The Stability of Diacetyl and Acetyl Propionyl and the Conversion of Acetoin to Diacetyl in e-liquids

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INTRODUCTION

Diacetyl (DA) and acetyl propionyl (AP) have been reported to be used for the creation of sweet flavour directions in e-cigarette liquids.[1] DA, whilst a food-grade flavouring, has known respiratory toxicity, and can cause a clinical condition known as bronchiolitis obliterans. Research has shown that AP has similar respiratory toxicity to diacetyl.[2] Both DA and AP are potential contaminants of e-liquids. Part one of this study investigates the stability of these ingredients in e-liquids.

Acetoin (AC) has been used as an alternative to DA in e-cigarette liquids, but while the toxicological data on it shows little of concern, its chemical similarity to DA means that conversion is a possibility (Figure 1), and may act as a source of DA in e-liquids. Part two of this study measured the potential for conversion of AC to DA in a number of different e-liquid matrices.

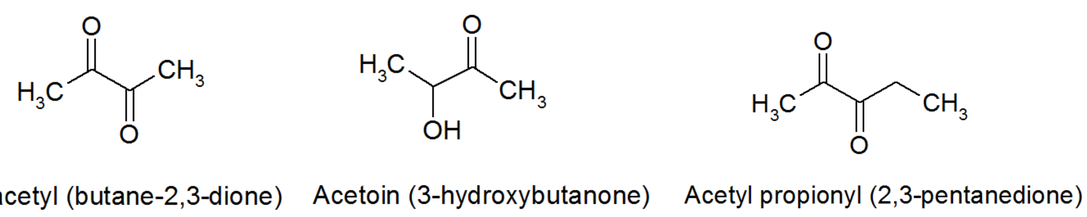

Figure 1: Chemical structures of diacetyl, acetoin and acetyl propionyl

PART ONE: Diacetyl and acetyl propionyl stability in e-liquids

METHOD:

E-liquid formulations were spiked with DA and AP. The samples were stored in amber glass bottles, with controlled environmental room temperature conditions of 20°C ± 2°C. The samples were analysed 18 days after spiking for three replicate measurements of AC, AP and DA using GC-MS at Enthalpy Analytical, Inc.

RESULTS AND DISCUSSION

After 18 days, the amount of DA and AP had significantly reduced from the amount that was originally injected (Figure 2).

