

Comparative Assessment of Heavy Metal Levels in the Peel, and Seeds of Tangerine (*Citrus reticulata*) and Grapefruit (*Citrus paradisi*)

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ABSTRACT

This work measured seven heavy metals — chromium (Cr), copper (Cu), cadmium (Cd), nickel (Ni), iron (Fe), lead (Pb), and zinc (Zn) — in the inedible portions (peels and seeds) of sweet tangerine (*Citrus reticulata*) and grapefruit (*Citrus paradisi*) bought from Kawo Market, Kaduna State, Nigeria. Samples underwent wet digestion with a tri-acid mixture ($\text{HNO}_3\text{:HClO}_4$) before analysis by Atomic Absorption Spectrophotometry (AAS). Seeds consistently carried a heavier metal load than peels across both fruits. Benchmarked against World Health Organization (WHO) limits, peel samples showed Cu, Cd, and Fe above safe thresholds, Ni and Cr within limits, and Zn below the recommended level, while seed samples exceeded limits for Cu, Ni, Pb, and Cd, with Cr remaining comfortably under its ceiling. These results point to meaningful contamination in these fruit by-products, raising possible health concerns should they be consumed or reused inadvertently, and underscore the importance of ongoing environmental monitoring and regulatory oversight of heavy metals in the region's agricultural produce.

Keywords: Heavy metals; Tangerine; Grapefruit; Peels and seeds; Atomic absorption spectrophotometry; Food safety.

INTRODUCTION

Citrus Fruits: Nutritional Importance and Bioactive Components of Peels and Seeds

Fruit is a cornerstone of human and animal diets, delivering nutrients essential to health, including water, calcium, and iron. Citrus varieties such as tangerine (*Citrus reticulata*) and grapefruit (*Citrus paradisi*) are especially valued for their vitamin, mineral, and fibre content, and their consumption has been linked to protective antioxidant effects and reduced disease risk (Elbagermi et al., 2012; D'mello, 2003). The peel — termed zest in citrus, rind in fruits such as passion fruit and mangosteen — functions chiefly as a barrier against environmental stress and pathogens, yet is far from biologically inert: it concentrates dietary fibre and phytonutrients, and citrus peel oils in particular carry bioactive compounds such as coumarins, flavonoids, carotenes, terpenes, and linalool (Mandello et al., 2015). Seeds, for their part, serve as the plant's nutrient reserve for germination and are correspondingly rich in vitamins and minerals, while also holding industrial value in soap manufacture, detergent sulfonation and sulfation, and fatty-acid derivative production. Because peels and seeds carry this nutritional and commercial significance, and because they are also the parts most often discarded, repurposed in traditional remedies, or fed to livestock, what they accumulate alongside these beneficial compounds — including contaminants such as heavy metals — is directly relevant to food safety.

Overview of the Study Fruits: Tangerine and Grapefruit

Tangerine (*Citrus reticulata*), commonly known as mandarin orange, is a member of the Rutaceae family cultivated widely across tropical and subtropical regions, Nigeria included, and valued for both flavour and medicinal properties (Piccinelli et al., 2008). The evergreen tree grows to about 9–10 metres, bearing spherical-to-oblong, segmented fruit that holds several seeds within a porous white mesocarp (albedo). Tangerine is a rich

source of vitamin C and phytochemicals such as beta-carotene, lutein, and naringenin (Perez-Cacho et al., 2008); its peel is a particularly concentrated source of antioxidants, reported at up to twenty times the capacity of the juice, with associated benefits for digestion, cholesterol, and blood-sugar regulation, while its seeds contain volatile oils, flavonoids, and antioxidant polyphenols, including limonoids with reported anti-malarial activity.

Grapefruit (*Citrus paradisi*) is a subtropical citrus tree bearing fruit that ranges from sour to semi-sweet, with red-, white-, or pink-fleshed varieties distinguished mainly by pulp colour; its characteristic aroma derives largely from grapefruit mercaptan (Buettner & Schieberle, 1999). The fruit is notable for interacting with a wide range of medications, chiefly by inhibiting intestinal cytochrome P450 enzymes and drug-absorption transporters, which can raise blood drug levels or reduce a medication's effectiveness (Buettner & Schieberle, 1999). Grapefruit peel is a dense source of dietary fibre, essential oils, and vitamin C with reported anti-cancer potential, while its seeds and their extracts show notable antioxidant and antimicrobial activity, properties that have prompted their use as a natural preservative and in textile and leather processing (Armando et al., 1997; Ignacio & Thai, 2005; Sakamoto et al., 1996; Von Woedtke et al., 1999; Takeoka et al., 2001, 2005).

