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Investigation of lead and cadmium in counterfeit cigarettes seized in the United States

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ABSTRACT

Information of toxic elements such as lead (Pb) and cadmium (Cd) in counterfeit cigarettes offers insight on the potential public health impact of consuming counterfeit cigarettes and the technology used by counterfeiters in the illicit cigarette trade. In this study, the concentration of Pb and Cd in twenty-three packs of counterfeit cigarettes seized in the US by various law enforcement agencies were evaluated and compared with their genuine equivalents using microwave digestion followed by inductively coupled plasma – mass spectrometry (ICP-MS) analysis. Both Pb and Cd concentration in counterfeit cigarettes were markedly higher than those in their genuine equivalents, and exhibited greater sample to sample variability. The average Pb and Cd mass fraction values in counterfeit cigarettes were (5.13 ± 2.50) mg/kg (n = 23) and (5.13 ± 1.95) mg/kg (n = 23) respectively, compared with (0.59 ± 0.08) mg/kg (n = 9) and (1.08 ± 0.08) mg/kg (n = 9) respectively in the genuine equivalents. Results suggest that counterfeit cigarettes may impose higher risks to public health. Studying these toxic elements could provide important information regarding the illicit trade, including the level of organization among counterfeiters, who broker between availability of supplies and consumer demand for a cheaper product that is assumed to be genuine.

1. Introduction

It has long been known that smoking has an adverse impact on human health. The substances inhaled during smoking include both organic and inorganic chemical species. The health hazards of organic components such as nicotine, polycyclic aromatic hydrocarbons, and nitroamines have been well investigated (Hecht, 1999). In comparison, available information on the health effects of toxic inorganic components is relatively limited. Nevertheless, inhalation of heavy metals from cigarette smoke, in particular lead (Pb) and cadmium (Cd), has been correlated with potential health risks (Pappas et al., 2007).

The toxicity of Cd and Pb to humans has been well studied and documented. Cadmium is a Group I and Pb is a Group IIA carcinogen with links to various cancers (Pappas, 2011). Exposure to Pb has also been attributed to non-cancer problems such as respiratory and cardiovascular diseases (Pappas, 2011). Cadmium and Pb enter the human body via various pathways and exposure to cigarette smoke is one of them. Intake of Cd and Pb from smoke can occur actively through inhaling mainstream smoke, or passively from exposure to side stream smoke in an ambient environment. This means smoking not only directly affects the smokers, but also indirectly affects non-smokers close to the source such as children of a smoking parent. For example, results from the National Health and Nutrition Examination Survey (NHANES) which was conducted between 1999 and 2004 (Ritcher et al., 2009) found that smokers' urine had higher Cd and Pb levels than non-smokers. Urinary Pb levels among adult non-smokers with high second-hand smoke exposure were equal to the urinary Pb levels of adult smokers. The highest urinary Pb levels of those exposed to second-hand smoke were found in children. Older smokers had urinary Cd levels signaling the potential for Cd-related toxicity.

The presence of counterfeit tobacco products on the illegal cigarette market has made the assessment of the health impact of toxic heavy metals quite challenging. Compared with their genuine equivalents, the origins of tobacco used in counterfeit cigarettes in most cases are unknown, and the manufacturing process lacks regulation and quality control. Unsurprisingly, elevated concentrations of heavy elements have thus been found in counterfeit tobacco products, with Pb and Cd identified as two leading toxic elements (Pappas et al., 2007; Stephens et al., 2005; Swami et al., 2009). Greater sample

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to sample variability was also observed in the counterfeit tobacco products that were investigated.

