

## CADMIUM LEVEL IN COCOA POWDER AND CHOCOLATE AND THEIR CONFORMITY TO NATIONAL AND INTERNATIONAL REGULATIONS

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**ABSTRACT** - Contamination of heavy metals in human diets is a topic of concern in many countries around the world since high concentration of heavy metals may cause health problems in human. Cadmium is a toxic heavy metal. It is persistent in the soil and its bioavailability changes depending on the properties of the soil. Cadmium is absorbed and bioaccumulated by cocoa trees, resulting in unacceptably high levels of cadmium in cocoa beans. The Joint FAO/WHO Expert Committee on Food Additives (JECFA) in their recent exposure assessment of cadmium in all food sources concludes that cocoa products are not a major contributor of dietary cadmium. Malaysia currently regulates cadmium for cocoa and cocoa products at 1mg/kg. The European Union implement a new regulation for chocolate and cocoa powder starting January 2019. Codex has adopted maximum residue limits for two categories of chocolate and in the process for the adoption of maximum residue limits for cocoa powder and two more chocolate categories. From 2017 to 2020, 95 samples of cocoa powder were analysed. The cadmium level in these samples ranged between 0.05-1.61 mg/kg with an average of 0.47 mg/kg. Based on 81 samples of milk chocolate, the cadmium level ranged between 0.00-0.22 mg/kg with average of 0.05 mg/kg. Meanwhile cadmium content from 72 samples of dark chocolate ranged from 0.02-0.36 mg/kg with average of 0.10 mg/kg.

**Key words:** Cadmium level, cocoa powder, chocolate, national regulation, international regulations

### INTRODUCTION

Cadmium is a heavy metal that predominantly enters the environment through human activities such as ores processing, burning fuels, and waste. Agricultural activities that contribute to cadmium contamination in soil includes phosphate and sewage-containing fertilizers applications. Volcanic activity, marine shale soil and erosion or sea-salt aerosols also contribute to cadmium contamination in soil (CX/CF 21/14/7).

The half life of cadmium in soil is estimated to vary between 15 to 1100 years. Cadmium is absorbed and bioaccumulated by cocoa trees (*Theobroma cacao*) which in some cases results in unacceptably high levels in cocoa beans. Data showed that cocoa beans from Latin American and Caribbean region usually contains higher level of cadmium compared to cocoa beans from other regions (CX/CF 21/14/7).

The Joint FAO/WHO Expert Committee on Food Additives (JECFA) in their 91<sup>st</sup> assessment on cadmium (JECFA91), reported that cocoa products contributed only 0.1-9.4% of dietary cadmium exposure across all cluster diets. The

assessment also identified the main sources of dietary cadmium exposure as cereals, cereal-based products, vegetables, fish and seafood especially molluscs.

On the international level, Codex are in the process of setting maximum level for cadmium, European Union (EU) has implemented maximum level for chocolate and cocoa powder since January 2019. Malaysia has established a maximum permissible level for cocoa and cocoa products of 1 mg/kg as described in Schedule 14<sup>th</sup> Malaysian Food Act (1983) and Regulation 1985 (Food Regulation, 2010).

The proposed and adopted maximum level for four categories of chocolates proposed by Codex are illustrated in Table 1 (CX/CF 21/14/6). The categories proposed by Codex are similar to the European Union regulation in term of classification based on total cocoa solid content on dry matter basis. However, Codex proposed extra chocolate categories. The additional categories are chocolate containing or declaring more or equal to 50% but less than 70% total cocoa solids on dry matter basis; and chocolate containing or declaring more or equal to 70% total cocoa solids on dry matter basis.

Table 1: Codex maximum level for chocolate which has been adopted and under discussion

Commodity/Product name	Maximum level (ML) (mg/kg)	Status (after CCCF14 meeting-3-13 May 2021)
Chocolates containing or declaring < 30% total cocoa solids on a dry matter basis	0.3	Forward to CAC44 at Step 8 (ML 0.3mg/kg)
Chocolate containing or declaring ≥30% to < 50% total cocoa solids on a dry matter basis	0.7	Forwarded to CAC44 at Step 5/8 (ML 0.7mg/kg)
Chocolate containing or declaring ≥50% to < 70% total cocoa solids on a dry matter basis	0.8	Adopted
Chocolates containing or declaring ≥70% total cocoa solids on a dry matter basis	0.9	Adopted

The discussion on maximum level for cocoa powder by Codex Committee on Contaminant at its 14th meeting (CCCF14) decided to postpone any decision for one year and more data will be collected (Rep21/CF). The level proposed by the electronic working group was 2.0-3.0 mg/kg based on analysis of GEMS Food data and 1.3-1.5 mg/kg based on proportionality approach (based on the previously adopted level for chocolate) (Table 2). The proposed maximum level of 2.0 mg/kg will account for rejection rates of 5.39% (worldwide) and 13.42% for Latin America and the Caribbean.