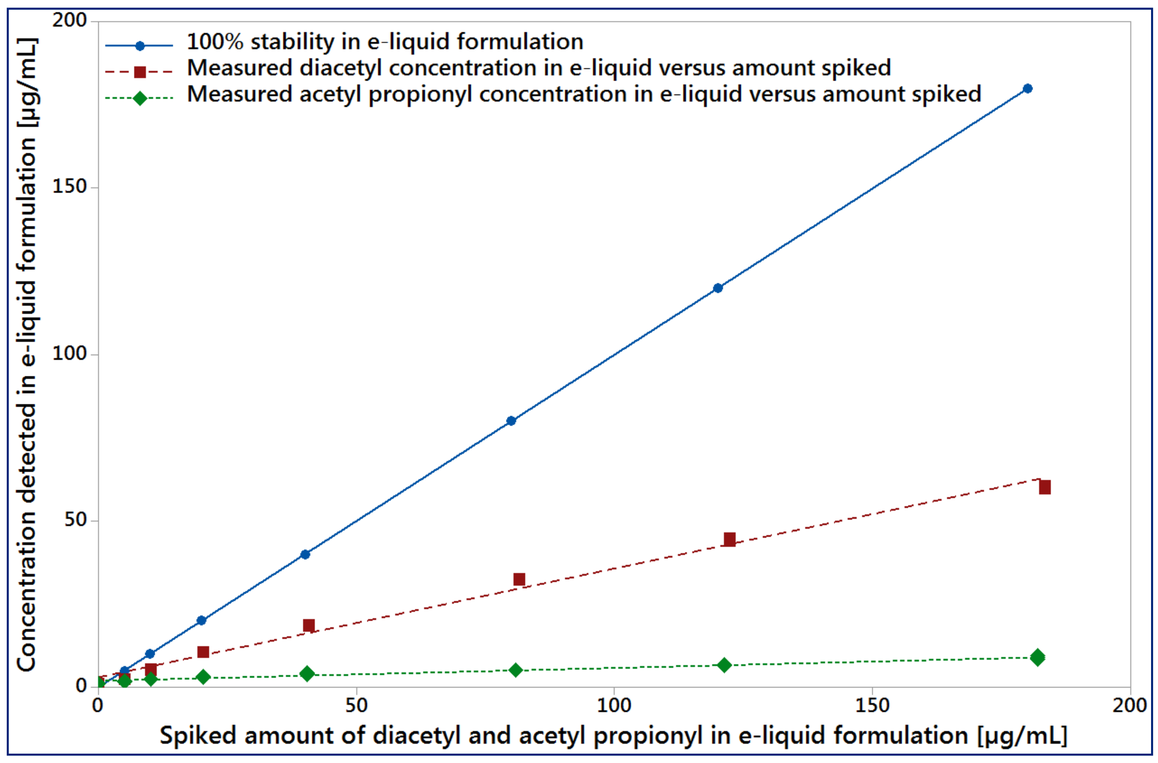

Figure 2. Measured concentration of DA and AP in an e-liquid formulation versus the amount initially spiked into e-liquid

Both DA and AP have been shown to have limited stability in a nicotine-containing e-liquid formulation. As DA and AP are both alpha-diketones, they could undergo reversible and irreversible addition reactions with glycerol or propylene glycol to form hemiketals and ketals, (Figure 3).

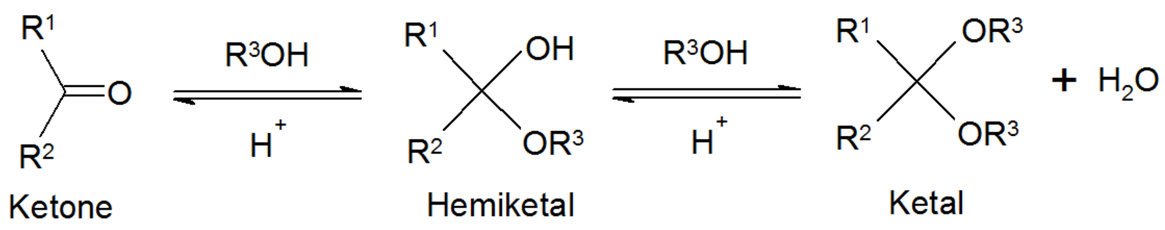

Figure 3. Generic ketone to hemiketal and ketal conversion

PART TWO: Acetoin conversion to diacetyl in e-liquids

METHOD

E-liquid formulations were spiked with 1000µg/mL AC. A further formulation was spiked at six different concentrations of AC. Samples were analysed at various time points to observe stability. Three replicate measurements of AC and DA were performed by GC-MS at Enthalpy Analytical, Inc.

RESULTS

Spiked e-liquids with an alkaline pH showed conversion of AC to DA over this time period.

