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The Elemental Composition of Hemp Flower: Sources of Elemental Impurities and Implications for Consumer Product Safety



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Reference

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ABSTRACT

The rapid growth of the cannabis market in the United States has led to increasing focus on the consumer product safety of the myriad of new products entering commerce. The cannabis plant is widely accepted to be an efficient accumulator of potentially toxic elements, and its resinous nature makes it well suited to accumulate surface contaminants. Unfortunately, limited data are currently available on the occurrence of many elements in consumer cannabis, and even fewer studies have examined surface adhered particulate matter. As part of this study, 26 elements (silver, aluminum, arsenic, barium, beryllium, calcium, cadmium, cobalt, chromium, copper, iron, mercury, potassium, magnesium, manganese, molybdenum, sodium, nickel, lead, antimony, selenium, thorium, thallium, uranium, vanadium, and zinc) were quantified in hemp produced for cannabidiol and commercially available hemp marketed for smoking. Additionally, surface adhered particulate matter was examined as a potential contributor of elemental impurities, and various methodologies were compared. Results confirm that hemp entering commerce in the United States contains a variety of elemental impurities and frequently contains adhered particulate matter such as soil minerals, agricultural additives, microplastics/textile fibers, and materials from harvesting/processing equipment. Consumer exposure potential for some elements such as copper was found to be high enough to warrant additional investigation as to the possible health effects and may justify additional oversight from regulators, who should consider expanding testing panels to a larger suite of elements than arsenic, cesium, mercury, and lead.

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Keywords

cannabis, heavy metals, foreign matter, exposure, copper

Introduction

The removal of industrial hemp, cannabis containing <0.3 % tetrahydrocannabinol (Δ^9 -THC) by weight, from Schedule I control by the Agricultural Improvement Act of 2018 (Farm Bill) has led to an increase in popularity and consumer use, creating a market for hemp-derived products focused on nonpsychoactive cannabinoids including cannabidiol (CBD).^{1,2} In addition to the 2018 Farm Bill legalizing hemp products, there has been an increase in the legalization of cannabis products containing THC across a multitude of states, including the State of Michigan. This challenge is then compounded by the fact that marijuana (cannabis containing >0.3 % tetrahydrocannabinol (Δ^9 -THC) by weight) currently maintains a Schedule I status federally, so there is no federal governing body whose jurisdiction encompasses the health and safety surrounding the use of these products in the United States. This has led to an inconsistent patchwork of state-level regulations based on limited scientific evidence, and the potential for unsafe consumer products to enter the marketplace.³

Cannabis is well known for its ability to accumulate contaminants during its life cycle—particularly through the soil and water in which it grows. ^{4,5} One group of contaminants of particular concern is the presence of potentially toxic elements, especially in cannabis products intended for inhalation. Although the availability of published studies on elemental accumulation by cannabis plants is still somewhat limited, a general consensus has emerged that cannabis is both tolerant of elevated heavy metals in soils and is an efficient accumulator of several potentially toxic elements such that it is a promising candidate for phytoremediation applications. ⁵ There remain, however, little publicly available data on the elemental composition of cannabis for many elements of interest. Additionally, the resinous nature of cannabis leads to a significant potential to accumulate surface contaminants. This may further increase consumer exposure, but to our knowledge, there is no literature documenting this potential route of accumulation.

There has been increased use of cannabinoids in edible and distillate forms, but the most common method of use is through inhalation by smoking.⁶ Elemental exposure by inhalation poses a long-term health risk of 5accumulation in the body.^{7,8} Various elements may be toxic, carcinogenic, or both, and can lead to the development of potentially serious health conditions.⁷ For example, chromium is carcinogenic, and human exposure may increase mortality and the incidence of lung, larynx, bladder, kidney, testicular, bone, and thyroid cancer,^{7,9} and inhalation of copper has been shown to cause pulmonary inflammation, altered gene expression, and cytotoxicity.^{10,11} Of the various routes of exposure, consuming cannabis through inhalation (smoking) likely poses the greatest risk because cannabis smoke has been shown to contain elevated concentrations of various potentially toxic elements,^{4,12} which may play a central role in carcinogenicity.¹³