Trace elements in tobacco leaves can be measured using such techniques as *atomic absorption spectroscopy* (AAS) (Ajab et al., 2008; Golia et al., 2007), *inductively coupled plasma optical emission spectroscopy* (ICP-OES), *inductively coupled plasma mass spectrometry* (ICP-MS) (Swami et al., 2009), and polarized energy dispersive X-ray fluorescence (PEDXRF) (Stephens et al., 2005). Among the available analytical techniques, solution-based ICP-MS is an attractive choice due to its speed, superior sensitivity and wide linear range. Microwave digestion is now a well-accepted method for dissolving solid samples for subsequent elemental analysis. Compared with conventional hot plate digestion, microwave digestion is faster and more user friendly. Loss of volatile components in sample solutions could be effectively prevented and contamination to both sample solutions and the ambient environment could be significantly reduced by using the microwave technique.

In this study, Pb and Cd concentrations in counterfeit cigarettes seized on the East Coast of the US and in California by various law enforcement agencies were investigated using microwave digestion followed by ICP-MS analysis. Twenty-three counterfeit samples from seven brands were analyzed. As a comparison, the concentration of Pb and Cd in commercially available genuine equivalent cigarettes were also determined.

The purpose of this study is threefold. First, it is not assumed that compared with previous research published between 2005 and 2009, counterfeit cigarettes still differ from genuine cigarettes in trace metal concentrations. High levels of trace metals have been linked to the quality of soil used for tobacco cultivation rather than the process of manufacturing cigarettes (Stephens et al., 2005). It is possible that cigarette counterfeiters have turned to higher-grade tobacco with accompanying lower levels of trace metals. This study seeks to determine whether there are indications that such a shift has taken place. Second, this study seeks to gauge the present public health implications of counterfeit cigarettes specifically with regard to the levels of Pb and Cd. Third, this study places these findings in a criminological context and provides a more nuanced understanding of the logistics involved in the illegal cigarette market. More specifically, we examine the decisions that criminals make in terms of how they produce counterfeit cigarettes and service their customers.

2. Materials and methods

2.1. Cigarette samples

Counterfeit cigarettes were confiscated in multiple seizures and were provided by local, state and federal law enforcement agencies in New York, Virginia, Maryland, Washington DC and California. Twenty-three counterfeit cigarette samples under seven different brand names were examined. Nine commercially available genuine cigarette samples manufactured in the US were analyzed as a comparison. Four of these genuine cigarette samples had corresponding counterfeit brands analyzed in this study. All genuine cigarettes were purchased from cigarette retailers in New Jersey in November 2012.

All cigarettes were kept in their sealed original package at room temperature until analyzed. For each sample, two cigarettes, labeled as sample 1 and sample 2, were randomly picked from a pack and processed.

2.2. Reagents and standards

The elemental standards used were diluted from the National Institute of Standards and Technology (NIST), Gaithersburg, MD Standard Reference Material (SRM) 3128 Lead (Pb) Standard Solution (9.995 ± 0.014) mg/g and SRM 3108 Cadmium (Cd) Standard Solution (10.005 ± 0.019) mg/g. Samples were digested and diluted using nitric acid (Optima grade) purchased from Fisher (Pittsburgh, PA, USA). Deionized water (18 MΩ cm) was used throughout the entire work. NIST SRM 1573a Tomato Leaves was analyzed along with the samples for quality assurance.

2.3. Sample preparation and instrument analysis

A CEM MARSXpress microwave sample digestion system with a 40-vessel rack (CEM Corporation, Matthews, NC, USA) was used to dissolve tobacco tissues. The temperature of each individual digestion vessel was monitored during digestion using an IR-based temperature system.

Table 1
Parameters used to measure Cd and Pb in cigarette samples.