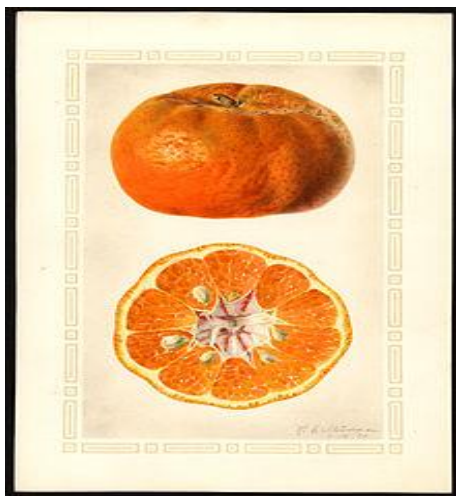


Figure 1. Tangerine (*Citrus reticulata*)



Figure 2. Grapefruit (*Citrus paradisi*)

Heavy Metals: Sources and Environmental Pathways

Although heavy metals occur naturally, human activity is largely responsible for their build-up in the environment, through mining, industrial waste disposal, vehicle exhaust, and the use of fertilizers, paints, and treated wood; deteriorating water-supply infrastructure adds a further source (Balasubramanian, 2009).

Plants, animals, and people all pick up these metals through several routes — breathing contaminated air, eating or drinking polluted food and water, and skin contact chief among them. Vehicle exhaust alone contributes substantial airborne cadmium, cobalt, nickel, lead, and zinc, while industrial waste can leach into water bodies, a process acid rain speeds up by freeing metals bound in soil. For people specifically, diet — both plant- and animal-based — remains the dominant exposure pathway (Radojevic & Bashkin, 1999).

Within cells, heavy metals cause harm by binding to structural proteins, enzymes, and nucleic acids, interfering with their normal function; over time, this kind of chronic exposure has been tied to cancer, nerve damage, and harm to the circulatory system.

Essential vs. Non-Essential Metals

Not all heavy metals are alike biologically: some, including iron (Fe), zinc (Zn), copper (Cu), chromium (Cr), manganese (Mn), and molybdenum (Mo), are essential, acting as cofactors that keep key metabolic reactions running, needed only in trace amounts (Banfalvi, 2011). Others — cadmium (Cd), lead (Pb), and mercury (Hg) among them — serve no known biological purpose and are simply toxic; they enter organisms either by hitching a ride along the same pathways used by essential metals or through direct exposure from industrial, agricultural, or consumer sources (Tchounwou et al., 2012).

Profiles of Selected Heavy Metals

As an essential trace element, copper (Cu) supports enzymes involved in iron metabolism, nervous-system function, and bone formation (Murry et al., 2000; Kalagbor et al., 2014), though too much of it can damage the liver and kidneys.

Iron (Fe), best known for its role in haemoglobin, is indispensable for carrying oxygen through the body, yet an excess of it turns toxic, damaging tissue and impairing organ function (Kalagbor et al., 2014).

Zinc (Zn) plays an important role in both plants and animals, activating enzymes and supporting protein synthesis and the metabolism of carbohydrates and nucleic acids (Anju et al., 2011); too little causes metabolic disorders, while too much can damage the pancreas and disturb protein metabolism (Kalagbor et al., 2014).

In trace amounts, nickel (Ni) can activate certain enzyme systems, but it is more widely known for its toxicity at higher levels, where prolonged overexposure has been linked to elevated cancer risk across several tissue types.

Trace amounts of chromium (Cr) support glucose and lipid metabolism, and while deficiency is possible, the greater concern is toxicity at high concentrations, which can disrupt metabolic function and organ systems (Kalagbor et al., 2014).

Lead (Pb) serves no biological function whatsoever and instead acts as a powerful, cumulative toxin, harming virtually every organ system, with the nervous, renal, and blood-forming systems particularly affected (Lokeshappa et al., 2012; Taupeau et al., 2001).

Cadmium (Cd) is highly toxic in its own right; prolonged exposure can cause irreversible kidney damage, lung injury, bone loss, anaemia, and cancer.

