Faculty Development Committee (FDC) Interim Report

2021 Summer Faculty Development Grant

Trace Metal Profiling of Commercially Available Hemp Derived CBD Oils

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Rank of the PI:	Associate Professor
Department:	Chemistry & Physics
Award Date:	March 9, 2021

Introduction:

The objective of this project is to perform a "market basket" study of the trace element content of commercially available cannabidiol (CBD) oils to determine:

1) Do the products comply with current regulatory requirements as they pertain to established maximum contaminant levels for arsenic (As), mercury (Hg), lead (Pb), and cadmium(Cd).

2) Is there evidence suggesting that additional elements should be regulated beyond As, Hg, Pb, and Cd.

To answer these questions, CBD oils from 18 different manufacturers were purchased over the internet. Three discrete purchases of each manufacturer were made approximately 6 months apart so that batch to batch reproducibility across three (3) separate product lots could be assessed. This resulted in a total sample population of ~ 50 samples. A method for microwave acid digestion followed by inductively coupled plasma mass spectrometry (ICP-MS) analysis was developed and validated for over 20 elements of toxicological interest as defined by the National Institute of Health. The acquired samples were analyzed using the validated method with subsequent report out of the results.

Project Status:

The analytical method was developed and validated. The method validation protocol demonstrated the mean limit of detection (LOD) for each element in the CBD oil, the mean limit of quantitation for each element in the CBD oil, spike recovery at 5 parts per million (ppm), within run and between run percent relative standard deviation (%RSD), and an analysis of variance (ANOVA) at a 95% confidence level to demonstrate the lack of systematic error in the method (see Appendix).

Following validation, the first sample lot was purchased and analyzed. The second sample lot was then purchased, and the samples digested. Half the samples were analyzed and then the ICP-MS instrument failed. The third lot of samples was purchased and is awaiting processing.

The original quote from the manufacturer to service the ICP-MS instrument is ~$20,000. Rather than request the University absorb that expense, efforts are underway to troubleshoot and repair the instrument internally for the cost of parts. If unsuccessful, it would be necessary to request a service call from the manufacturer.

Results to date

As noted previously, a method has been developed and validated for use in this investigation. The majority of trace elements measured in the first lot of purchased CBD oil were below the method LOQ with several notable exceptions. Two of the samples formulated in moderate chain triglyceride (MCT) oil contained high levels of lead even though the certificate of analysis posted on the manufacturer's website stated the product was compliant with stated regulatory limits (the maximum contaminant level for most states is 1ppm):

Sample A	3.6 +/- 0.1 ppm Pb
Sample B	1.6 +/- 0.1 ppm Pb

This could be lead contribution from the biomass, product processing and/or the result of erroneous reporting. Another possible source was recently reported by the Florida State Department of Agriculture [ASTM Workshop on Measurement of Elemental Contaminants in Cannabis & Hemp, June 28 – 10, 2021] which found several commercial products in MCT oil which were believed to be leaching lead from the product packaging – specifically from the ink used on the dropper bottle. This has yet to be investigated for sample purchased in this study.

It was also found that all 5 samples formulated in hemp seed oil contained measurable quantities of Mn:

Sample C	0.32 +/- 0.06 ppm Mn	Sample F	0.63 +/- 0.06 ppm Mn
Sample D	0.63 +/- 0.06 ppm Mn	Sample G	0.26 +/- 0.04 ppm Mn
Sample E	0.26 +/- 0.02 ppm Mn		

As a reference, the WHO drinking water quality guidelines are 0.4 mg/L Mn, and the FDA has established that the manganese concentration in bottled drinking water should not exceed 0.05 mg/L.

As these levels were only found in the products formulated from hemp seed oil, it is likely that the Mn is originating from that diluent, however, this needs to be confirmed through analysis of pure hemp seed oil vs MCT.

These preliminary results suggest that the potential does exists for heavy metal contamination either from the biomass, processing, or packaging in CBD oils. Mn was found at elevated levels yet is not a currently regulated metal in CBD products. When the instrumentation is operational again, the two remaining product batches will be analyzed to assess lot to lot variability.

Presentations

Despite the delays in completing the entire project as outlined, there is significant interest in topic amongst both the regulatory and the scientific community. As a result, several oral and poster presentations have been given detailing these preliminary results.

- 2021 Middle Atlantic Regional ACS Meeting, June 9- 11, 2021, virtual (student poster)
- ASTM Workshop on Measurement of Elemental Contaminants in Cannabis and Hemp, June 28-30, virtual (PI - invited speaker)
- 2021 NOBCChE National Conference, September 16 – 18th, virtual (student poster)
- 93rd Annual National Technical Association Conference, September 22-26, Lincoln University (student presentation)
- 2021 SOFT Annual Meeting, September 27 – Oct 1, Nashville, TN (PI - poster)
- 2021 Lincoln University Annual Science Fair, November 12th, Lincoln University (student poster)
- 2022 AAFS Annual Meeting (accepted), February 21-24, 2022, Seattle, WA (PI - poster)

Project Completion

In light of the instrument issues, a request for project extension until the end of the spring semester was approved on 12/13/2021.

Appendix

Results of Method Validation

Element	Mean LOD (ppb) in oil	Mean LOQ (ppb) in oil	5ppm % Spike	Within Run %RSD	Between Run %RSD
Be	2.7	9.1	99	0.7	2.1
Al	34	113	102	1.7	3.8
V	5.0	17	100	2.1	2.8
Cr	22	73	99	1.7	3.1
Mn	9.9	33	102	1.6	2.5
Co	1.5	4.8	100	1.1	2.9
Ni	57	191	99	1.5	4.3
Cu	28	93.1	91	1.1	2.0
Zn	208	692	100	1.9	2.2
As	31	103	99	1.7	4.3
Se	223	744	105	2.4	9.9
Sr	0.7	2.2	100	1.1	2.9
Ag	1.2	3.9	91	0.5	2.9
Cd	1.6	5.2	98	0.6	2.8
Cs	0.5	1.5	99	0.9	2.5
Ba	2.9	9.6	98	1.8	2.2
Hg	2.0	5.0	97	1.1	2.3
Tl	0.3	1.1	101	0.8	2.0
Pb	1.3	4.4	98	0.7	1.7
Th	18	59	96	0.3	3.5
U	0.3	0.8	98	0.5	1.7

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	21.35	5	4.270	1.377	0.238	2.294
Within Groups	353.6	114	3.102			
Total	374.9	119				