Thus, there is a significant need for additional data on the elemental composition of cannabis, particularly with respect to cannabis that may be used medicinally by sensitive or immunocompromised populations. Here we present one of the first studies to document the elemental composition of hemp produced for its CBD content and intended for potential human consumption including quantification of silver, aluminum, arsenic, barium, beryllium, calcium, cadmium, cobalt, chromium, copper, iron, mercury, potassium, magnesium, manganese, molybdenum, sodium, nickel, lead, antimony, selenium, thorium, thallium, uranium, vanadium, and zinc. Additionally, we examine the potential of cannabis to acquire surface contaminants and briefly evaluate methods for analysis of surface contaminants. Finally, we estimate possible exposures to potentially toxic elements through inhalation when smoking.

Materials and Methods

SAMPLE SELECTION

The majority of the samples analyzed in this study came from the United States Department of Agriculture (USDA) Hemp Tribal Research Initiative for Michigan (TRIM) Study. Seventy three samples of hemp flower

from CBD producing verities (36 cultivars) were obtained from the 2020 field trials at three sites in Michigan (Chatham, Bay Mills, and Harbor Springs). Samples were harvested and dried prior to delivery to the laboratory and stored in paper bags prior to homogenization. The relatively large quantity of available biomass enabled homogenization of >10 g of sample prior to analysis.

Additional samples (n = 18) of commercially available CBD cultivars were obtained from various retailers. Packaging of consumer samples consisted of plastic bags, some of which were metallized. For commercial samples, individual flowers were homogenized due to limited sample quantity for analysis, with additional intact flowers from the same package retained for foreign matter/particle analysis.

INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY ANALYSIS

The elemental composition of hemp flower was quantified by inductively coupled plasma mass spectrometry (ICP-MS). Dry flower was homogenized with a metal free grinder (Distek Inc.) and microwave digested with 9:1 trace metal grade nitric acid/hydrochloric acid (HNO₃/HCl) (ARISTAR PLUS) using the manufacturer's recommended program for plant materials (CEM Mars 6). Each digest batch consisted of 18 samples, a duplicate, a matrix spike, and four digestion blanks. Following digestion, sample digests were transferred to 50-mL, acid-cleaned polypropylene centrifuge tubes and diluted with ultrapure water (18.2 M Ω) to a final volume of 50 mL.

ICP-MS analysis was performed with an Agilent 7800 and 4× aerosol dilution using EPA 6020, modified. ¹⁴ The instrument was tuned using the vendor-recommended autotune procedure. Internal standard solution containing 1 mg l⁻¹ ⁶Li, Sc, Ge, In, Tb, and Bi was added with an online mixing tee. Sample was aspirated using a concentric nebulizer (MicroMist U series) into a double pass spray chamber chilled to 2°C. Forward radio frequency power was set to 1,600 W and sampling depth to 10 mm to maximize sample decomposition and ionization of high first ionization potential elements (mercury, cadmium, etc.). A five-stage rinse cycle consisting of 2 % HNO₃/0.5% HCl (stages 1, 2, and 5), 5 % HCl (stage 3), and 1 % HCl (stage 4) was used to minimize carryover issues with long washout time elements (i.e., mercury). The use of high forward power and elevated concentrations of HCl necessitated the use of nickel-plated cones to minimize corrosion of the copper base.

Elements were quantified using the manufacturer-recommended isotope, with confirmation by additional isotopes when possible. Doubly charged interferences were corrected for as described in ASTM D8469-22, Standard Test Method for Analysis of Multiple Elements in Cannabis Matrices by Inductively Coupled Plasma Mass Spectrometry (ICP-MS).¹⁵ Lead was quantified as the sum of masses 206, 207, and 208. The samples provided from the TRIM study were analyzed in 2020 and 2021, whereas the commercially available hemp was analyzed in 2023, following the addition of antimony to our analytical method. Therefore, antimony is only reported for the commercial samples.

