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Assessing toxicant emissions from e-liquids with DIY additives used in response to a potential flavour ban in e-cigarettes

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ABSTRACT

Significance Electronic cigarettes (e-cigarettes) aerosolise liquids that contain nicotine, propylene glycol, glycerol and appealing flavours. In the USA, regulations have limited the availability of flavoured e-cigarettes in pod-based systems, and further tightening is expected. In response, some e-cigarette users may attempt to make their e-liquids (do-it-yourself, DIY). This study examined toxicant emissions from several aerosolised DIY e-liquids.

Methods DIY additives were identified by reviewing users' responses to a hypothetical flavour ban, e-cigarette internet forums and DIY mixing internet websites. They include essential oils, cannabidiol, sucralose and ethyl maltol. E-liquids with varying concentrations and combinations of additives and tobacco and menthol flavours were prepared and were used to assess reactive oxygen species (ROS), carbonyl and phenol emissions in machine-generated aerosols.

Results Data showed that adding DIY additives to unflavoured, menthol-flavoured or tobacco-flavoured e-liquids increases toxicant emissions to levels comparable with those from commercial flavoured e-liquids. Varying additive concentrations in e-liquids did not have a consistently significant effect on the tested emissions, yet increasing power yielded significantly higher ROS, carbonyl and phenol emissions for the same additive concentration. Adding nicotine to DIY e-liquids with sucralose yielded increase in some emissions and decrease in others, with freebase nicotine-containing e-liquid giving higher ROS emissions than that with nicotine salt.

Conclusion This study showed that DIY additives can impact aerosol toxicant emissions from e-cigarettes and should be considered by policymakers when restricting commercially available flavoured e-liquids.

WHAT IS ALREADY KNOWN ON THIS TOPIC

⇒ Electronic cigarette (e-cigarette) liquids are available in many flavours; however, policies have been proposed or implemented that prohibit many flavoured e-liquids.
⇒ E-cigarette users often add unregulated additives to their e-liquids to obtain desired flavours.
⇒ Previous research has demonstrated that liquid composition can impact e-cigarette aerosol toxicant profile.

WHAT THIS STUDY ADDS

⇒ This study identified ingredients that e-cigarette users may add to e-liquids in response to flavour-limiting policies and systematically assessed the impact of do-it-yourself (DIY) additives on e-cigarette toxicant emissions.

HOW THIS STUDY MIGHT AFFECT RESEARCH, PRACTICE OR POLICY

⇒ Because DIY e-liquids may result in similar or increased toxicant emissions to commercially available flavoured e-liquids, regulations that limit e-cigarette flavours may have unintended consequences if the use of DIY e-liquids increases.
⇒ Policies that prevent the use of DIY liquids, such as prohibiting 'open-system' e-cigarettes, may strengthen the effects of policies that limit e-cigarette flavours.

INTRODUCTION

Electronic cigarette (e-cigarette) use has increased globally in the last decade, especially among youth.[1–3] Epidemiological data have linked e-cigarette use trends to efficient nicotine delivery, flavour availability and harm reduction claims.[4–7] The availability of flavours marketed heavily with vivid descriptions of taste and sensory experiences has contributed to e-cigarette experimentation and use among youth.[8 9] Importantly, using multiple flavours is associated with increased frequency of e-cigarette use,[10 11] and adolescents who use flavoured e-cigarettes are more likely to become regular users.[12]

Recently, the US Food and Drug Administration implemented an enforcement policy removing all flavoured (except tobacco or menthol) 'cartridge-based e-cigarettes' from the market.[13] However, many flavoured e-liquids are still available and can be used in refillable 'open-system e-cigarettes', until the enforcement of the Premarket Tobacco Product Authorization which denied the marketing of ~1 million flavoured e-cigarettes.[14] Some users of open systems prepare their e-liquids,[15] a practice known as do-it-yourself (DIY).[16] DIY liquids can be prepared from propylene glycol and glycerol (PG/G), ready-to-use DIY flavour concentrates with unknown ingredients, and/or other DIY additives that may or may not be intended for use in

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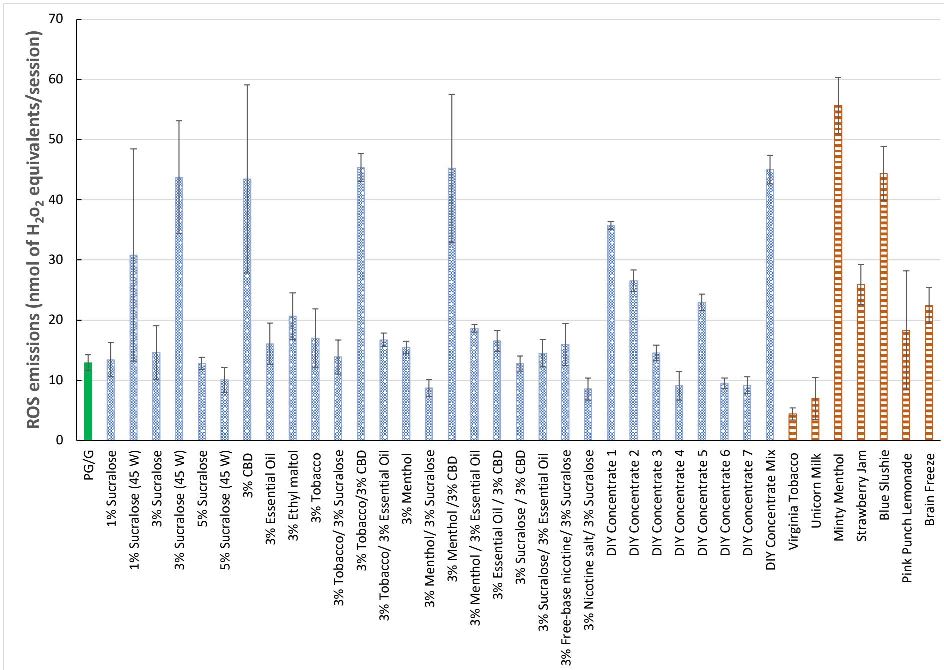