Element	Mass (amu)	Integration time/point (s)	Read time/mass (s)	Number of runs
Cd	111	0.20	12	2
Pb	208	0.20	12	2
Y	89	0.20	12	2

All cigarette samples and NIST SRM 1573a Tomato Leaves were dried in a desiccator at room temperature (approximately 22 °C) for at least 120 h over fresh anhydrous magnesium perchlorate before processing. For the dried cigarettes, the tobacco component was separated from its wrapping paper and filter. Approximately 0.5 g aliquots of the tobacco component from each dried sample or SRM 1573a were transferred to a 55 mL Teflon microwave digestion vessel. Ten milliliters of concentrated Optima grade HNO_3 were added to each vessel and the closed vessels were loaded symmetrically into the microwave digestion rack. The samples were digested according to the following program: power 1600 W, power setting 100%, ramp time 20 min, temperature 200 °C, hold time 15 min. In order to check for background interference, three sample preparation blanks were processed in the same manner as the samples for every digestion batch.

After digestion, sample vessels were cooled to room temperature and carefully vented in a fume hood. The digestate was quantitatively transferred to a 60 mL Nalgene bottle and diluted to approximately 50 mL using 1.5% (volume fraction) HNO_3 (Optima). The exact weight of the digestate solution was measured using an analytical balance (Mettler AT261 Delta Range). Each digestate was then further diluted five times and spiked with 4 µg/L yttrium (Y) as an internal standard prior to ICP-MS analysis to improve precision of the instrumental measurements. The Y solution was prepared from SRM 3167a Lot # 120314, Yttrium (Y) Standard Solution using 1.5% volume fraction HNO_3.

Lead and Cd in all sample solutions were determined using an Agilent 7500cs ICP-MS (Agilent, Palo Alto, CA, USA) equipped with ChemStation software. SRM 1573a Tomato Leaves and the sample preparation blank were frequently analyzed along with tobacco samples throughout the entire work. The analytes in the solutions were measured according to the parameters in Table 1. The argon flow on the ICP-MS was set to 15 L/min, the auxiliary flow to 0.8 L/min, and the nebulizer flow to 1 L/min. The radio-frequency (RF) power was set to 1500 W and H_2 was used as a collision gas to minimize polyatomic interferences.

3. Results

Table 2 summarizes the characteristics of counterfeit cigarettes analyzed in the study. The majority of these 23 samples were from US brands. For the geographic origins of these samples, eleven were identified from China, one was from Paraguay, and the origins of the remaining samples were unknown.

The quality of packaging and of individual cigarettes of the counterfeit products was found to vary under preliminary visual inspection. Some were relatively easy to identify as counterfeit due to the use of inferior wrapping paper and poor printing quality; however, to the untrained eye, most counterfeit products were virtually indistinguishable from their genuine equivalents.

Tax stamps on sample packaging were examined. Examining tax stamps is a straightforward and commonly used method to identify contraband cigarettes, specifically cigarettes bootlegged from

Table 2
Characteristics of counterfeit samples (n = 23).

Characteristic	%	n
Brand		
United States	91%	21
International	8%	2
Geographic origin		
China	48%	11
Paraguay	4%	1
Unknown	48%	11
Tax stamps		
No tax stamp	83%	19
Counterfeit[a]	17%	4

[a] Includes Virginia, New Jersey, New York State/New York City, and Maryland.