Lower maximum levels increase the rejection rates of cocoa powder in trade further and will affect trade.

Codex are in the process of developing a Code of Practice for the Prevention and Reduction of Cadmium contamination in cocoa beans (CX/CF 21/14/7) to guide member states and the cocoa production industry in preventing and reducing cadmium contamination in cocoa beans during the production and processing phases.

Table 2: Codex proposed maximum level for cadmium in cocoa powder (100% total cocoa solids based on dry matter)

Commodity/Product name	Maximum level (ML) (mg/kg)	Status (after CCCF14-3-13 May 2021)
Cocoa powder (100% total cocoa solids based on dry matter basis) ready for consumption	2.0-3.0 (analysis of the GEMS/Food data)  1.3-1.5 (proportionality approach)	Step 4 Still under discussion

Maximum level for cadmium in chocolate products and cocoa powder was established in 2014 and enforced on 1 January 2019 in the European Union (Commission Regulation (EU) No. 488/2014). Maximum level of cadmium for chocolate was established based on level of cocoa solids as

indicated in Table 3. Three categories of chocolate, i.e, milk chocolate with less than 30% total dry cocoa solids, milk chocolate with higher or equal to 30% but less than 50% dry cocoa solid and lastly chocolate with higher or equal to 50% total dry cocoa solids.

Table 3: Maximum level of cadmium in EU regulation for three types of chocolate and cocoa powder starting from January 2019

Subsection	Cadmium	mg/kg
3.2		
3.2.7	Specific cocoa and chocolate products as listed below	
	-milk chocolate with <30% total dry cocoa solids	0.10
	-Chocolate with <50% total dry cocoa solids; milk chocolate with ≥30% total dry cocoa solids	0.30
	-Chocolate with ≥50% total dry cocoa solid	0.80
	-Cocoa powder sold to the final consumer or as an ingredient in sweetened cocoa powder sold to the final consumer (drinking chocolate)	0.60

## MATERIALS AND METHODS

### Reagents and materials

Water was purified through an Elga Purelab Option-Q system (High Wycombe, UK). Ultrapure nitric acid (HNO<sub>3</sub>, 65 %) and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>, 30 %) were obtained from Merck (Darmstadt, Germany). Certified Reference Standard solution: 1000 mg/L single element of arsenic, cadmium, lead and antimony were purchased from Ultra Scientific (North Kingstown, USA). Mixed standard solutions for calibration curve ranging from 0.1 µg/L to 50 µg/L were prepared by successive dilution using 8% nitric acid. Internal standard solution containing Bi, Ge, Li, Lu, Rh, Sc and Tb for instrument control and optimization was purchased from Agilent Technologies (Santa Clara, USA).

### Cocoa powder and chocolate samples

Cocoa powder (95) samples obtained from cocoa grinder and also purchased from bakery store and supermarkets. Locally produced and imported chocolates were purchased from local supermarket. A total of 81 milk chocolate samples and 72 dark chocolate samples were collected from 2017-2020.

### Sample digestion.

Digestion was performed using microwave digestion system Milestone Ethos Up (Milestone, Sorisole, Italy). Samples (1 g) were weighed into dry and clean PTFE vessels. Then, 6 mL of HNO<sub>3</sub> and 2 mL of H<sub>2</sub>O<sub>2</sub> were added. The vessels were capped and introduced to the microwave digestion system and digested using a two-step digestion program. Temperature of the vessels was increased to 200 °C in 15 min and stayed another 15 min for digestion to complete. The resulting solutions were cooled, diluted with 50 mL ultrapure water and filtered using filter paper.

### Instruments optimization.

Cadmium quantification was carried out by inductively coupled plasma quadrupole mass spectrometry (ICP-MS) Agilent 8800 ICP-QQQ equipped with a quartz spray chamber, glass concentric nebulizer, on-line internal standard (ISTD) addition kit, and Ni interface cones (Agilent Technologies, Tokyo, Japan). The ICP-MS was allowed to stabilize for 20-30 minutes and the performance was optimized based on radio frequency (RF) power, sampling depth, argon flow rate, collision cell gas flow rate, lens voltage and sample uptake rate. For quantification, the <sup>111</sup>Cd analyte isotope was used.

## RESULTS AND DISCUSSIONS

The analytical protocol for the determination of cadmium and other heavy metals implemented in the Analytical Services Laboratory Malaysian Cocoa Board is a validated method (Mohamed, Zainuddin and Yaakob, 2020). The Analytical Services Laboratory is also accredited to carry out this analytical protocol by the Department of Standard Malaysia under MS ISO/IEC 17025:2017.