Figure 1 ROS emissions from 27 laboratory-prepared DIY e-cigarette liquids (dotted fill) in comparison with unflavoured PG/G liquid (solid fill) and seven commercially available flavoured e-liquids (horizontal stripes), all generated at 30 W. Emissions from e-liquids with different levels of sucralose at 45 W were added for comparison. CBD, cannabidiol; DIY, do it yourself; e-cigarette, electronic cigarette; G, glycerol; PG, propylene glycol; ROS, reactive oxygen species.

e-cigarettes. There are limited data regarding the prevalence of DIY behaviours (and subsequent health risks), although previous research has reported less than 5% of current users make DIY liquids.[17] However, flavour concentrates used in DIY preparations constitute a large share of the e-cigarette market.[18] Additionally, research examining the potential impact of a hypothetical e-cigarette flavour ban suggests some users may consider making their e-liquids in response.[19 20] Hence, this study aimed to assess the impact of DIY behaviours on toxicant emissions from e-cigarettes.

METHODS

DIY flavour additive identification

A concept mapping study was conducted to identify and describe reactions that current e-cigarette users (n=71) may take in response to a hypothetical policy that only allows the sale of tobacco, menthol or unflavoured e-liquids.[19] A primary response was making DIY e-liquids and adding compounds such as cannabidiol (CBD) and essential oils. To identify DIY recipes that contained the identified ingredients, a search of 42 e-cigarette forums[21] and a Google search for 'DIY e-liquid recipes' were conducted.

Study design

We differentiate between DIY additives that are based on chemical compounds (eg, sucralose, CBD, ethyl maltol and essential oil), DIY concentrates that could be added to e-liquids as flavourings (eg, concentrates 1–7 in figure 1) and ready-to-use flavoured e-liquids (eg, menthol, tobacco and flavoured e-liquids mentioned in figure 1). We prepared e-liquids with one additive (3% concentration) in PG/G (30/70) or a mixture of two additives (each at 3% concentration). A constant mass of CBD powder (m=0.25 g) was used for all CBD-containing liquids. Tobacco-flavoured or menthol-flavoured e-liquids were prepared at 3% level with or without additives. To assess the impact of nicotine on toxicant emissions, two e-liquids were prepared with sucralose (3%) and either freebase nicotine (3%) or nicotine salt (3%). Also, e-liquids with different sucralose levels (1%, 3% and 5%) were tested at two different powers (30 W and 45 W). Seven DIY concentrates were tested individually or as a mixture. Seven commercially available flavoured e-liquids selected from top flavours in the market were tested for comparison. We measured reactive oxygen species (ROS), carbonyl and phenol emissions in machine-generated aerosols. Statistical significance was evaluated using t-test on head-to-head comparisons ($p<0.05$).

Aerosol generation and sampling

The AUB Aerosol Lab Vaping Instrument[22] was used to generate aerosols from a Kangertech Subox Mini e-cigarette. The puffing regimen consisted of 10 puffs of 4 s puff duration, 10 s interpuff interval and 8 L/min flow rate. The flow was split into three branches: a 1 L/min branch was used for carbonyl quantification using a 2,4-dinitrophenylhydrazine gas sampling cartridge placed downstream a filter pad,[23] a 3.5 L/min branch was used for quantification of phenols in the particle phase trapped on the filter pads,[24] and the filter of the other 3.5 L/min branch was directly submerged in 2′,7′-dichlorofluorescein probe solution for ROS analysis. Each condition was tested in triplicate.[25]

RESULTS

Data showed that ROS emissions from DIY concentrates or from menthol and tobacco flavours mixed with DIY additives were often significantly higher than PG/G base e-liquid and like those emitted from commercially available flavoured e-liquids (figure 1). Mixing menthol flavour with CBD or essential oil yielded significantly higher (192% and 20% increase, respectively) while sucralose yielded significantly lower (44% decrease)

ROS emissions than menthol with no additives. Only CBD additive significantly increased ROS emissions from tobacco flavour (166%). Although the nature of the additive was impactful on ROS emissions, different concentrations of sucralose did not yield higher emissions than unflavoured PG/G liquid. Also, the effect of power increase (from 30 W to 45 W) was dependent on sucralose concentration (significant at 3% only). Interestingly, nicotine form affected ROS emissions from a liquid containing sucralose, with nicotine salt yielding lower ROS emissions than freebase nicotine.

