



## Monitoring of Chemical Hazards: Pesticide Residue, Heavy Metals and Mycotoxin Detection during Production of Mango and Guava Pulp Production Line

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### Abstract

Food safety has become a top priority for humanity worldwide. Chemical hazards are considered among the most important risks that pose a real danger to the consumer. International bodies specialized in food safety have set maximum limits for these risks in food not which effect on health and safety for consumer. The aim of this research is to estimate the potential chemical hazards presented in mango and guava fruits used in pulp processing in one of the local companies working in the field of juice production. Samples were taken from raw mango and guava fruits that are supplied to the company before washing by hot water at 80 C°/60 sec and after pasteurization processes mango and guava pulp at 110 C°/40 sec. Results were compared with the maximum limits which recommended by the Codex in (2019) and obtained results showed that the residues of pesticides and heavy metals before washing mango and guava fruits by hot water and after pasteurization processes of pulp within the permissible limits which recommended by the Codex, 2019 while, mycotoxins namely Patulin and Ochratoxin A were not found in all investigated samples. Obtained results showed that, there were pesticide residues before rinsing by hot water in mango fruits: Insecticides namely Cypermethrin is 0.01 mg/kg and Lambada-Cyhalothrin was less than limit of quantification (LOQ) while Chlorpyrifos is 0.01 mg/kg. Fungicides (Boscalid and Carbendazim) were less than limit of quantification (LOQ), while after pasteurization processes of mango pulp, no pesticide residues were detected except Carbendazim fungicides and it was also less than limit of quantification (LOQ). Detection of heavy metals indicated that, Cadmium is the only heavy metal was detected before rinsing by hot water and after pasteurization processes of mango pulp and it was less than those of limit of quantification (LOQ), while Lead, Arsenic and Mercury not found before rinsing by hot water or after pasteurization processes. Results of mycotoxins namely Patulin and Ochratoxin A not found before rinsing by hot water or after pasteurization processes.

Results of guava fruits showed that there pesticide residue detected before rinsing by hot water namely Cypermethrin 0.05 mg/kg and Acetamiprid was less than limit of quantification (LOQ) while Chlorpyrifos was 0.01 mg/kg. Also Carbendazim Fungicides was less than limit of quantification (LOQ). While after pasteurization processes of guava pulp, Cypermethrin was decreased to 0.029 mg/kg, while the amount of Acetamiprid and Chlorpyrifos remained the same after pasteurization processes of guava pulp, while Carbendazim was not found.

Two heavy metals (Lead and Cadmium) were detected before rinsing by hot water. Pb 0.071 mg/kg and Cadmium less than limit of quantification (LOQ) and were in legal limit while Arsenic and Mercury not found. Lead decreased after pasteurization processes of guava pulp into 0.04 mg/kg while Cadmium, Mercury and Arsenic not found after pasteurization processes of guava pulp. Results of mycotoxins namely Patulin and Ochratoxin A not found before rinsing or after pasteurization. It can be conclude from the results of this study that, rinsing by hot water and thermal treatment process effect positively to remove or reduce some of pesticides and heavy metals in mango and guava pulp production line. Thus, ensuring that the final product is free of chemical hazards or that they are within the permissible legal limits.

**Keywords:** Food Safety; Pesticide Residues; Heavy Metals; Mycotoxin

## Introduction

Food safety has become a major concern worldwide due to the alarming rise of chemical pollutants in the environment [24]. Food safety is to warranty that food will not harm the consumer [13]. The hazard in food are categorized into biological, chemical and physical [19]. Estimation of risk of pesticide residues in foods is a high priority in risk assessment, where an assessment must be made of the possibility of reaching the consumer, as well as the severity of the effects resulting from it if it reaches the consumer. Therefore, international legislation must be complied with, which establishes maximum levels in food products that do not harm the health of consumers [17]. Compared to another food groups, pesticide residue are detected at high concentrations in fruit and vegetables and a higher percentage of these samples exceeded the permissible level (MRL) [49]. In fact, fruits and vegetables are a major exposure way to intake the pesticide residue in the general pollution [10], especially when these foods are eaten without peeling and uncooked [48]. Different food processing processes can change the pesticide residue concentration content in the final product, either by an increase or a decrease [5,52].