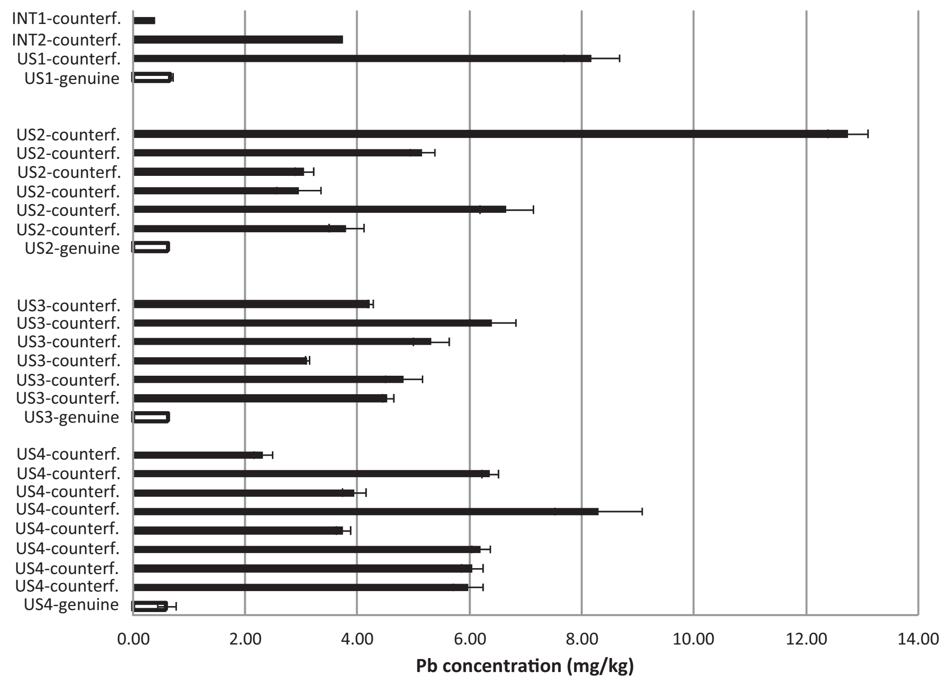

Fig. 1. Comparison of lead mass fraction found in genuine and counterfeit cigarettes. The clear columns represent genuine samples, and the solid columns are counterfeit samples. Error bars represent one standard deviation. (INT1: international brand; INT2: international brand; US1: US brand; US2: US brand; US3: US brand; US4: US brand.)

low-tax jurisdictions to high-tax jurisdictions (Chernick and Merriman, 2013; Kurti et al., 2013). In contrast, counterfeit cigarettes are not commonly marketed with taxes paid in either low-tax or high-tax jurisdictions. Where tax stamps appear on packs of counterfeit cigarettes, these tax stamps can be expected to be counterfeit as well. While tax stamps were not found in most counterfeit samples involved in this study, four counterfeit packages did bear presumably counterfeit tax stamps from New Jersey, Virginia, New York City and Maryland.

The Pb mass fraction values in counterfeit samples ranged from 0.40 mg/kg to 12.74 mg/kg with an average mass fraction value of (5.13 ± 2.50) mg/kg (n = 23). For the genuine equivalent samples, the range was from 0.42 mg/kg to 0.67 mg/kg with an average of (0.59 ± 0.08) mg/kg (n = 9). The uncertainties represent one standard deviation. Comparing results obtained from these two groups of cigarettes, except for a counterfeit cigarette from Paraguay with (0.40 ± 0.28) mg/kg, Pb in all other counterfeit samples was significantly higher, ranging from approximately 4 to 22 times higher than their genuine equivalents. On average, Pb in counterfeit cigarettes was about 9 times higher. The sample with the highest Pb concentration was a counterfeit US brand, which originated from China. Fig. 1 illustrates the Pb concentration in counterfeit cigarettes among different brands compared with their genuine equivalents. The method detection limit (MDL) for Pb was estimated to be 0.02 mg/kg and the limit of quantification (LOQ) was estimated to be 0.08 mg/kg. The MDL, estimated as three times the standard deviation of the method procedural blanks, is the level at which the analyte can be seen above the signal of the blanks. The LOQ, estimated as ten times the standard deviation of the method procedural blanks, is the minimum level at which mass fractions of an analyte can be accurately measured. Two method procedural blanks were run before and after each analysis.

