high similarity to the most prevalent flavour categories found in our study.³⁴ Farag *et al* selected different flavoured waterpipe products based on availability and popularity as recommended by suppliers in Egypt for subsequent chemical analysis.²⁸ Their flavour selection included apple, grape, watermelon and strawberry, showing high similarities with the aforementioned subcategories. Flavour preference and (EU-CEG) market data showed high overlap on an international level.

Mint was not frequently used primary flavour in waterpipe tobacco, but it was predominantly found as a secondary flavour. Additional research is required to reveal the prevalence and importance of menthol/mint flavour in waterpipe products.

Fruit flavours were more prevalent in our sample of waterpipe tobacco products (78%), compared with flavours in EU-CEG registered e-liquids (34%),³⁵ whereas other sweet flavours, such as dessert, candy and other sweets, are more prevalent in e-liquids. A US survey from 2018 indeed showed that the most

Table 2 Semiquantification of flavourings and nicotine in waterpipe tobacco samples T1–T8, herbal molasses M1–M7 and steam stones S1–S4 by GC–MS.

Main flavour	Secondary flavour(s)	Anethole	Benzyl alcohol	Dihydro-coumarin	Ethyl acetate	Ethyl butyrate	Ethyl 2-methylbutyrate	Ethyl vanillin	Isoamyl acetate	Maltol	Menthol	Vanillin	Nicotine
Waterpipe tobacco													
T1 Strawberry	Watermelon, mint	–	–	–	0.20 (2.3%)	0.19 (2.8%)	1.33 (1.3%)	–	–	0.26 (1.2%)	8.27 (1.2%)	0.10 (3.6%)	1.32 (2.6%)
T2 Mango	Passionfruit, mint	–	–	–	0.29 (2.6%)	0.09 (0.7%)	–	–	–	0.06 (1.1%)	6.65 (0.3%)	–	1.39 (0.4%)
T3 Grape	–	–	–	–	0.14 (0.9%)	–	0.07 (19%)	<0.03	–	0.42 (0.2%)	–	0.67 (3%)	0.79 (2.9%)
T4 Chewing gum	–	–	–	–	–	–	–	<0.03	–	0.06 (1.4%)	0.69 (0.6%)	–	0.62 (3.1%)
T5 Watermelon	Mint	–	–	–	0.25 (10%)	0.04 (1.4%)	3.59 (0.7%)	1.27 (1.7%)	0.12 (5.1%)	<0.03	0.62 (2.2%)	–	0.80 (2.3%)
T6 Blueberry	Mint	–	–	–	0.30 (8.8%)	0.08 (1.1%)	0.81 (1.1%)	–	–	0.60 (17%)	0.72 (0.6%)	0.73 (1.9%)	0.57 (3.0%)
T7 Mint	–	–	–	–	0.10 (73%)	–	–	–	–	0.06 (4.9%)	1.46 (2.5%)	–	0.53 (5.1%)
T8 Apple	7.44 (1.8%)	1.12 (1.5%)	–	–	0.06 (1.3%)	–	0.71 (0.2%)	0.69 (1.3%)	–	<0.03	2.19 (0.1%)	0.26 (2.2%)	0.53 (4.1%)
Herbal molasse													
M1 Apple	7.14 (18.4%)	–	–	–	–	–	–	–	–	–	–	–	–
M2 Mint	–	–	–	–	–	–	–	–	–	–	5.99 (18%)	–	–
M3 Apple	5.61 (7.5%)	1.07 (0.5%)	–	–	0.07 (417%)	0.18 (9.2%)	0.23 (7.9%)	0.40 (8.7%)	<0.03	–	0.27 (4.2%)	0.38 (5.7%)	–
M4 Blueberry	–	0.38 (1.8%)	–	–	2.02 (7.2%)	1.01 (5.5%)	0.76 (8.1%)	0.09 (8.3%)	–	0.90 (0%)	0.63 (4.4%)	0.99 (5.8%)	–
M5 Grape	–	–	–	–	0.27 (13%)	0.97 (2.8%)	0.16 (1.6%)	0.19 (5.3%)	–	0.60 (2.5%)	–	0.82 (2.3%)	–
M6 Strawberry	–	–	–	–	–	0.56 (0.8%)	1.14 (1.4%)	0.04 (0.4%)	<0.03	–	–	–	–
M7 Mixed Fruits	0.77 (8.7%)	1.97 (3.4%)	–	–	0.04 (20%)	0.37 (9.9%)	–	0.05 (3.7%)	–	0.15 (11%)	0.05 (2.3%)	0.20 (2.3%)	–
Steam stones													
S1 Chewing gum	Ice	<0.03	–	–	<0.03	–	–	>0.03	–	–	0.62 (12%)	–	–
S2 Grape	Ice	<0.03	–	–	0.07 (2.6%)	0.58 (3.6%)	0.58 (4.1%)	<0.03	–	0.06 (0.2%)	0.04 (2.4%)	0.32 (1.1%)	–
S3 Mint	–	–	–	–	–	–	–	–	–	–	5.62 (51%)	–	–
S4 Apple	0.12 (11%)	<0.03	–	–	<0.03	–	<0.03	0.25 (11%)	–	0.06 (9.6%)	–	0.17 (9.6%)	–

