CANNABIS - WHITEPAPER COLLECTION

Preheated Cartridge method for uniform aerosol in high viscocity oils

CERULEAN

a coesia company

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Introduction

Vaping cannabis oil with a high cannabinoid content has proved challenging due to the high viscosity of the oil and the tendency for localised heating not to be sufficiently strong to deliver consistent aerosol.

The established method of loading a sample of cannabis oil (predominantly containing the lipid (-)-trans-delta-9-tetrahydrocannabinol which is commonly referred to as THC) into a cartridge for vaping testing at Kaycha Laboratory (Denver Colorado) was to mildly heat the cannabis oil in an oven set at approximately 50°C before injecting the oil into the cartridge that was to be used. This method overcame the high viscosity in many of the samples.

Once the cartridges were filled with the viscous cannabis oils a method was developed for volatilising the THC liquids, that involved the pre-heating of the cartridge through mild heating in an oven before vaping started. It was noted that the process of vaping, if sufficiently frequent, would mildly "self-heat" the liquid allowing aerosol formation throughout an experiment.

This 'gentle' method of heating the liquid, although not essential for the current Colorado State metal recovery regulations, since metals transfer and recovery rates are not affected by heat, anticipates wider toxicological analysis of THC aerosol in the future that may be required as knowledge and legislation regarding cannabis vapour develops. For example, should the Colorado State Regulators in the future wish to analyse other compounds such as carbonyls which can be produced by overheating rather than just directly transferred, it would be important to ensure that the cartridges are not overheated when producing the aerosol thus creating artificially high carbonyl results.

Additionally, the method might be useful for understanding the dose of THC/ Terpenes delivered to the consumer by a particular oil / device combination.

This has obvious implications when the oil is being used for therapeutic purposes – consistent delivery with a particular chemical profile would be essential for therapeutic purposes even when self-administered ad libitum. In recreational use the consumer would expect a similar dose from every usage and so uniformity is equally desirable.

By analogy the European Tobacco Products Directive, as it relates to nicotine vaping, demands that the dose per puff is uniform for the purpose of ensuring consumer satisfaction and safety.

An experiment was conducted to establish uniformity of delivery for three different cannabis oils from a common device type under common experimental conditions where a cartridge preheating method was used.

Experimental

Using the Cerulean CETI1 aerosol generation machine an experiment was performed on three cannabis oils of varying viscosities. These were supplied to Kaycha labs in small syringes (figure 1) which were placed in an oven overnight before the contents were transferred into a standard empty cartridge compatible with the Select vaping system (figure 2)

Figure 1: Typical THC cannabis oil as supplied to Kaycha labs.

Three high total cannabinoid content cannabis oils were selected for the vaping test – high, medium, and lower viscosity. Despite the designation all required preheating to allow transfer to the vaping cartridge. The samples were labelled 18HV (High Viscosity), 16MV (Medium Viscosity), 17LV (Low Viscosity).

Puffing on the product was conducted in “puff blocks”. These are a sequence of puffs that are interrupted to make measurements or to change capture systems. This allows intermediate measurements to be made while a cartridge is puffed to exhaustion.

For instance, a series of capture pads can be prepared and exchanged after each puff block and sent for chemical analysis to not only ensure a consistency in total aerosol mass delivered but also the concentration of a particular component such as THC being delivered throughout the use session. Puff blocks for this experiment were defined as being 40 puffs of 3 second duration, volume 55ml with a square shaped profile.

Delivery uniformity of THC Aerosol using "preheated Cartridge" method when vaping high viscosity cannabis oils. Continued..

Figure 2: Typical Select Vaping system with cannabis oil in cartridge.

Puffs were taken on a 30 second cycle. It was observed that the noticeably warm cartridge was still warm at the end of the puff block through heating from the coil.

Before use the cartridge was heated to approximately 50°C in an oven. A series of capture pads was prepared so that these could be removed, capped, and a new pre-weighed capture pad fitted within 30 seconds so that the cartridge did not cool whilst the exchange took place. The capture pad removed could be weighed whilst the machine continued puffing.

For the vaping experiment the device was angled at 15° to the horizontal to mimic user preferred use patterns.