Aim and Objectives

This study set out to measure selected heavy metals — chromium, copper, cadmium, nickel, iron, lead, and zinc — in the peels and seeds of tangerine (*Citrus reticulata*) and grapefruit (*Citrus paradisi*) sold at Kawo Market, Kaduna State, Nigeria, and to assess the health risks these levels might pose. Specific objectives were to:

1. digest and prepare samples of tangerine and grapefruit peels and seeds for heavy metal analysis using a tri-acid mixture;
2. quantify the concentrations of Cr, Cu, Cd, Ni, Fe, Pb, and Zn in these samples using Atomic Absorption Spectrophotometry (AAS);
3. compare the levels of heavy metal accumulation between the peel and seed components of each fruit; and
4. evaluate the obtained concentrations against the permissible limits established by the World Health Organization (WHO) to determine compliance and potential safety concerns.

MATERIALS AND METHODS

Reagents and Chemicals

Every reagent used was analytical grade: concentrated nitric acid (HNO_3 , 69–72%) and perchloric acid (HClO_4 , 70%) served for sample digestion, while deionized water was used throughout for dilution, standard-solution preparation, and glassware rinsing.

Sample Collection and Preparation

Fresh tangerine (*Citrus reticulata*) and grapefruit (*Citrus paradisi*) were bought from Kawo Market, Kaduna State, Nigeria, then washed thoroughly in distilled water to remove surface contaminants. Peels and seeds were separated by hand, air-dried, and finished with oven-drying at 70°C to a constant weight. The dried material was

ground to a fine powder with a porcelain mortar and pestle and kept in clean, labelled, airtight containers until analysis.

Digestion Procedure

Digestion followed a wet method. Precisely 1.03 g of each powdered sample was weighed on an analytical balance and placed in a 150 mL digestion beaker, then treated with a 10 mL aliquot of a 1:4 (v/v) perchloric acid (70%) / nitric acid (69–72%) mixture. Samples were heated gently at 120°C on a hot plate inside a fume hood until the digest cleared, signalling complete oxidation of organic matter. Once cooled, the digest was filtered through Whatman No. 42 paper into a 20 mL volumetric flask, the residue rinsed with deionized water, and the filtrate made up to volume. Digested samples were held at 4°C pending analysis (Kalagbor et al., 2014).

Instrumental Analysis

Chromium, copper, cadmium, nickel, iron, lead, and zinc concentrations were quantified using an Atomic Absorption Spectrophotometer (AAS), calibrated against certified intermediate standards for each metal, with a blank digest run alongside the samples to correct for background contamination and every analysis performed in triplicate for reproducibility. Instrument detection limits (IDL) for direct-aspiration flame AAS were taken from the standard reference values of USEPA Method 7000B (2007) [10] and converted to a sample-weight basis using the digestion parameters above (1.03 g sample to 20 mL final volume), giving Cd 0.10, Cu 0.39, Fe 0.58, Cr 0.97, and Zn 0.10 mg/kg; values reported as "ND" (Not Detected) in Tables 1 and 2 for these metals fell below these thresholds. For Pb and Ni, the generic USEPA reference limits (equivalent to approximately 1.9 mg/kg and 0.8 mg/kg respectively at this dilution) exceed some of the concentrations reported as detected in Tables 1 and 2, indicating that a more sensitive calibration or measurement approach than the generic standard was evidently achieved for these two metals; the authors should report the instrument-specific detection limits actually verified for Pb and Ni on the day of analysis.

RESULTS AND DISCUSSION

A. Heavy Metal Concentrations in Fruit Peels

Table 1 presents heavy metal concentrations (mg/kg dry weight) measured in tangerine and grapefruit peels, set against the maximum limits established by the World Health Organization (WHO).

Table 1. Concentration of Heavy Metals in Fruit Peels (mg/kg dry weight)

Metal	Tangerine (mg/kg)	Grapefruit (mg/kg)	WHO Permissible Limit (mg/kg)
Cu	2.50	3.75	0.5
Ni	ND	0.60	0.14
Pb	ND	0.28	0.3
Cr	1.43	ND	1.5
Cd	0.31	0.63	0.03
Zn	1.19	1.19	99.4
Fe	1.09	2.17	0.8

ND = Not detected (below the instrument detection limit for that metal; see Section III.D).