Concentrations (mg/g) in this table are an average of n=2 with the relative standard deviations (RSD) in parentheses. Below limit of detection (LOD); –; –; above LOD, but below limit of quantification (LOQ); <0.03; estimated value (above range calibration curve) are shown in *italics*.
GC–MS, gas chromatography–mass spectrometry; LOD, limit of detection; LOQ, limit of quantification.

popular flavours are fruit and mint in pod-based e-cigarettes (35% and 29%), e-cigarettes (53% and 23%) and waterpipe (45% and 19%, respectively).³⁰ Candy was shown to be a little more popular for e-cigarettes (21%) than waterpipe (15%).

The 10 most frequently added flavourings in waterpipe tobacco showed a lot of similarity to the most frequently added flavourings in e-liquids.²⁴ Both (top 10s) contain: vanillin, ethyl vanillin, maltol, ethyl butyrate and ethyl acetate. All these flavourings are described as fruity or sweet (table 1). Restricting such flavourings could be considered for all tobacco and related products in order to reduce the attractiveness of any type of smoking.

Chemical analysis of flavourings and nicotine in waterpipe tobacco, herbal molasses and steam stones

Our selection of herbal molasses and steam stones did not contain nicotine, whereas waterpipe tobacco did. Waterpipe tobacco nicotine levels ranged from 0.53 to 1.39 mg/g. Nicotine levels were not registered in EU-CEG as this is not an additive but a component of tobacco. Also, nicotine concentrations were rarely mentioned on packaging materials. Erythropel *et al*³⁶ detected nicotine concentrations of 0.65–1.8 mg/g in different flavours of waterpipe tobacco after extraction with methanol, which is similar to our findings.

We found that herbal molasses contained similar median concentrations of flavourings investigated in this study (0.38 mg/g) as waterpipe tobacco (0.28 mg/g). Steam stones, however, contained lower median flavourings concentrations (0.06 mg/g) than waterpipe tobacco or herbal molasses. Steam stones were shaken in the liquid they are dispersed in before extraction, but this liquid was not extracted and analysed separately. We expect no difference in the identified flavourings between the liquids and the steam stones dispersed in the liquids, since the flavourings are most likely dispersed in the liquid before adding the liquid to the porous stones. It is not known whether users add more of the liquid to the (saturated) stones before smoking the waterpipe. There are limited scientific reports on the flavourings in steam stones. Clutterbuck *et al*³⁷ identified various volatile organic compounds, including some flavourings, in the smoke condensate of steam stones against a mass spectral library. However, they did not confirm the presence of these compounds with chemical analytical standards. More research is required on both flavouring and nicotine contents of nicotine-free and nicotine-containing steam stones and on the amount of these stones and liquid used in a waterpipe smoking session.