FOREIGN MATTER/PARTICLE ANALYSIS

Examination by stereomicroscopy was performed with either a Nikon SMZ1000N or an Olympus SZH-10 equipped with an apochromatic common main objective. The intact flower (1-3 g) was examined with a thorough screening of the whole flower surface at $20 \times$ magnification and then at higher magnification, up to $80 \times$, when potential foreign material was identified. Typical examination time for each sample was $\sim 30-60$ min.

Analysis of particulate material was performed by scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDS, JEOL JSM IT200LA) or laser-induced breakdown spectroscopy (LIBS, Keyence EA300). SEM-EDS analysis was performed on intact uncoated flower samples under low vacuum conditions (50 Pa) with an acceleration voltage of 13 kV using a segmented backscattered electron detector operating in composition mode to visually identify particles of interest. Elemental analysis was then performed using EDS using the integrated 30 mm² EDS detector. Element concentrations in identified particles were estimated using standardless ZAF (atomic number, absorbance, florescence) quantification in the instrument software. LIBS data were collected on intact flowers using an ultraviolet laser with automated elemental identification performed using three emission lines per element and standardless quantification from the instrument software.

EXPOSURE POTENTIAL ESTIMATES

Exposure potentials were estimated based on ICP-MS results using a maximum exposure model (i.e., the total elemental content was assumed to be potentially available for inhalation). Our methodology is similar to that employed by Zumbado et al. ¹⁶ and thus has similar limitations but was used here for several reasons: (1) because of the limitations of available data on actual elemental exposure for a broad suite of elements, we lacked a basis to estimate the fraction inhaled, (2) regulatory standards based on user consumption estimate typically employ this methodology, and (3) underestimating exposure may place consumers at elevated risk.

Exposure potential was calculated based on the elemental concentration of each sample using daily paper consumption via smoking (i.e., Daily Use) as follows:

$$\label{eq:paper_mass} Daily \; consumption \\ (g \; day^{-1}) \times \frac{Paper \; mass \; (g)}{Cannabis \; mass \; (g)} \times \frac{Element \; mass \; (g)}{Paper \; mass \; (g)}$$

Daily Use was estimated at both 2 g and 5 g per day for a heavy and very heavy smoker, respectively. Estimates based on 5 g per day consumption were considered to be conservative, as consumption rates for a daily smoker are likely to average \sim 1–2 g day⁻¹.¹⁷

Results and Discussion

ELEMENTAL COMPOSITION OF CANNABIS

The median and maximum dry weight elemental composition of hemp samples from the TRIM study (n = 73) are presented in **Table 1**. For the majority of elements, the maximum concentrations observed are within a factor of 2–3 of the median concentration. Sodium (15×), chromium (6×), silver (5×), and cadmium (8×), however, show a greater range, though it is unclear the degree to which this reflects genotypic/phenotypic variability in the plants, environmental conditions, or potential contamination from processing, etc.

The median and maximum dry weight elemental composition of the commercially available hemp samples (n = 18) are presented in **Table 2**. Typical elemental compositions are broadly similar to those found in the TRIM study. A statistical comparison (t-test or similar) was not performed because of concerns about nonrandom sampling (i.e., there is no certainty about how comparable the two data sets are or how well either data set represents "typical" hemp on the consumer market). Thus, the two data sets were analyzed separately. With these caveats in mind, however, both data sets show relatively little difference between TRIM and commercial samples suggesting that the maximum concentrations observed may be due to localized conditions possibly including excess metal in the growing medium, soil pH, soil composition, or a combination thereof.

TABLE 1Maximum and median concentrations of elements quantified in hemp samples from the USDA TRIM study

$\rm g~kg^{-1}$	Ca	Mg	Na	K	Fe	Al				
Max	20	6.3	0.130	27	0.35	0.109				
Median	13	4.5	0.009	21	0.21	0.050				
mg kg ⁻¹	Ве	V	Cr	Mn	Со	Ni	Cu	Zn	As	Se
Max	0.0045	0.22	0.9	247	0.068	0.66	24	101	0.10	0.14
Median	0.0017	0.10	0.2	142	0.038	0.27	14	65	0.03	0.05
mg kg ⁻¹	Mo	Ag	Cd	Ba	Hg	Tl	Pb	Th	U	
Max	2.6	0.091	0.527	16.6	0.008	0.077	0.181	0.025	0.005	
Median	1.0	0.017	0.060	6.6	0.004	0.023	0.083	0.011	0.003	