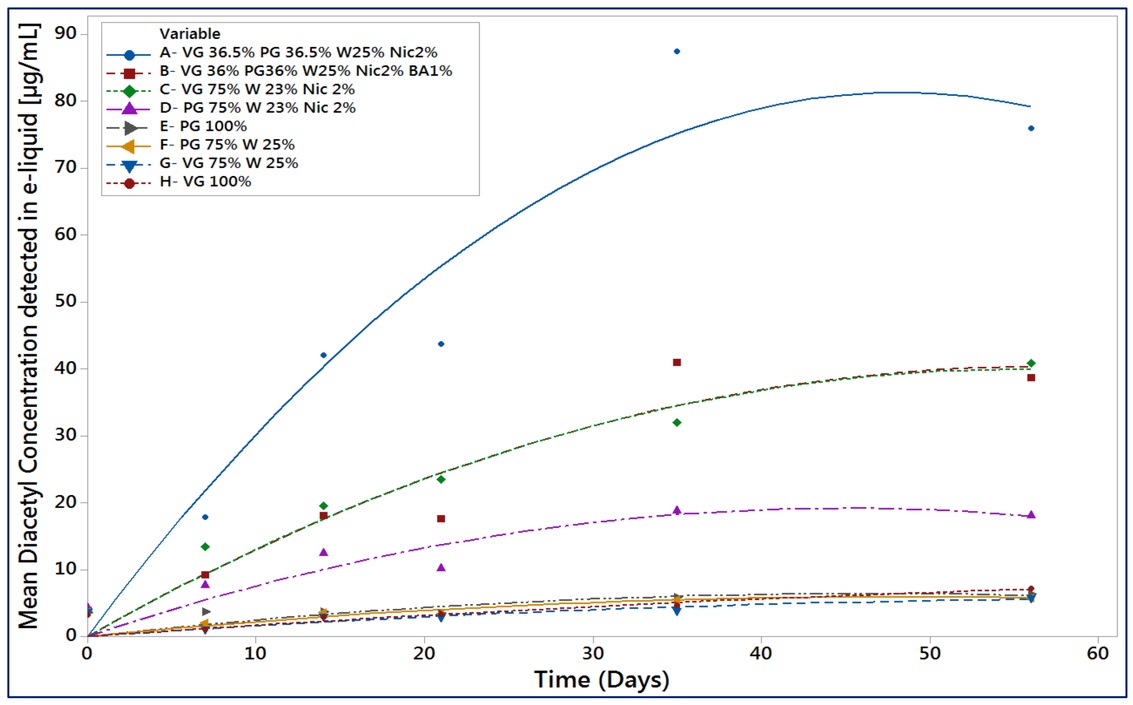

Figure 4: Measured diacetyl concentration over an 56 day time period

The formulation spiked with six different concentrations showed a quasilinear relationship between the level of spiked AC and the amount of DA formed. There was a lower extent of conversion at the highest AC concentration, consistent with a combination of linear conversion of AC to DA, combined with instability of generated DA in the e-liquid[2].

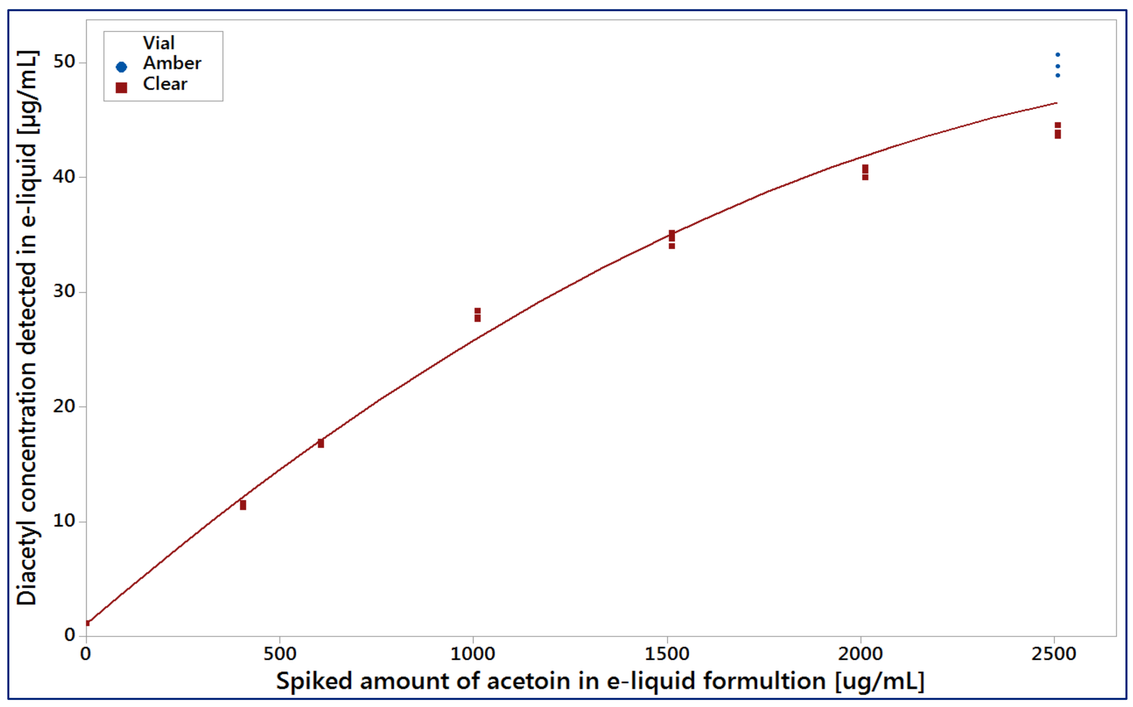

Figure 5: Measured diacetyl concentration in e-liquid 21 days after spiking

CONCLUSIONS

- Both diacetyl and acetyl propionyl are unstable in a common e-liquid formulation. Further work is underway to explain these observations
- Acetoin is a precursor to diacetyl in nicotine-containing e-liquids. Action should be taken by e-liquid manufacturers and flavouring suppliers to eliminate acetoin as a flavour ingredient

REFERENCES

1. Flavoring Chemicals in E-Cigarettes: Diacetyl, 2,3-Pentanedione, and Acetoin in a Sample of 51 Products, Including Fruit-, Candy-,and Cocktail-Flavored E-Cigarettes, Allen et al., Environmental Health Perspectives , 2016, 124 (6), DOI:10.1289/ehp.1510185
2. Evaluation of electronic cigarette liquids and aerosol for the presence of selected inhalation toxins, Farsalinos et al., Nicotine and Tobacco Research, 2015, 17 (2), 168 – 174

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