Anethole was included for analysis due to high quantities in EU-CEG, and it has been found an important ingredient for apple-flavoured waterpipe products.²⁸ Our findings are in line with the aforementioned study, since apple-flavoured waterpipe products (M1, M3, S4 and T8) all contained anethole, whereas little anethole was found in non-apple-flavoured products. We found benzyl alcohol to be mostly present in apple-flavoured products as well, although in lower quantities than anethole. Apple is the largest subcategory in 'fruit other' and makes up 7.5% of all registered waterpipe tobacco products, indicating anethole and benzyl alcohol are crucial additives for waterpipe products. Menthol is added to nearly every waterpipe product, in varying concentrations. Reasons for adding mentholic flavours to tobacco products are adding a fresh taste or facilitating smoke inhalation by reducing the harshness of smoke by providing a cooling sensation.³⁸ It is likely that high menthol concentrations contribute to a menthol/mint flavour, especially with concentrations over 1 mg/g.³⁹ However, lower concentrations can still provide a cooling effect without adding a characterising

menthol/mint flavour to the smoke.⁴⁰ The cooling property of lower menthol concentrations should be addressed more carefully in the future, since it may facilitate inhalation for especially young or new smokers that are not used to the harshness or irritating properties of tobacco smoke.⁴¹

Registered flavouring concentrations were available for waterpipe tobacco products T3–T8 in the EU-CEG database (data not shown). We identified most registered flavourings; however, registered quantities showed little similarity with our findings. Since there are many explanations for these deviations (such as erroneous entered values, non-matching CAS numbers in EU-CEG or storage and transportation of the products), additional research is required to properly compare the quantitative yields of flavourings found by chemical analysis and the registered data in EU-CEG. Still, with many possible reasons, the exact cause(s) for deviations between quantities found by chemical analysis or registered in EU-CEG will be difficult to establish with full certainty.

Limitations

Tobacco product data in EU-CEG are entered by manufacturers or importers. However, such information may not always be correct, complete or up to date. Some flavourings such as menthol are or can be part of an extract or oil, which limits quantitative comparison with registered quantities. No recovery experiments were performed, so the measured flavouring and nicotine concentrations are semiquantitative and might be an underestimation of actually present concentrations. We did not determine the percentage of liquid for each steam stone sample. We recommend calculating the weight percentage of liquid in steam stone samples in future studies to be able to correct for changing amounts in glycerol liquid per steam stone sample.

CONCLUSION

Database (EU-CEG) analysis showed that the most prevalent waterpipe tobacco flavours, to be marketed in The Netherlands,

What this paper adds

What is already known on this topic

- ⇒ Flavours contribute to the onset and continued use of waterpipe tobacco smoking, especially in youth.
- ⇒ European Common Entry Gate database analysis is a potential source to observe trends in flavours of registered products and chemical analysis can be used for identification and quantification of flavourings in tobacco and related products.

What this study adds

- ⇒ A flavour wheel has been created containing all flavours identified in the EU-CEG database, providing a quick overview of waterpipe tobacco flavours. This will aid communication among experts around the globe.
- ⇒ We identified and obtained semiquantitative information on a selection of commonly added flavourings in waterpipe tobacco, herbal molasses and steam stones (which are widely available in EU countries and the UK) by gas chromatography–mass spectrometry analysis.

How this study might affect research, practice or policy

- ⇒ All products in our study were flavoured, and none of them had a tobacco flavour. This has implications for policy makers that consider banning flavours in waterpipe products.

are fruit-flavoured. Fruity, sweet and minty flavours covered nearly all categorised waterpipe tobacco products. It is known that especially these flavours are appealing to youth. Flavoured products contribute to the onset of waterpipe smoking and continued use of the waterpipe. GC–MS analysis revealed that the most prevalent flavourings are present in similar levels in herbal and tobacco waterpipe products. Steam stones contain lower flavouring concentrations; however, it is not known whether additional flavour-containing glycerol liquid is added by users. Anethole is present in high quantities in apple-flavoured products and menthol is present in the majority of waterpipe products in varying levels. To discourage or reduce smoking among youth by product regulation, limiting or banning flavours or flavourings could be a promising strategy.

Contributors RT and IMEB-H conceptualised and designed the study. IMEB-H designed the flavour wheel and wrote the manuscript and is the guarantor of this manuscript. FB and NW performed laboratory experiments. JLAP and SE preprocessed data from the European Common Entry Gate (EU-CEG) system. RT provided critical feedback on the manuscript.

Competing interests None declared.

Patient consent for publication Not applicable.

Ethics approval This study does not involve human participants.

Provenance and peer review Not commissioned; externally peer reviewed.

Data availability statement No data are available. Only institutions that have access to the EU-CEG system will have access to the raw data, since much of the data is confidential information from manufacturers.

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