TABLE 2
Maximum and median concentrations of elements quantified in consumer cannabis (hemp) intended for smoking

$g kg^{-1}$	Ca	Mg	Na	K	Fe	Al				
Max	24	7.8	0.236	33	0.54	0.679				
Median	13	5.1	0.015	23	0.25	0.024				
mg kg ⁻¹	Be	V	Cr	Mn	Со	Ni	Cu	Zn	As	Se
Max	0.0140	1.00	0.7	318	0.271	1.75	28	165	0.41	0.24
Median	< 0.01	0.09	0.1	126	0.061	0.44	19	80	0.03	0.03
mg kg ⁻¹	Мо	Ag	Cd	Sb	Ва	Hg	Tl	Pb	Th	U
Max	5.0	0.033	1.329	0.06	46.6	0.013	0.186	2.105	0.125	0.019
Median	0.8	0.011	0.112	0.01	11.1	0.004	0.021	0.026	0.005	0.002

Biologically useful elements like copper, manganese, molybdenum, nickel, and zinc have limited concentration distributions among the samples compared with nonessential elements like cadmium, chromium, and lead, which have much broader concentration ranges (fig. 1). This suggests that the concentration of nutrient elements (including calcium, copper, iron, manganese, molybdenum, nickel, potassium, and zinc) may be controlled primary by plant uptake and homeostasis mechanisms. The copper, manganese, molybdenum, nickel, and zinc concentrations in our hemp flower most likely represent the range of required concentrations for proper function of the cannabis plant. The concentrations of these metals representing the necessary functional ranges are corroborated by comparing with other studies that show similar concentration ranges in cannabis flower for copper, manganese, and zinc from Ghana. Conversely, elements such as cadmium and lead likely enter plants through nonspecific transport mechanisms such as capillary action, leading to inconsistent concentrations at lower levels.

EXPOSURE POTENTIAL

Exposure potentials were calculated separately for samples from the TRIM study (Table 3) and for the commercially available samples (Table 4) and compared with acceptable exposures by inhalation for pharmaceuticals from United States Pharmacopeia (USP) 232 or International Council for Harmonisation (ICH) Q3D.^{20,21} Although several elements could potentially exceed these limits for very heavy smokers or based on the highest concentrations that were measured, the element in these data sets that is likely to exceed acceptable exposure

FIG. 1 Distributions of observed elemental concentrations in TRIM samples, n = 73 (T) and consumer samples, n = 18 (C) for (A) major elements and (B) trace elements.

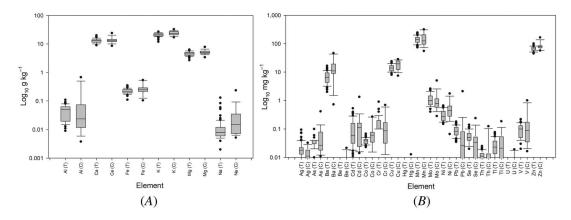


TABLE 3Exposure potential based on maximum and median concentrations of elements quantified in hemp samples from the USDA TRIM study

Exposure Potential	Parameter	Ag	As	Ba	Cd	Co	Cr	Cu	Hg	Mo	Ni	Pb	Tl	V
Reference value (µg day ⁻¹	7	2	300	2	3	3	30	1	10	5	5	8	1	
5 g day ⁻¹ consumption	Max	0.5	0.52	83	2.6	0.3	4.6	122	0.04	13.0	3.3	0.9	0.4	1.1
	Median	0.1	0.16	33	0.3	0.2	0.8	72	0.02	5.0	1.3	0.4	0.1	0.5
2 g day ⁻¹ consumption	Max	0.2	0.21	33	1.1	0.1	1.8	49	0.02	5.2	1.3	0.4	0.2	0.4
	Median	0.0	0.06	13	0.1	0.1	0.3	29	0.01	2.0	0.5	0.2	0.0	0.2

Note: Reference values are from USP 232 or ICH Q3D.