The mass fraction values of Cd in counterfeit samples ranged from 0.73 mg/kg to 10.39 mg/kg with an average mass fraction value of (5.13 ± 1.95) mg/kg (n = 23). For the genuine equivalent samples, the range was from 0.90 mg/kg to 1.17 mg/kg with an average of (1.08 ± 0.08) mg/kg (n = 9). Uncertainties represent one standard deviation. This is the same pattern as obtained for the Pb mass fraction values; except for a counterfeit cigarette from Paraguay (0.73 ± 0.07) mg/kg, all other counterfeit samples exhibited significantly higher Cd mass fraction levels, ranging from approximately 3 to 10 times more Cd than the genuine equivalent samples. On average, Cd in counterfeit cigarettes was about five times higher than that of their genuine equivalents. The sample with the highest Cd content was a counterfeit cigarette from China. Fig. 2 shows Cd distribution in counterfeit cigarette samples compared with their genuine equivalents. The MDL for Cd was estimated to be 0.06 mg/kg and the LOQ was estimated to be 0.2 mg/kg. The MDL and LOQ were calculated in the same manner as for the Pb. Two or three method procedural blanks were run before and after each analysis.

The determined Pb mass fraction value for SRM 1573a was (0.605 ± 0.020) mg/kg (n = 10) and the determined Cd mass fraction value was (1.48 ± 0.02) mg/kg (n = 10). The uncertainties for Pb and Cd in SRM 1573a represent one standard deviation. The certified mass fraction value for Cd is (1.52 ± 0.04) mg/kg. There is no certified mass fraction value for Pb in SRM 1573a, but our result agreed with the Pb value reported in literature obtained using two methods: (0.599 ± 0.010) mg/kg and (0.600 ± 0.015) mg/kg (Lin and Jiang, 2013).

4. Discussion

4.1. Pb and Cd in counterfeit cigarettes

Elevated Pb and Cd values were observed in 22 out of 23 investigated counterfeit cigarette samples and are markedly higher than in comparable genuine brands. Such findings agreed with results obtained in previous studies (Stephens et al., 2005; Swami et al., 2009). No significant change of Pb and Cd distribution pattern was observed when comparing data reported in the above mentioned studies.

High Pb and Cd concentrations found in counterfeit cigarettes are most likely attributed to the unregulated and uncontrolled growing environment for tobacco. The tobacco plant is known as

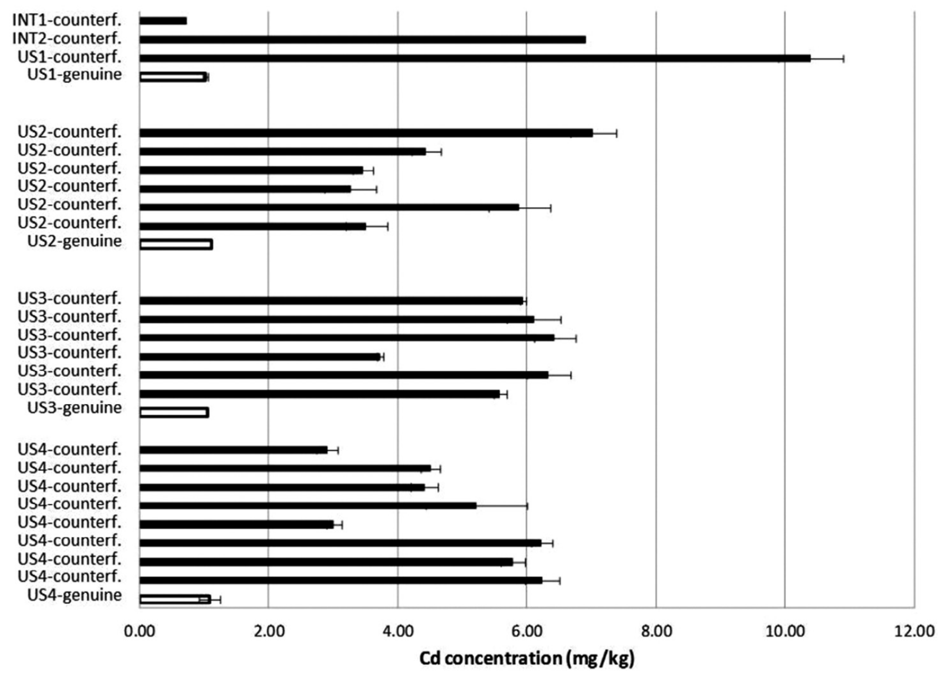

Fig. 2. Comparison of cadmium concentration found in genuine and counterfeit cigarettes. The clear columns represent genuine samples, and the solid columns are counterfeit samples. Error bars represent one standard deviation. (INT1: international brand; INT2: international brand; US1: US brand; US2: US brand; US3: US brand; US4: US brand.)