Chocolate and cocoa powder samples monitoring was carried out as part of the scope of five years (2016-2020) project, 'Development of Analytical Protocols for Food Safety and Quality in Cocoa and Cocoa Products'.

A total of 81 samples of milk chocolates were collected and analysed between 2017 and 2020. Mean, minimum, maximum and median concentration of cadmium in the samples are as shown in Table 4. Only four samples had cadmium

level higher than the level permitted by EU, 0.1 mg/kg. The EU imposed the strictest maximum

level, compared to Codex (0.3 mg/kg) or Malaysia (1 mg/kg).

Table 4: The descriptive statistic for cadmium content in milk chocolate sampled between 2017 and 2020.

Milk chocolate						
Sampling Year	Mean (mg/kg)	Min (mg/kg)	Max (mg/kg)	Median (mg/kg)	No of samples	No of sample exceeding regulation
2020	0.044	0.01	0.22	0.02	19	One sample exceed EU regulation
2019	0.047	0.01	0.16	0.03	13	Two samples exceed EU regulation
2018	0.046	0.02	0.14	0.04	17	One sample exceed EU regulation
2017	0.05	0.00	0.08	0.035	32	No samples exceed regulations
Total	0.047	0.00	0.22	0.03	81	Four samples exceeding EU regulation

Results of cadmium analyses for dark chocolate are tabulated in Table 5. All 72 samples analysed are

within the permissible limit allowed by the three regulations (Malaysia, Codex and EU)

Table 5: Mean, min, max and medium level of cadmium in dark chocolate sampled in 2017 to 2020.

Dark chocolate						
Sampling year	Mean (mg/kg)	Min (mg/kg)	Max (mg/kg)	Median (mg/kg)	No of samples	No of sample exceeding regulation
2020	0.098	0.02	0.36	0.075	16	No samples exceed regulations
2019	0.065	0.03	0.12	0.06	8	No chocolate samples exceed regulation
2018	0.10	0.02	0.19	0.085	20	No chocolate samples exceed regulation
2017	0.12	0.04	0.20	0.135	28	No chocolate samples exceed regulation
Total	0.102	0.02	0.36	0.075	72	No samples exceed regulations

Cocoa powder samples analysed showed the highest non-compliance compared to chocolate samples. A total of 5 samples were found to exceed the maximum permissible limit of 1 mg/kg (Malaysia Food Regulation, 2010) and 22 samples exceed the maximum level of 0.6 mg/kg by EU (EU). The breakdown for samples exceeding the

Malaysian Food Regulation is 3 samples in 2020 and 4 samples in 2017 which is 13% and 6.9% respectively. The number of samples exceeding the EU regulation are 6 samples in 2020, 2 samples in 2019, 10 samples in 2018 and 8 samples in 2017 which are 26%, 15%, 33% and 6.9% respectively.

The Codex Electronic Working Group for Maximum levels for cadmium in chocolates and cocoa-derived products analysed 5,781 data on the occurrence of cadmium for cocoa powder and concluded that maximum level from 2.0 mg/kg to 3.0 mg/kg would present between 5.39% and a

2.49% of samples rejected worldwide, which will mean rejection rates of between 13.42% and 6.33% for the Latin American region and Caribbean (CX/CF 21/14/6). Normally Codex would like to set a maximum level with rejection rate under 5%.

Table 6: Mean, min, max and median concentration of cadmium in cocoa powder sampled in 2017-2020.

Cocoa powder						
	Mean (mg/kg)	Min (mg/kg)	Max (mg/kg)	Median (mg/kg)	n	No of sample exceeding regulation
2020	0.473	0.05	1.61	0.31	23	3 samples exceed the Malaysian Food Regulation  6 samples exceed the EU regulation
2019	0.41	0.13	0.90	0.39	13	2 samples exceed the EU regulation
2018	0.483	0.13	0.93	0.57	30	10 samples exceed the EU Regulation
2017	0.492	0.12	1.49	0.36	29	2 samples exceed the Malaysian Food Regulation  4 samples exceed the EU Regulation
Total	0.473	0.05	1.61	0.39	95	5 samples exceed the Malaysian Food Regulation  22 samples exceed the EU regulation

## CONCLUSIONS

The level of cadmium occurrence in milk chocolate analysed from 2017 to 2020 showed that only four samples exceed the limit set by EU. All milk chocolates samples comply with Malaysian Regulation. Similarly, all the dark chocolate samples comply with Malaysian Regulation and EU. The occurrence of cadmium in cocoa powder samples showed more samples exceeding both Malaysian and EU regulation. However, this should not be a concern since JECFA assessment showed that the contribution of cadmium from chocolate and cocoa-derived products are minor compared to other food sources which were consumed in larger quantities such as cereals, vegetables, fish and seafoods.

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