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App Note

Lead Levels in Nutraceuticals: Spice and Cannabis Nutraceutical Hemp Products

Introduction

The consumption of botanical products has increased over the past two decades as consumers trend to what are perceived to be natural and high quality botanical products. The cannabis industry has taken the world by storm and has flooded market with new products. Recently, concerns have arisen around the safety of this largely unregulated market. Cannabis testing laboratories emerged to fill the need for specialized testing for cannabinoid potency, pesticides, bacteria/mold, and other potential contaminants.

Toxic heavy metals are too often found in many common food and health products as well as in our environment. Products which often originate from other countries around the world can be targets for higher contamination, adulteration and counterfeiting with or by heavy metals. The primary regions of spice and tea production around the world have often been cited as having less stringent safety and quality standards in regards to consumer products. Products from these regions have been noted to contain a variety of adulterants and contaminants including wear metals and toxic elements.

Cannabis, potentially, with its mixed legal status, may be poised to become the next crop to be adulterated. Recreational cannabis and hemp are both the same species. Federally, legal hemp products are easy to obtain by the general public. Hemp products are also used as base for cannabis products and cannabinoid extracts. However, due to a ban on hemp cultivation in the US, virtually all of the hemp based in the US is imported from China, India, Eastern Europe, and Canada. Studies of other commodities exported from these countries have reported widespread heavy metal contamination (i.e. spices, teas, grains, etc.).

The scope of this study was to analyze various spice and legal hemp nutraceutical products, currently on the market, for lead contamination and compare those levels to other foodstuff and nutraceutical products examined in past SPEX CertiPrep studies. Samples were digested using microwave digestion and analyzed by ICP-OES and ICP-MS.

Background

Plants are potential bioaccumulators of heavy metals. In the production of spice and cannabis products, a large amount of plant material is processed to extract dried materials, concentrates and oils, thereby increasing the risk of heavy metal contamination. One of the most widespread heavy metals often found in food is lead. Lead is one of the most ubiquitous toxic substances in the world present in soil, plants, water, and air. Over the centuries, lead has been dispersed by daily use of lead products, factory emissions, gasoline combustion, paint decay, pesticide application, and industrial use. Some lead contamination is natural through bioaccumulation of plants and animals exposed to lead-containing soil and water. Lead can accumulate in dense tissues within the body such as in bones and organs, or found condensed into dehydrated (nuts, dried fruit and spices) or concentrated food/health preparations (concentrates, tinctures, extracts). Some of our previous studies of calcium supplements, dried spices and nutraceuticals found as high as 4,800 µg/kg of lead in calcium supplements and up to 2,800 µg/kg of lead in cinnamon.

Methods and Materials

Samples:

Hemp products were purchased online from various sources including vitamin distribution companies, CBD suppliers and online auction sites. Two of the samples were encapsulated hemp oil and two samples were hemp product extracts in another matrix such as olive oil or alcohol base. One sample was identified as an essential oil. Spice supplements were purchased at online vitamin retailers (see Table 1).

Table 1. Nutraceutical Product Sample Sources and Descriptions.

ID	Form	Purchased From	Description	Suggested Daily Dose
HEMP CAP 1	Capsule	Vitamin Distribution Company	Cold Pressed Unrefined Hemp Seed Oil	4 g
HEMP CAP 2	Capsule	Vitamin Distribution Company	Hemp Oil from Premium Hemp Seeds, Cold Pressed	4 g
CBD EXT 1	Extract Supplement	CBD Cannabinoid Producer	Premium Hemp Extract Supplement (5000) in Olive Oil	1 g
CBD EXT 2	Extract Supplement	CBD Cannabinoid Producer	Hemp Classic Concentrate (1,500 mg) Tincture	1 g
HEMP EO	Essential Oil	Hemp Online Supplier	Cannabis Essential Oil 100% Clean, Chemical Free Sun Grown	1 g
HEMP SO	Supplement Oil	Vitamin Distribution Company	Cold Pressed Organic	28 g
CIN CAP	Capsule	Vitamin Distribution Company	Cinnamon	0.75 g
MUS CAP	Capsule	Vitamin Distribution Company	Mustard Seed	0.4 g
GIN CAP	Capsule	Vitamin Distribution Company	Ginger	1.04 g
TUR CAP	Capsule	Vitamin Distribution Company	Turmeric	1.44 g
CA CAP	Capsule	Vitamin Distribution Company	Calcium Supplement	1 g

Sample Preparation

Sample Digestion

- Samples were digested using a CEM Mars 5 Microwave
  - Microwave conditions
    - Easy Prep vessels and XP vessels
    - 0.2 g sample
    - 10 mL HNO_3
      - Some samples 1-2 drops HF
      - 15 minute ramp to 210 °C
      - 15 minute hold