an effective accumulator of heavy metals from the soil (Tsadilas, 2000). Tobacco plants grown in soil with elevated Pb and Cd concentrations exhibited correspondingly higher levels of these elements in plant tissue. Soil pH plays an important role in heavy metal enrichment as well. Tobacco plants are not intentionally grown on contaminated soil; however, it has been found that growing tobacco on strongly acidic soil may enrich Cd by as much as fivefold (Mulchi et al., 1987). Major manufacturers began avoiding these tobacco crops and, as a result, it may be that counterfeiters began using these readily available crops. A three-year survey conducted in Greece showed that soil pH appeared to be the most important soil factor that influenced the concentration of Pb and Cd in tobacco produced (Golia et al., 2007). In addition, over-application of phosphate and/or nitrate fertilizers also contributes to trace element enrichment in counterfeit tobacco products (Stephens et al., 2005).

Considering environmental factors that affect tobacco quality, major manufacturers of genuine brands control the quality of tobacco used in their products. As a result, the contents of heavy metal in genuine cigarettes are stable. In this study, we found that although under different brand names, variation of Pb and Cd concentrations within genuine products was less than 14% and 2% respectively, suggesting that these two elements, especially cadmium, were consistently controlled during tobacco growing and cigarette manufacturing. However, on the contrary, wide concentration variability of Pb and Cd was observed in counterfeit cigarettes in both this and a previous study (Stephens et al., 2005).

Geographic origin is a factor of concern when studying heavy metal enrichment in counterfeit cigarettes. Although most counterfeit cigarettes exhibited higher levels of Pb and Cd than genuine brands, there were exceptions. In this work, we found a counterfeit international brand originating from Paraguay exhibiting a Pb and Cd concentration profile comparable with genuine commercial brands. Paraguay is a landlocked agricultural country in South America. Potentially the soil for tobacco growing is less contaminated from industrial activities. The samples with the highest Pb and Cd were counterfeited in China, which has more advanced industry and serious environmental problems. In addition, the heavy use of animal waste and excessive use of phosphate fertilizers in China are significant contributors to toxic metal concentrations in crops (Cheng, 2003).

Variation of Pb and Cd concentrations was not only observed in counterfeit cigarettes, but also in genuine cigarettes originating from different countries, showing the effect of geographical factors on heavy metal distribution in tobacco products. For example, the following is a list of mean Pb and Cd mass fraction values, respectively, found in genuine cigarettes from the following countries: UK, Pb values were (0.7 ± 0.2) mg/kg $(n = 8)$ and Cd values were (0.6 ± 0.1) mg/kg $(n = 8)$ (Stephens et al., 2005); from Brazil, Pb values were (0.27 ± 0.054) µg/g and Cd values (0.65 ± 0.091) µg/g (Viana et al., 2011); and from Turkey, Pb values were 3.7 µg/g and Cd values 1.1 µg/g (Duran et al., 2012).

4.2. Counterfeit cigarettes and implications for public health

Markedly high Pb and Cd concentrations commonly found in counterfeit cigarettes make using them a source of greater exposure to heavy metals than genuine brands. The quantity of heavy metals delivered to the lung through smoking positively correlated to the initial concentrations of the metals in tobacco leaves (Wu et al., 1997). It was found that mainstream smoke levels of Pb and Cd were far greater for counterfeit than for the authentic brands, in some cases by an order of magnitude (Pappas et al., 2007). Inhaling smoke generated from combustion of counterfeit cigarettes therefore is more likely to result in additional health risks from exposure to higher levels of Pb and Cd. Moreover, Cd in cigarette smoke is more bioavailable to the human body than Cd taken from other pathways such as from food or water. Forty percent to 60% of the Cd inhaled in smoke is absorbed into the bloodstream as opposed to 5–10% absorbed through foods, and additional cadmium is retained in lungs (Pappas, 2011; Suwazono et al., 2009). Although notlimited to heavy metals, a survey conducted Australia-wide showed that smokers of illicit tobacco report significantly worse health than other smokers (Aitken et al., 2009).