The CETI1 aerosol generation machine was manufactured and supplied by Cerulean, Rockingham Drive, Milton Keynes UK and consists of a programmable puff engine with a maximum sweep volume of 70ml based around a stepper driven controlled precision aluminium bore syringe.

The primary capture pad was a glass fibre pad supplied by Whatman, specified to capture 99.9% of all particles with a diameter of 0.3 micrometres or greater in a Cambridge Filter Holder (CFH).

The device under test was activated by flow and was fitted with a square shaped mouthpiece. This was connected to the CFH labyrinth seals, a set of thin silicone seals that allow a seal to be formed gently around test products with cylindrical symmetry, via a short length of silicone tubing that pushed into the CFH seal.

At the start of the experiments a freshly charged battery pack was used. This was exchanged for a new battery pack after 120 puffs. This introduced some inconsistencies as some battery packs did not have sufficient charge to deliver a full 120 puffs – see results section.

A Baratron transducer was placed in the circuit beyond the primary capture pad to allow measurement of the pressure drop / flow in the puffing circuit during the puff blocks. This had a two-fold purpose; firstly to observe puff shape distortion through increased pressure drop through resistance to flow in either the capture pad or device and secondly to ensure that pressure drop was not rising and so restricting flow.

A 0.1mg resolution balance was used for mass balance calculations.

The experimental protocol required that the capture pads used were weighed before and after use to determine the weight of aerosol captured for a puff block.

Results

The three samples of lower, medium, and high viscosity oils were tested under identical conditions. During the tests, the limitations of the battery packs became evident with the sample 18HV fourth block being completely discarded as the battery pack failed after 120 puffs – it had not been changed – and in the case of Sample 17LV where the third puff block was terminated after 30 puffs as the battery pack began to fail. This was observed by a significant change in pressure drop that was being monitored. The test on Sample 16MV involved both monitoring and changing the battery packs during use.

Three tables are appended showing the delivery per puff block for the three oil samples.

Table 1: Delivery per puff for product with medium viscosity product 16MV

Product 16MV			
Puffs	**Weight captured per block (g)**	**Delivery per puff (g)**	**Total weight captured (g)**
40	0.0634	0.001585	0.0634
80	0.0426	0.001065	0.1060
120	0.0572	0.001430	0.1632
160	0.0424	0.001060	0.2056
200	0.0594	0.001485	0.2650
Average per puff delivery		0.001325	
Std dev per puff delivery		0.000246	
COV		0.18566	

Table 2: Delivery per puff for low viscosity cannabis oil Product 17LV

Product 17LV			
Puffs	**Weight captured per block (g)**	**Delivery per puff (g)**	**Total weight captured (g)**
40	0.0641	0.001602	0.0641
80	0.0423	0.001057	0.1064
110	0.035	0.001167	0.1414
150	0.0434	0.001085	0.1848
190	0.0354	0.000885	0.2202
Average per puff delivery		0.001159	
Std dev per puff delivery		0.000268	
COV		0.23123	

Table 3: Delivery per puff for high viscosity cannabis oil Product 18HV

Product 18HV			
Puffs	**Weight captured per block (g)**	**Delivery per puff (g)**	**Total weight captured (g)**
40	0.0363	0.000907	0.0363
80	0.0323	0.000807	0.0686
120	0.0231	0.000577	0.0917
160	0.0313	0.000783	0.1230
200	0.0295	0.000737	0.1525
Average per puff delivery		0.000762	
Std dev per puff delivery		0.000121	
COV		0.15879	

The uniformity of delivery throughout a puffing session can be established by plotting puff mass against puff number as shown in figures 3 through 5.