Materials

- SPEX CertiPrep Standards
  - CLMS-1, CLMS-2, CLMS-3, CLMS-4: Multi-Element Solution Standards 1-4
  - USP <232> Standards: USP-TXM2, USP-TXM3, USP-TXM5

- Reagents
  - High Purity Nitric Acid

Instrumentation

- Perkin Elmer ICP-OES - Macroelements
- Agilent ICP-MS 7700 - Heavy Metals
  - Meinhard nebulizer
  - Cyclonic spray chamber
  - Analysis performed
    - Normal mode: Air
    - Collision mode: Helium

Method Design

This study was designed to examine heavy metal contamination present in nutraceutical preparations including CBD and hemp. The samples were first examined by ICP for macroelement content and to determine possible ICP-MS interferences for heavy metal quantitation. Limits for lead imposed by various organizations (FDA, EPA, AHPA, USP) were used as benchmarks for heavy metal exposure. Most limits were designed to apply to a 150 lb (68 kg) adult (Table 2). The AHPA (American Herbal Products Association) has produced some of the most stringent limits we found specifically for cannabis and botanical products.

In some cases, hemp and cannabinoid products have been popularized for the treatment of children with seizure disorders and other childhood illness. Other nutraceuticals are commonly given to children for other ailments such as ginger for nausea. The limits for heavy metals have not been generally examined for their application to children's health. For the purpose of understanding the possible exposure limits for children, the adult levels were calculated to the weight of a 23 kg child (approximately 50 lb) (Table 2).

Table 2. Daily Lead Limits from Various Sources Calculated for Adults and Children (µg/day).

Source	EPA	AHPA (2012)	ATSDR	USP <232>	Min	Max
Route	Oral RfD	Oral	Oral	Oral		
	Calculated Daily Limit	Calculated Daily Limit	Calculated Daily Limit	Daily Total		
70 kg Adult	None	6	10	5	5	30
23 kg Child (Calculated)	None	2	3	5	2	5

Lead Concentrations in Hemp Samples

The overall most prevalent heavy metal detected in the hemp samples was lead with samples ranging from 13 to 137 ppb (see Figure 1).

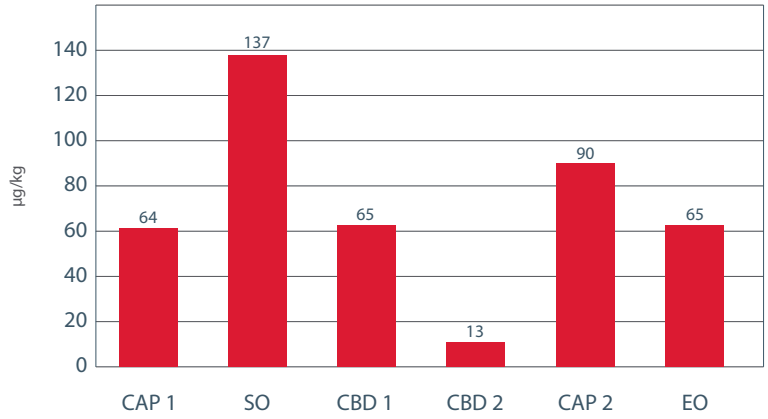

Figure 1. Pb Levels in Hemp Nutraceutical Products (µg/kg).

The suggested dosage for the various products was on average 1-2 tsp up to twice the daily limit for oils which calculated to about a minimum of 28 grams per day dosage. The dosage suggested on the extracts was one or more 0.5 mL doses, twice a day. This dosage was calculated out to be about a minimum of 1 gram per day dosage. The capsule dosage called for up to four 1,000 mg capsules a day (4 g dosage).

The concentrations measured in the hemp oil were calculated to give a final concentration of heavy metals for the stated dosages. The lead levels found in the oils were of the most concern when compared to daily limits. For an adult there is a 5 µg limit per day. Several samples of nutritional or medicinal hemp oil were found to give 50% or more of an adults limit. For children, many of the oil samples were well over a potential daily limit for a child. A 28 g dose of the highest oil would have provided over 200% of a potential daily limit for a child (Figure 3).

Lead Concentrations in Nutraceuticals

Lead concentration levels were significantly high in the spice nutraceuticals and calcium supplements ranging from 300 to 4,800 µg/kg (Figure 2).