Carbonyl and phenol emissions from DIY concentrates or from menthol and tobacco flavours mixed with DIY additives were also comparable with commercial flavoured e-liquids (online supplemental table S1 and figures S1–S4). The addition of 1% and 5% of sucralose to PG/G led to a significant decrease in acetone (Ac) (~50%), crotonaldehyde (CA) (~30%), methacrolein (MAcr) (~40%) and catechol (not detected, ND), yet a significant increase in propionaldehyde (PA) (255% and 123%, respectively). The addition of 3% sucralose and 3% CBD had no significant effect on emissions and 3% essential oil increased CA and MAcr (19% and 6%, respectively). Adding 3% ethyl maltol resulted in increasing acetaldehyde (AA) (9%) and methyl glyoxal (MGA) (38%), while decreasing Ac (47%), CA (27%), MAcr (41%) and catechol (ND). Higher power (45 W vs 30 W) led to higher emissions of AA (458%), Ac (288%), MAcr (300%) and phenol (290%) for e-liquids with 1% sucralose, higher CA (1661%), glyoxal (GA) (2347%), MGA (4094%) and phenol (237%) for 3% sucralose, and higher AA (346%), Ac (267%), MAcr (269%) and GA (359%) for 5% sucralose. The influence of sucralose percentage on emissions at 30 W was mixed, as was the influence of mixing more than one additive with PG/G base.

The addition of freebase nicotine or nicotine salt to PG/G with sucralose led to a significant increase in AA (50% for freebase) and PA (203% and 300%, respectively) and a significant decrease in Ac (~40%), MAcr (~34%), GA (~25%) and catechol (ND with nicotine). DIY additives increased certain emissions from tobacco-flavoured e-liquids like Ac (54%), CA (32%), MAcr (46%) and phenol (144%) with sucralose, formaldehyde (FA) (534%), PA (102%) and CA (33%) with CBD, and Ac (79%), CA (52%), MAcr (57%) and MGA (114%) with essential oil. However, for menthol-flavoured e-liquids, CBD showed no significant effects (due to the presence of an outlier), while sucralose and essential oil increased Ac (62% and 74%), CA (37% and 69%) and MAcr (46% and 62%). It should be noted that additives decreased some emissions, like the case of sucralose added to menthol-flavoured e-liquids (FA: 47%; AA: 47%; Acr: ND; and PA: 86%).

DISCUSSION

The results highlight that if flavoured e-liquids were removed from the market, as well as DIY concentrates, the addition of DIY additives to unflavoured PG/G or tobacco and menthol-flavoured e-liquids may yield similar toxicant exposure. Also, mixing concentrates and/or additives, which is a common practice among e-cigarette users,[10] may lead to significantly higher ROS emissions compared with individual concentrates (figure 1).

The data on carbonyl and phenol emissions showed mixed observations with increase in some emissions and decrease in others for the same e-liquid condition. This could be attributed to the different mechanisms of formation during e-cigarette operation,[26 27] or to coil variability in triplicate measurements that can mask the impact of liquid ingredients on emissions.[28] Also, focusing on toxicants that are mainly formed from PG/G degradation[23 24 29] may lead to overlooking other degradation pathways, as in the case of sucralose leading to chloropropanols.[28 30] Moreover, toxicant assessments should consider secondary interactions between e-liquid constituents.[31] This could explain the lower ROS emissions from nicotine salt that could be attributed to ROS trapping on the benzene ring of the benzoate counter anion.[32 33] Nevertheless, the current work shows that DIY additives can contribute significantly to e-cigarette toxicant emissions and questions the need for 'open-system' e-cigarettes that allow for such exposure to unregulated additives.[34]

Limitations of the current work include using ready-to-use DIY additives dissolved in PG (eg, sucralose) or containing unidentified chemicals (eg, essential oil). The data only represent 10 puffs, and more puffs or chronic use may reveal further toxicant emissions. Additionally, after a wider flavour ban is implemented, more 'novice' DIY users may start making liquids with additive concentrations that exceed those assessed in this study. It may be worth considering more 'extreme' scenarios in the future.

CONCLUSION

E-cigarette users vaping e-liquids with DIY concentrates or menthol/tobacco flavours mixed with DIY additives may be exposed to the same or greater levels of toxicants as from the 'to-be banned' commercial flavoured e-liquids. If 'open-system' devices remain on the market allowing users to add DIY e-liquids to their e-cigarettes, regulators should consider how DIY behaviours could undermine the impact of any potential flavour ban.

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Contributors AE-H, ES, RS, REH and ST conceived of the study idea and study design. RS, REH, MD, ME-K, OA and AY conducted the experimental work. AE-H and ES wrote the first and final version of the manuscript. All authors revised the manuscript and approved its final version.

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Competing interests AS is a paid consultant in litigation against the tobacco and e-cigarette industry and is named on one patent for a device that measures the puffing behaviour of e-cigarette users and another patent application for a smoking cessation intervention.

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