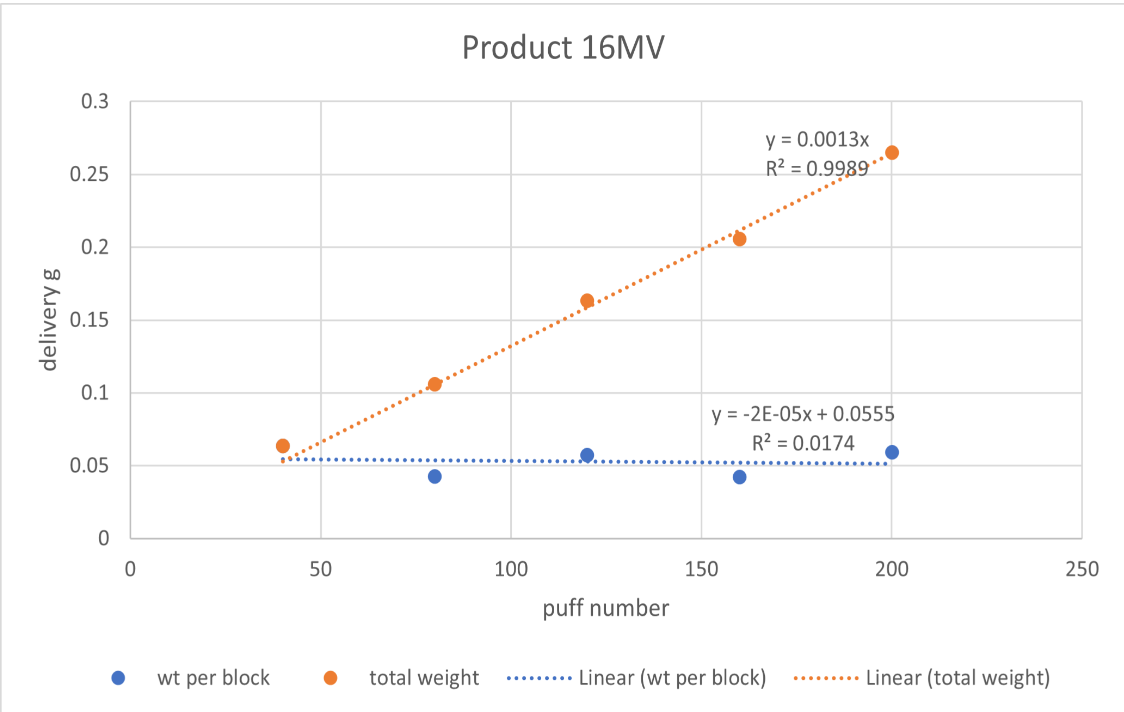

Figure 3: Plot of delivery from Product 16MV per puff and on a cumulative basis

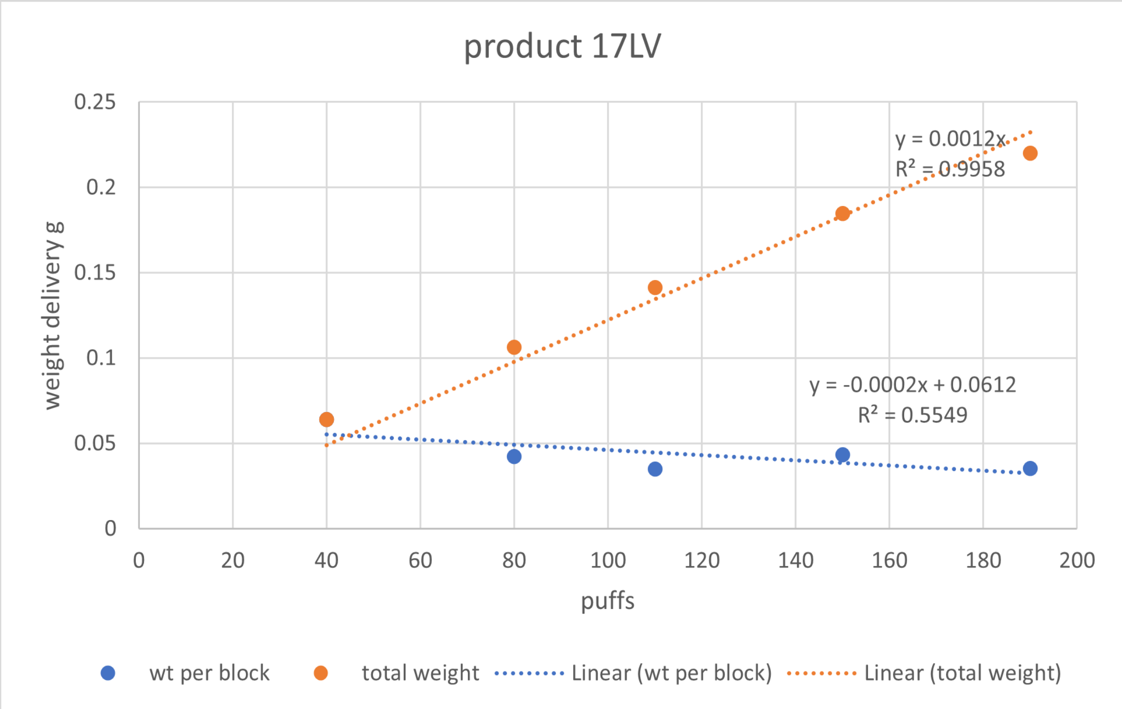

Figure 4: Plot of delivery from Product 17LV per puff and on a cumulative basis

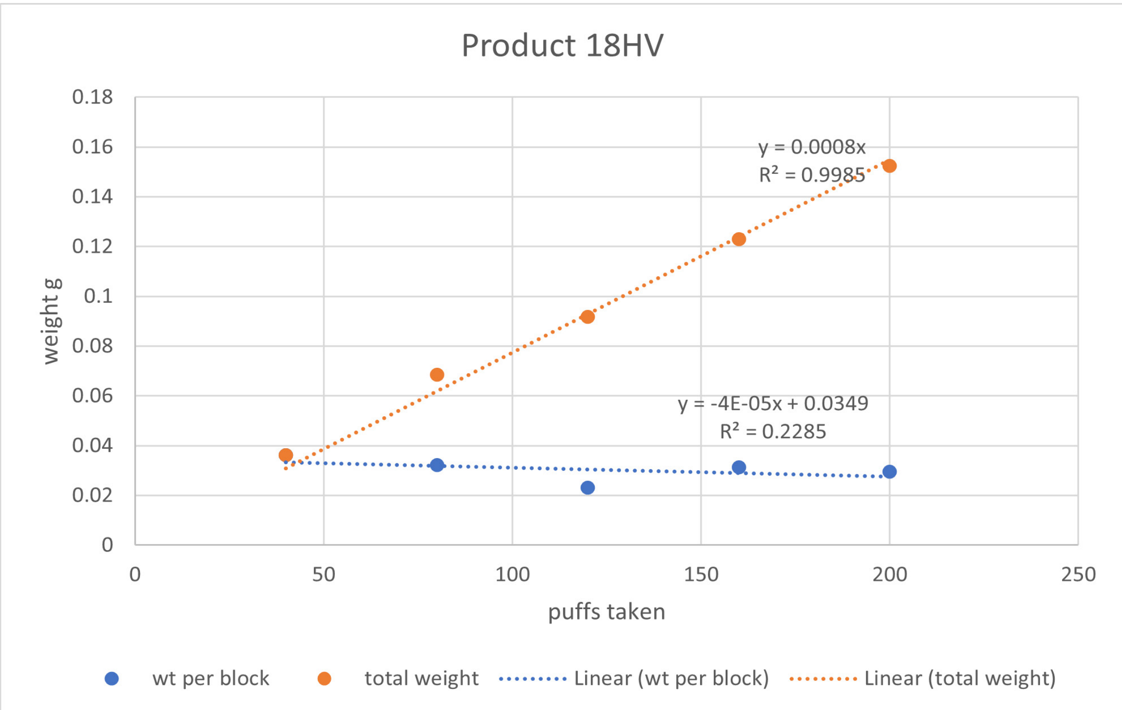

Figure 5: : Plot of delivery from Product 18HV per puff and on a cumulative basis

Discussion

The limitations of the battery packs indicates that if battery powered vaping is to be conducted then some care must be taken in experimental design to avoid problems with unplanned changes in delivery. It is suggested that before any analytical tests are performed the discharge characteristics of the battery packs with the vaporising cartridges are established. This would be establishing the number of puffs that can be delivered consistently from a single full charge. This could be established by using the battery pack and an easily volatilised mixture in a cartridge of the correct type, propylene glycol (PG) or vegetable glycerine (VG) such as used in nicotine vaping devices would be suitable.