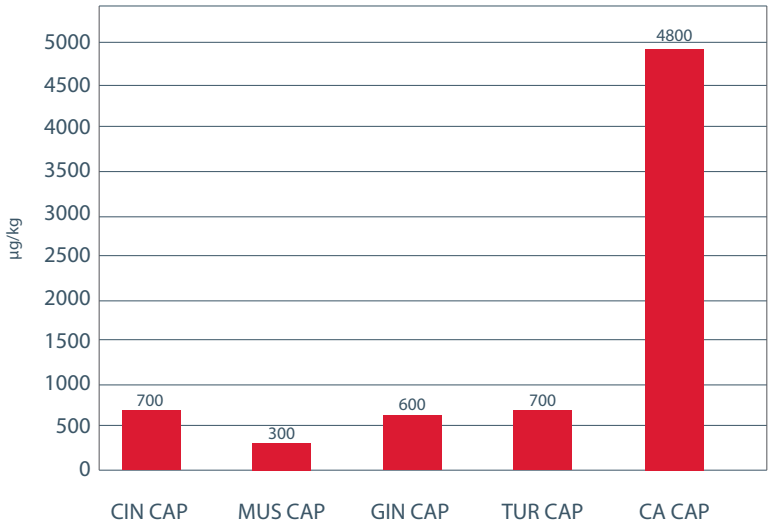

Figure 2. Lead Concentration in Spice and Calcium Supplements (µg/kg).

The suggested dosage for the various products was between 1-2 capsules of various weights. Dosages are listed in Table 1.

The concentrations measured in spice and calcium supplements were calculated to give a final concentration of lead for the stated dosages. For an adult there is a 5 µg limit per day. Several samples of nutraceuticals had more than 200% of an adults limit. For children these samples were 500% of potential daily limit for a child (Figure 4). In the case of the common ginger supplement often taken for nausea or illness, it would have been 125% of an adult limit and 310% of a child's daily Pb limit.

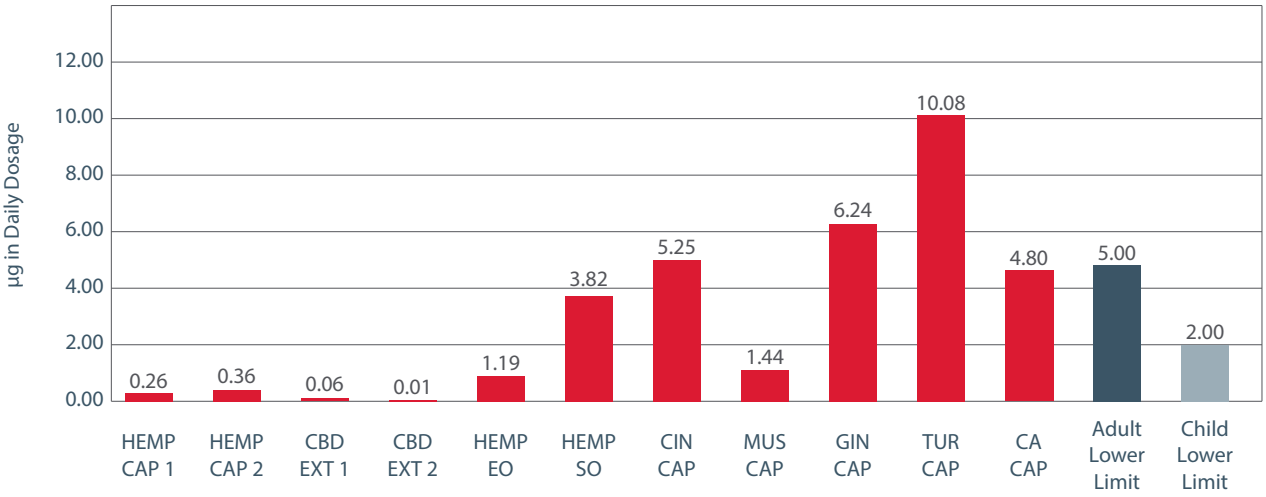

Figure 3. Total Exposure to Lead in a Daily Dosage of Hemp and Spice Supplements for Adult and Child.

Conclusions

Lead was the most prevalent heavy metal found in the products. The concern for these heavy metal concentrations comes from the dosage of these common nutraceutical products. The dosages suggested are fairly large and instructions are absent on dosage for children. There is possibly a misconception that the product is safe at all dosages due to its pretense of being a supplement or natural product. The limits imposed by organizations such as the AHPA, who are attempting to create limits for cannabis and other nutraceutical products, do not necessarily provide limits which are applicable for a child's exposure to these natural products. By using adult limits and calculating them against the body weight of a child, the exposure to heavy metals from these products can be potentially very high. Products that at first did not exceed the heavy metal limits for adults could then be seen as potentially hazardous to a child, especially a child with health concerns.

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