Heavy Metal Concentrations in Fruit Peels vs. WHO Permissible Limits

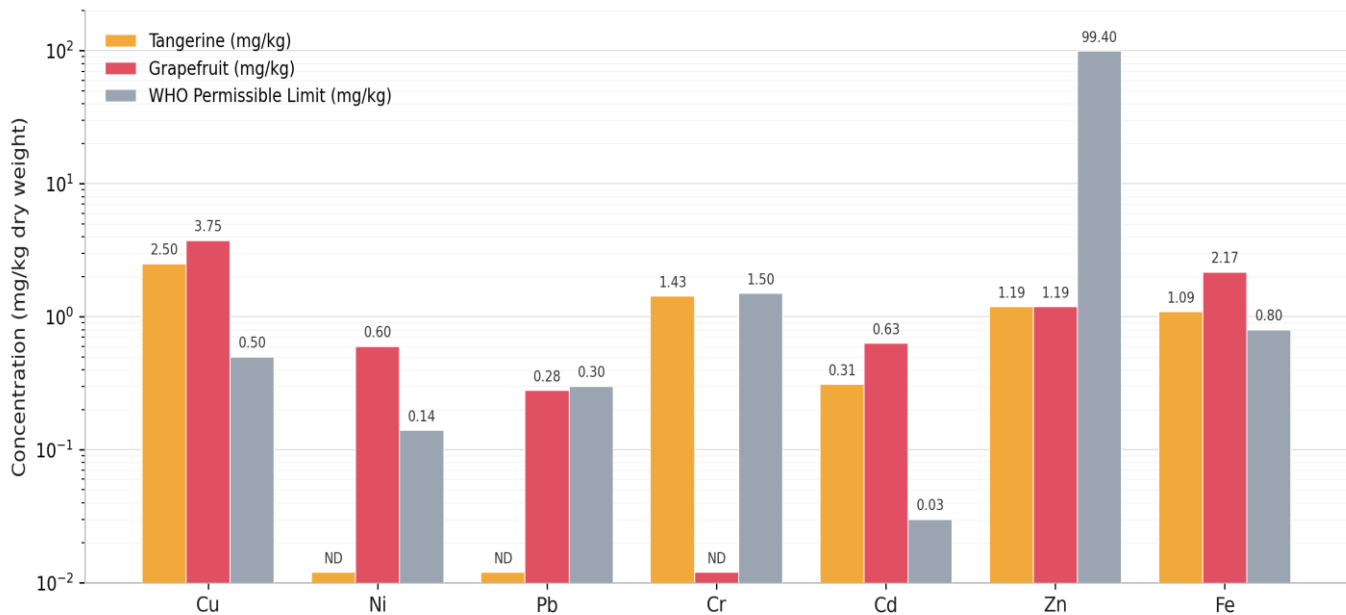


Figure 3. Comparison of heavy metal concentrations in tangerine and grapefruit peels against WHO permissible limits.

Copper (Cu)

Copper concentrations in both peel samples (2.50–3.75 mg/kg) exceeded the WHO limit of 0.5 mg/kg, with grapefruit peel carrying the heavier load. Copper is needed in small amounts for enzyme function and iron metabolism, but sustained exposure at elevated levels can damage the liver and kidneys (Kalagbor et al., 2014).

Nickel (Ni)

Nickel was detected only in grapefruit peel, at 0.60 mg/kg, exceeding the WHO limit of 0.14 mg/kg. Trace nickel can activate certain enzymes, but at higher concentrations it is a recognized carcinogen, and long-term exposure has been tied to various cancers (Divrikli et al., 2006; Khodadoust et al., 2004).

Lead (Pb)

Lead was not detected in tangerine peel but was present in grapefruit peel at 0.28 mg/kg, just below the WHO threshold of 0.3 mg/kg. Although within the permissible limit, this concentration warrants monitoring given lead's cumulative toxicity to the nervous system and kidneys (Lokeshappa et al., 2012).

Chromium (Cr)

Chromium was detected only in tangerine peel, at 1.43 mg/kg, within the WHO safety limit of 1.5 mg/kg. Since trivalent chromium plays a role in glucose metabolism, the levels found here do not point to immediate toxicity.

Cadmium (Cd)

Cadmium concentrations in both peels (0.31–0.63 mg/kg) substantially exceeded the strict WHO limit of 0.03 mg/kg. As a highly toxic, non-essential metal with a long biological half-life, chronic cadmium intake is linked to kidney dysfunction, bone loss, and cancer.