TABLE 4Exposure potential based on maximum and median concentrations of elements quantified in hemp samples from consumer cannabis (hemp) intended for smoking

Exposure Potential	Parameter	Ag	As	Ba	Cd	Co	Cr	Cu	Hg	Mo	Ni	Pb	Sb	Se	Tl	V
Reference value (μg day ⁻¹)			2	300	2	3	3	30	1	10	5	5	20	130	8	1
5 g day ⁻¹ consumption	Max	0.2	2.05	233	6.6	1.4	3.4	142	0.06	25.2	8.7	10.5	0.3	1.2	0.9	5.0
	Median	0.1	0.13	55	0.6	0.3	0.4	97	0.02	3.9	2.2	0.1	0.0	0.2	0.1	0.4
2 g day ⁻¹ consumption	Max	0.1	0.82	93	2.7	0.5	1.4	57	0.03	10.1	3.5	4.2	0.1	0.5	0.4	2.0
	Median	0.0	0.05	22	0.2	0.1	0.2	39	0.01	1.6	0.9	0.1	0.0	0.1	0.0	0.2

Note: Reference values are from USP 232 or ICH Q3D.

standards most often is copper. Both data sets show median copper exposures at (within experimental error) or in excess of the exposure limits for a 2 g d $^{-1}$ smoker. For all other elements, the exposure potential is <20 % of the limit for 2 g d $^{-1}$ smoker based on the median element content. If the copper concentrations quantified here are typical as suggested based on the similarity in the two disparate data sets, then it is plausible that copper could pose a general and poorly recognized inhalation risk to heavy cannabis smokers.

INDOOR VERSUS OUTDOOR PRODUCTION

It has been previously hypothesized that hemp grown outdoors could contain higher levels of potentially toxic elements due to the reduced ability to control growing conditions in outdoor agricultural fields.²² For the commercially available hemp samples in our data set, we were able to identify based on information from the distributor, nine of which were grown in an indoor environment. The median and maximum elemental contents of these were compared with the samples from the TRIM study (Table 5) to see if there were any obvious trends or

TABLE 5Comparison of maximum and median elemental concentrations from samples grown indoors (n = 9) and those grown outdoors in the TRIM study (n = 73)

$g\ kg^{-1}$	Ca	Mg	Na	K	Fe	Al				
Median indoor	13	5.1	0.015	23	0.25	0.023				
Median outdoor	13	4.5	0.009	21	0.21	0.050				
mg kg ⁻¹	Be	V	Cr	Mn	Со	Ni	Cu	Zn	As	Se
Median indoor	0.001	0.09	0.1	126	0.061	0.44	19	80	0.03	0.03
Median outdoor	0.002	0.10	0.2	142	0.038	0.27	14	65	0.03	0.05
mg kg ⁻¹	Мо	Ag	Cd	Ba	Hg	Tl	Pb	Th	U	
Median indoor	0.8	0.011	0.11	11.1	0.004	0.021	0.026	0.005	0.004	
Median outdoor	1.0	0.017	0.06	6.6	0.004	0.023	0.083	0.011	0.003	

differences that might support this hypothesis. The results do not suggest a general trend toward lower potentially toxic element concentrations in cannabis grown indoors, as the median concentrations of most elements vary by a factor of ~2 or less. One possible exception, however, is lead. Soil contamination for lead is widespread in the United States due to past use of tetraethyl lead in automotive fuel, contaminated agricultural amendments, and other anthropogenic sources. ^{23,24} It is plausible that the widespread recognition of lead's toxicity as well as near universal regulation of lead in legal consumer cannabis has resulted in indoor growers taking additional precautions against lead contamination. This hypothesis warrants further investigation by future studies using larger, more representative data sets and may be testable for currently regulated elements using data sets currently available to regulatory agencies.