The presence of heavy metals in food at a rate higher than the permissible limits can lead to major health problems [21]. Natural processes, (i.e., volcanic eruptions, leaching from soil and rocks etc.), and anthropogenic activities including mining, industrialization increases the accession of heavy metals to foods through the food chain [4], and is the main concern in food safety, human health and quality assurance [46]. Also, the use of chemical fertilizers, pesticides and polluted water for irrigation are the main factor's responsible contamination by heavy metals, almost all in fruits and vegetables [31]. Owing to the possibility of accumulation of heavy metals inside the vital organs of the body and the lack of an adequate mechanism to get rid of them in the body, as well as their long biological half-life, they lead to many very dangerous diseases for humans [46]. As a result, mutagenic, carcinogenic, and cardiac, nervous system, liver, kidney, lung, bone and spleen diseases have arisen due to the bioaccumulation of these heavy metals in vital organs of the human body [36]. Heavy metal contamination of fruits is one of the most common problems because of the heavy use of fertilizers, pesticides and other chemicals - is the main common cause to presence it in fruits - due to the pursuit for fast economic development in the agriculture sector [34].

Mycotoxins are secondary metabolites produced by pathogenic fungi that colonize fruits and vegetables either during harvesting or during storage. The accumulation of mycotoxins in fruits and vegetables not only poses a threat to human and animal health, but also leads to serious economic losses. Therefore, these potential risks to humans as well as economic losses must be reduced by monitoring,

detecting and controlling these toxins [32]. Patulin and Ochratoxin A are the mycotoxin which associated more frequently with fruit juice [44].

The aim of this research is to estimate the potential chemical hazards such as pesticide residues, heavy metals (Lead, cadmium, Arsenic and Mercury) and mycotoxin (Patulin and Ochratoxin A) presented in mango and guava fruits used in pulp processing in one of the local companies working in the field of juice production.

## Materials and Methods

### Samples collection

Fruit samples (mango and guava ) were collected from the production line each 2 hours during the shift starting until the ended from 2 process step before rinsing and after pasteurization processes.

### Samples preparation

Fruit samples were divided into 2 groups before rinsing and after pasteurization processes of pulp: a) the first group (before rinsing by hot water at 80 C°/60 sec) mixed it in a mixer model molenix then a representative sample obtained B) group (after pasteurization processes of pulp at 110 C°/40 sec) mixed it in a sterilized flask, a representative sample obtained from it, all samples were kept under freezing storage for one day until analysis were carried out.

### Determination of pesticide residues

Pesticide residue were extracted from samples by using method called SPE-QuEChERS [20], as following: 15 g were taken from the representative sample, then, 15 ml of acetonitrile (acidified with acetic acid 1% v/v) was added and vortexed for 2-3 min. then 6 gm of magnesium sulfate (MgSO<sub>4</sub>) and 1.5 g of sodium chloride (NaCl) were added to this mixture and vortexed again for 3 min. then, the mixture was centrifuged at 3000 rpm for 5 min at room temperature and the upper layer was used for sample cleanup. Then, 0.150 g of MgSO<sub>4</sub>, 0.05 g of primary secondary amine (PSA) and 0.0075 g of graphitized carbon black (GCB) were added per one ml of sample collected and then manually mixed for 20 sec and centrifugation at 3000 rpm for 5 min at room temperature. The supernatant was transferred to a glass tube and evaporated to dryness in a nitrogen flow at 40 °C. Then, the supernatant was reconstituted by added 500 µL of acetonitrile and 500 µL of Milli-Q ultrapure water to prepare one ml of final extract for LC-MS/MS analysis. After extraction pesticide residue from samples by the method as previously described, LC-MS/MS analysis was carried out by using an Agilent 1100 HPLC system (Agilent, Santa Clara, CA, USA) [50].

### Determination of heavy metals concentrations

Heavy metals were extracted from samples by digested using a method called the dry ash process [6], as following: A weight of 5 g from sample was placed in a crucible and then dried at 105 °C in an oven. The crucible was placed in a Muffle oven (Vulcan 3-550, made in the USA), and the temperature was judiciously increased to 550 for 8 hours until digestion was completed. The sample was then cooled in a desiccator. Volume of 1 mL from HCL was utilized to dissolve the remained ash. Then it was transported by de-ionized water to complete a volume of 25 mL. The ash suspension was filtered through Whatman No. 42 and stored in a refrigerator for the determination of heavy metals by inductively coupled plasma optical emission spectrometry ICP-OES. Then, All analysis of samples were performed using the Agilent 5100 Synchronous Vertical Dual View (SVDV) ICP-OES [7].