Zinc (Zn)

Zinc concentrations were identical in both peels at 1.19 mg/kg, well below the WHO limit of 99.4 mg/kg, indicating no toxicological risk, though this may reflect relatively low bioavailable zinc in these fruits, an element essential for many enzymatic processes (Anju et al., 2011).

Iron (Fe)

Iron concentrations in the peels (1.09–2.17 mg/kg) exceeded the WHO limit of 0.8 mg/kg. Iron is essential for oxygen transport, but too much of it can drive oxidative stress and tissue damage (Kalagbor et al., 2014).

General Implication

Taken together, these findings show that fruit peels — often discarded as waste or repurposed in traditional remedies — can build up Cu, Cd, Ni, and Fe to levels that warrant public-health attention, reinforcing the case for monitoring local agricultural soils and irrigation water to trace contamination sources. Consumers would do well to be cautious about unregulated use or consumption of citrus peels bought in urban markets.

B. Heavy Metal Concentrations in Fruit Seeds

Table 2 lists heavy metal concentrations measured in tangerine and grapefruit seeds; the results indicate substantial contamination in both fruits.

Table 2. Concentration of Heavy Metals in Fruit Seeds (mg/kg dry weight)

Metal	Tangerine (mg/kg)	Grapefruit (mg/kg)	WHO Permissible Limit (mg/kg)
Cu	2.50	5.00	0.5
Ni	0.89	0.89	0.14
Pb	0.57	0.57	0.3
Cr	1.43	1.43	1.5
Cd	0.94	0.94	0.03
Zn	2.38	2.38	99.4
Fe	2.17	ND	0.8

ND = Not detected (below the instrument detection limit for that metal; see Section III.D).

■ Tangerine ■ Grapefruit — WHO permissible limit

Figure 3. Heavy Metal Concentrations in Tangerine and Grapefruit Peels and Seeds vs. WHO Limits

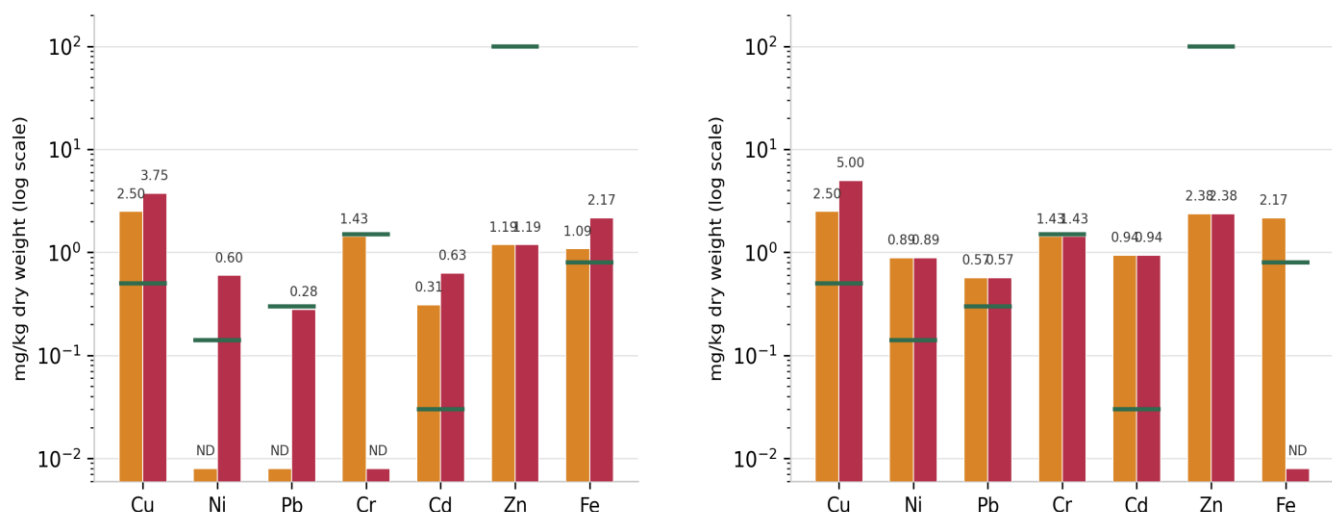


Figure 4. Comparison of heavy metal concentrations in tangerine and grapefruit seeds against WHO permissible limits.