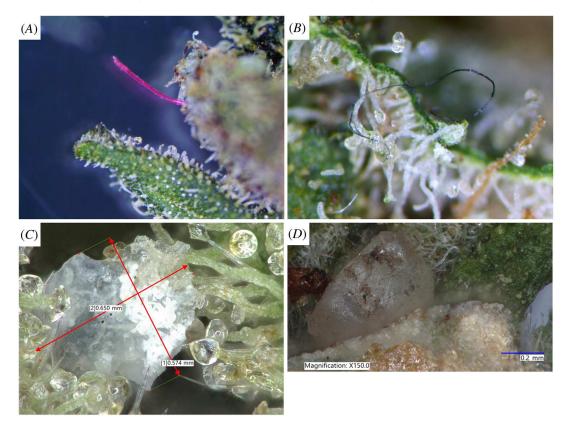
PARTICULATE MATTER CONTAMINATION

Evaluation of LIBS versus SEM-EDS for Use in Particle Analysis

For a subset of samples from the TRIM study and the commercial samples, surface contaminants were identified and analyzed with two goals: (1) evaluation of the suitability of SEM-EDS and optical microscopy paired with LIBS in comparison with traditional foreign matter screening with a stereomicroscope and (2) to determine if any particulate contaminants could be identified that might pose a risk to consumers.

With regard to the first goal, both techniques (SEM-EDS and LIBS) were found to be superior to stereomicroscopy for identification of observed particles, though each has distinct advantages and disadvantages. In the samples examined by stereomicroscopy (n > 60), the most common type of potential foreign matter that we could identify at magnifications between $20-80\times$ were potential synthetic fibers (possible microplastic fibers, fig. 2A

FIG. 2 Foreign matter in cannabis. (A) and (B) are typical potential microplastics identified by stereomicroscopy, (C) was identified as $CaCO_3$ by LIBS analysis, and (D) was identified as K_2SiO_3 by LIBS analysis.



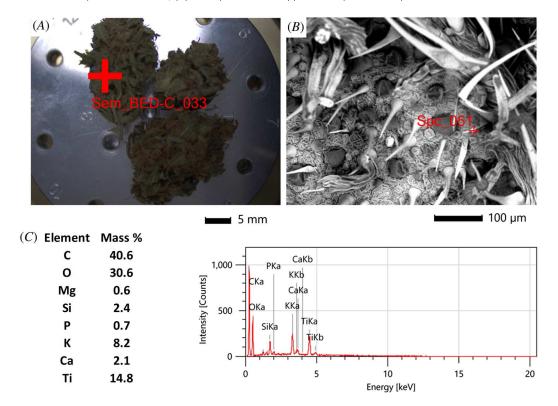
and 28). As both SEM-EDS and LIBS are elemental analysis methods, neither is definitive for distinguishing synthetic polymers from natural materials. The LIBS system consisted of an automatic focus stacking (z-stacking) function and a higher-maximum magnification (20× to 200×), which aided in visual recognition of particles. Elemental analysis could be performed in less than a minute (though it required the microscope to change to a second objective, which was automated). However, it was also destructive on a microscopic scale because the laser ablates the particle as part of the analysis, precluding any further analysis.

In the case of SEM-EDS, the technique has several advantages for detecting particles in cannabis flowers. The inherently higher depth of field and resolution compared with optical microscopy is very useful in the case of highly topographic specimens such as cannabis flower, especially when a backscattered electron detector is used to provide compositional contrast (higher atomic number elements are brighter). This aids rapid identification of particles of interest, particularly when the particles are small or have poor visual contrast with the underlying plant material. EDS analysis can then be used to analyze the elemental composition nondestructively. The primary drawbacks of SEM-EDS versus the LIBS instrument are the somewhat greater operator skill required, modestly higher cost of acquisition, facility requirements, and the loss of color information. Ultimately, both techniques were determined to be suitable, and each identified particles based on elemental analysis that could not be identified visually.