By puffing in puff blocks the final number of puffs that can be delivered can be determined as can any indications of decreasing performance of the battery pack. This then allows an assessment of the delivery for a fully charged pack.

Once the maximum puff number is known a suitable effective maximum number of puffs from a single battery can be established at 60% of the experimentally determined maximum.

Despite the designation of LV (low viscosity), MV (medium viscosity) and HV (high viscosity) all samples were very viscous and so this becomes a relative designation. The most “fluid” of the oils had the characteristics at room temperature of honey brought straight from a refrigerator and the highest viscosity product almost seemed to be “set” when examined at room temperature. The heating in the oven prior to use decreased the viscosity considerably to that of perhaps thick engine oil. The puffing regime self-heated the cartridge which maintained this level of viscosity throughout the experiment.

The average per puff delivery for the high viscosity product 18HV is significantly lower than the other two products which have similar average per puff deliveries (0.76mg/puff for 18HV vs 1.16 and 1.33mg/puff respectively for 17LV and 16MV). The COV for all products is similar.

All three products can be vapourised using the mild pre-heating method. The graphs show that each puff block delivers a similar amount of aerosol for capture as can be seen by the total weight of aerosol captured increasing in what at first sight appears to be a linear manner, the R^2 value being close to 1.

On closer examination, particularly of the aerosol captured per puff for each puff block, it can be seen in each case that there is a decrease in per puff delivery as the puff block experiment progresses.

The linear fit to the data is not good, with low R^2 values, but there is a clear trend and the per puff delivery for the first puff block in all cases is between 25% and 60% higher than the average per puff delivery for the remaining experiment. A better fit can be achieved with a second order curve with R^2 values approaching 0.8.

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The higher delivery for the first puff block appears part of a general decrease in the delivery of aerosol as the cartridge is used. However, the first block to second block drop is greater than the drop between subsequent blocks indicating a rapid initial drop off in aerosol formation. The higher delivery for the first puff block could be due to loss of cannabis oil at the interface with the coil/wick as it is depleted in use or it could be a function of the cannabis oil being warmer for this first puff block coming straight from the oven, giving greater mobility for the oil to reach the heater and wick. This is not clear from the data presented but other experiments where the cartridge is heated between puff blocks shows the same decay profile as seen here.

It is not entirely clear from the data generated if the drop in delivery through a session is a consequence of the battery/cartridge combination or a feature of the oil. However, given that the different oils produce different per puff deliveries it is a strong indicator that the oil composition is a dominant factor in the level of cannabis oil delivery per puff.

The consistency of delivery per puff throughout a use session may be of some interest to the product designer, the formulation scientists and ultimately the user. If the design of the cannabis oil, the proportion of particular terpenes within the oil for example, is critical to any claimed health benefit of the oil then it is critical that the terpene profile is consistently delivered as the cartridge is consumed. This should be determined experimentally through the capture and analysis of aerosol delivered in puff blocks. Where the cannabis is being consumed for recreational purposes then the consumer should at least expect that the same mass of aerosol is delivered per puff throughout a use session, again determined by puff block analysis.

Improvement to the test method and apparatus could be made with a consistent gentle external heating of the cannabis oil cartridge throughout the experiment that would ensure reasonable mobility of the cannabis oil within the cartridge and so remove any doubt as to the transport of cannabis oil to the internal heating element.

Conclusions

The method of preheating cannabis cartridges containing THC to a modest 50°C before commencing vaping has the desired effect of allowing reasonable aerosol deliveries of between 0.7 and 1.3mg per puff. By not allowing the cartridges to significantly cool between puff blocks a consistent delivery of cannabis oil aerosol can be achieved.

The cartridge /cannabis oil system evaluated showed that as the cartridge is used the delivery per puff slightly decreases. This is evident for all three oils tested despite the mass of oil delivered per puff varied by cannabis oil type.

The applicability of gentle external heating to vapour production for establishing consistency of aerosol production and delivery is clear.

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