Children are especially vulnerable to heavy metals in tobacco smoke. The health risk associated with Pb and Cd is not only related to the amount of intake but also to the small body mass of children. In addition, children are generally exposed to cigarette smoke involuntarily. Second-hand smoke has been potentially attributed to increased blood Pb levels in US children, and inhalation has been identified as a major pathway of exposure (Apostolou et al., 2012). Blood Pb levels were found to be 14% and 24% higher in children who lived with one or more smokers, than in children living with non-smokers. Reducing exposure to second-hand smoke was suggested as a method to lower blood Pb levels in children.

4.3. Counterfeit cigarettes and implications for research on the illegal trade

This study confirms previous research suggesting that counterfeit cigarettes pose a greater health risk than genuine cigarettes. Accordingly, it can be assumed that the greater the market share of counterfeit cigarettes, the greater the public health implications of the illegal cigarette trade. Given that this study was drawn on the convenience of samples of known counterfeit cigarettes seized by law enforcement agencies, no inferences can be drawn on the prevalence of counterfeit cigarettes in the US tobacco industry. Estimates cited in the press place the share of counterfeit cigarettes in New York City, arguably the largest local cigarette black market in the country, at up to 2% of all packs sold in New York City (Crudele, 2012).

The differences in the concentrations of Pb and Cd between genuine cigarettes and counterfeit cigarettes originating from China could be used to gauge the share of Chinese counterfeit cigarettes in the US provided representative samples of cigarettes can be collected. In light of the minimal differences in Pb and Cd levels between genuine cigarettes and the analyzed sample of counterfeit cigarettes originating from Paraguay, more counterfeit cigarettes from Paraguay would need to be measured to see if this was an anomaly. If indeed Pb and Cd levels in counterfeit cigarettes originating from Paraguay are minimally different than those of genuine brands, then information about counterfeit samples from Paraguay in future studies would need to include a more precise estimation of the Pb and Cd levels to determine the overall prevalence of counterfeit cigarettes on the US market that come from Paraguay.

Studying the chemical makeup of counterfeit cigarettes provides a more nuanced understanding of the illicit trade including the level of organization among counterfeiters, who broker between availability of supplies (i.e., premium tobacco leaves, filter tips, cigarette paper, etc.) and consumer demand for a cheaper product that is assumed to be genuine. Examining the market for changes in the chemistry of counterfeit cigarettes also provides greater insight on the availability of technology to produce cigarettes and the source of tobacco leaves. So far, research on examining the prevalence of counterfeit cigarettes has predominantly relied on examining tax stamps with the assistance of law enforcement personnel (Kurti et al., 2013). However, tax stamps may not be an accurate predictor of counterfeit tobacco. In fact, counterfeit tax stamps may be affixed onto genuine products to conceal the source of purchase (e.g., counterfeit NYC/NYS tax stamps affixed on a pack of cigarettes bootlegged from North Carolina). Future research should obtain samples of cigarettes from consumers in geographically defined regions (where counterfeit markets are theoretically and empirically more likely to appear) and chemically test the samples against markers of counterfeit products, which this study and others find to be elevated levels of Pb and Cd.

NIST disclaimer

Certain commercial equipment, instruments, materials, or entities are identified in this paper to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply the materials or equipment identified are necessarily the best available for the purpose.

Conflict of interest

The authors declare that there are no conflicts of interest.

Transparency document

The Transparency document associated with this article can be found in the online version.

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