Particle Contaminants in Cannabis

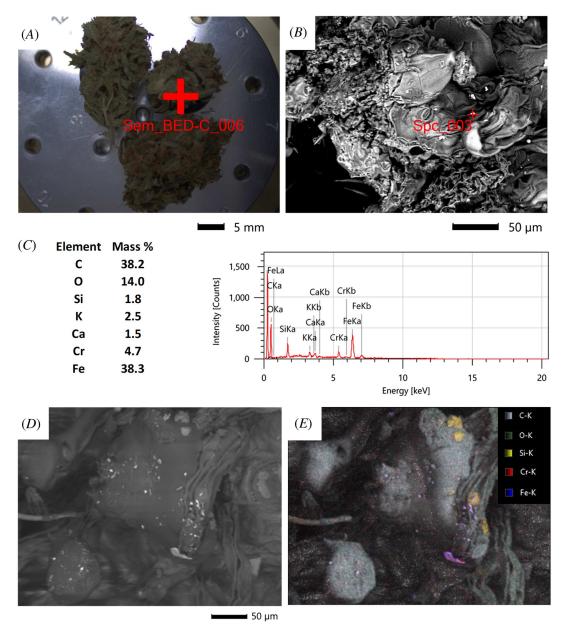
LIBS analysis of two samples from the TRIM study identified particles of calcium carbonate (CaCO₃, \sim 600 µm) and potassium silicate (K₂SiO₃, \sim 700 µm), which are both believed to be the result of agricultural soil amendment (fig. 2C and 2D). Although neither of these is likely to pose a human health risk, it does suggest that cannabis may

FIG. 3 SEM-EDS analysis of particulate contaminants. (A) Intact cannabis flower, (B) backscatter SEM image showing adhered particulate matter, (C) EDS spectrum and approximate particle composition.



retain material that it comes in contact with during production and processing. Detailed examination of six further samples by SEM-EDS (analysis time, 1–2 hours per sample) identified the presence of adhered soil/terrigenous particles (containing silicon, oxygen, titanium, aluminum, iron, calcium, etc.; fig. 3) and on one sample of commercial cannabis, a number of iron/chromium containing particles \sim 2–25 μ m that appear to be stainless steel shavings (fig. 4). Stainless steel shavings, silver chloride (AgCl) residue (possibly from feminization), and fly ash (spherical silicates) were also observed on samples from the TRIM Study analyzed previously by SEM-EDS at

FIG. 4 SEM-EDS analysis of particulate contaminants. (A) Intact cannabis flower, (B) backscatter SEM image showing adhered particulate matter, (C) EDS spectrum and approximate particle composition, (D) backscatter SEM image showing additional particles in an adjacent field of view, and (E) EDS map showing colocalization of iron and chromium.



JEOL USA (Peabody Massachusetts). In the case of silver and especially chromium/stainless steel residues, these particles contain elements of potential human health interest, with some being of respirable size ($<10 \mu m$; World Health Organization, 2021).²⁵ Particles were distributed in a highly heterogenous fashion, making potential estimates of total mass contribution of elements of interest difficult in practice, but further suggesting that control of potential surface contamination should be considered in the design of cannabis handling and processing procedures, especially when the final product is intended for smoking or inhalation.

Conclusions

With the increase in the number of states legalizing both the medicinal and recreational use of cannabis, the possible federal rescheduling of marijuana, and the federal legalization of hemp, establishing uniform and reasonable exposure limits is necessary for consumer safety. States that have legalized cannabis typically regulate the "Big Four" contaminants of arsenic, cadmium, mercury, and lead. However, other metals, such as copper, are inconsistently tested for, and the standards among states that do require testing are inconsistent. Our data suggest that the bioaccumulation of macro- and micronutrient elements in cannabis may potentially be controlled primarily through homeostasis mechanisms as opposed to contaminated growing conditions, but additional work will be necessary to elucidate these mechanisms, and genetic and phenotypic variations are likely to exist.

Consistent regulation of a broader suite of elements is justified based on the known potential of cannabis to bioaccumulate potentially toxic elements and its potential to acquire surface contamination in production and handling. States commonly use either 5 or 10 g d^{-1} exposures to set limits on user exposure, but the rationale for selecting a particular value is often unclear. A consistent regulatory approach and reducing barriers to academic research on cannabis would improve consumer protection and aid producers in developing and adopting best practices for minimizing product contamination.

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