Copper (Cu)

Copper concentrations measured 2.50 mg/kg in tangerine seeds and 5.00 mg/kg in grapefruit seeds, both exceeding the WHO limit of 0.5 mg/kg, with grapefruit notably higher. Sustained high copper intake can harm both liver and kidneys (Kalagbor et al., 2014).

Nickel (Ni)

Both seed types carried the same nickel concentration, 0.89 mg/kg, well above the WHO limit of 0.14 mg/kg. Given nickel's recognized carcinogenicity, this level represents a significant long-term health concern.

Lead (Pb)

Lead concentrations reached 0.57 mg/kg in both seed samples, nearly twice the WHO safety limit of 0.3 mg/kg — a notably elevated figure given lead's potency as a cumulative neurotoxin (Lokeshappa et al., 2012).

Chromium (Cr)

Chromium remained consistent at 1.43 mg/kg across both seed types, within the WHO limit of 1.5 mg/kg, suggesting relatively low immediate risk from chromium exposure through seeds.

Cadmium (Cd)

Cadmium levels matched across seed types at a markedly elevated 0.94 mg/kg, substantially exceeding the strict WHO limit of 0.03 mg/kg — given cadmium's severity as a nephrotoxin and carcinogen, this stands out as one of the study's most significant findings.

Zinc (Zn)

Zinc concentrations of 2.38 mg/kg remained well below the WHO limit of 99.4 mg/kg, indicating no toxicological concern for this element in seeds.

Iron (Fe)

Iron was detected at 2.17 mg/kg in tangerine seeds but was not detected in grapefruit seeds; the tangerine figure exceeds the WHO limit of 0.8 mg/kg, which could contribute to oxidative stress if consumed in quantity.

C. Comparative Analysis: Seeds vs. Peels

Wherever a direct comparison was possible, seeds carried higher heavy metal concentrations than peels from the same fruit, a pattern consistent for Cu, Ni, Pb, and Cd. Several physiological and environmental factors plausibly underlie this difference. Seeds are metabolically active reproductive tissue: during development, the embryo and its surrounding endosperm draw nutrients, and along with them non-essential trace metals, through the plant's vascular system, so that metals translocated via the xylem and phloem from root uptake tend to concentrate in these nutrient-storage organs. Seed tissue is also comparatively rich in metal-binding ligands — proteins, phytate, and polyphenolic compounds — that can chelate and retain heavy-metal ions once they arrive, effectively acting as an internal sink. The peel, by contrast, functions primarily as a protective barrier: its waxy cuticle limits ion penetration from the surrounding environment, and any metal deposited on the peel surface from airborne particulates, irrigation water, or agrochemical residues is largely superficial and can be partly removed by washing, rather than being translocated and internalized as it is in seeds.

This translocation-versus-surface-deposition distinction is consistent with the essential-versus-non-essential character of the metals involved: Cu and Ni, though needed in trace amounts, and the non-essential toxicants Pb and Cd, all showed markedly higher seed concentrations, suggesting active physiological uptake pathways rather than passive surface contamination alone. The consistently elevated Cu, Ni, Pb, and Cd levels found in seeds,

well beyond WHO safety limits, indicate a meaningful health risk associated with the inedible parts of these widely consumed fruits, and point to seed-bound metal translocation as a priority for further mechanistic study.

CONCLUSION

This study quantified seven heavy metals in the peels and seeds of tangerine and grapefruit sold at a local market in Kaduna State, Nigeria. Copper, cadmium, and iron in the peels exceeded WHO safety limits, while the seeds were more heavily contaminated still, with copper, nickel, lead, and cadmium all recorded well above permissible levels; cadmium and lead, both non-essential and highly toxic, reached the most concerning concentrations. Seeds consistently carried a heavier metal burden than peels in both fruits, a pattern most plausibly explained by internal metal translocation and binding during seed development rather than surface deposition alone.

These findings carry direct public-health and policy implications. Although the pulp is the part normally eaten, peels and seeds are sometimes repurposed into traditional remedies, animal feed, or processed foods, and their elevated toxic-metal content means unregulated use risks chronic heavy-metal exposure, with attendant harm to the kidneys, liver, and nervous system and elevated cancer risk. Two actions follow directly: regulatory agencies should extend routine heavy-metal monitoring of agricultural soils and irrigation water to the citrus-growing areas supplying urban markets such as Kawo Market, and public-health authorities should issue clear guidance discouraging the informal reuse of citrus peels and seeds in food, feed, or herbal preparations until such monitoring confirms safe levels.

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