### Determination of mycotoxins (Patulin and Ochratoxin-A) concentrations

Mycotoxins (Patulin and Ochratoxin-A) were extraction from samples by using QuEChERS method modified [43] for extract mycotoxins from juice as following: A weight of 5 g from the sample was added to 7.5 mL of acetonitrile, 4 g of MgSO<sub>4</sub>, and 300 mg of octadecyl (C<sub>18</sub>). The mixture was then vortexed for 3 minutes and centrifuged for 3 minutes at 4500 rpm. The upper layer (5 mL) was separated in another tube and added to a mixture of 900 mg MgSO<sub>4</sub> and 300 mg C<sub>18</sub>, which was vortexed for 30 seconds before centrifugation at 4500 rpm for 1 minute. The upper layer (2 mL) was separated in another tube and evaporated to dryness with a Turbovap LV Evaporator at 35 °C under nitrogen flow. Volume of 0.5 mL of the 70:30 v/v methanol/water mixture was used to dissolve the remaining ash by vortexing vigorously and finally filtering through a 0.22 mm nylon filter before the injecting into the LC-MS/MS. After extraction mycotoxins from samples by the method as previously described, LC-MS/MS analysis was carried out by using an Agilent HPLC 1200 Series (Agilent Technologies, Palo Alto, CA, USA) [27].

## Results and Discussion

### Determination of pesticide residues in mango and guava fruits before rinsing and after pasteurization processes of pulp

Pesticides are chemicals that are widely used in agricultural sector to control of pests and keep of the crops from any pathogens [35]. Despite that, pesticides can make a problems for the environment and human health [29].

Pesticide residues concentration in mango and guava fruit before rinsing by hot water and after pasteurization process mango and guava pulp are presented in table 1.

As shown in Table (1) two groups of pesticide residues namely insecticides and fungicides were detected in mango and guava fruits before rinsing by hot water at 80 °C/60 sec and after pasteurization processes at 110 °C/40sec of mango and guava pulp in a difference concentrations as following: three insecticides residues molecules were found in mango fruit samples before rinsing by hot water namely, Cypermethrin at 0.01 mg/kg and Lambada-Cyhalothrin was less than limit of quantification while Chlorpyrifos was 0.01 mg/kg, also, two Fungicides residues molecules were found before rinsing by hot water at 80 °C/60 sec namely, Boscalid and Carbendazim and these were less than limit of quantification (LOQ), while after pasteurization processes mango pulp, no pesticide residues were detected except Carbendazim fungicides and it was also less than limit of quantification (LOQ). In guava fruits, three insecticide residues molecules also found before rinsing by hot water namely, Cypermethrin at 0.05 mg/kg and Acetamiprid was less than limit of quantification (LOQ) while Chlorpyrifos was 0.01 mg/kg, also, carbendazim is the only fungicide residues was found before rinsing by hot water at 80 C/60 sec, while after pasteurization processes of pulp, Cypermethrin decreased into 0.029 mg/kg, while the amount of Acetamiprid, Chlorpyrifos remained the same after pasteurization processes guava pulp, while Carbendazim was not found.

Results in table 1 were compared with the Maximum Residue Limit (MRL) as defined as the highest levels of pesticide residues legally tolerated in foodstuffs [33], which is permissible as the highest limit authorized by Codex Alimentarius commission (2019a) [11]. It was observed that all pesticide residues found in mango and guava fruits before rinsing at 80 °C/60sec and after pasteurization processes mango and guava pulp did not exceed the maximum residue limit (MRL) as illustrated in the same Table (1), as authorized by Codex Alimentarius commission (2019a) [11]. Presence of pesticide residues in mango and guava fruits in legal limits as authorized by Codex Alimentarius commission (2019a) [11] before rinsing by hot water, refers to, mango and guava fruits suppliers applied the good agricultural practices (GAP) before supply the fruits to company by applying the dose of pesticides that is recommended and the harvesting time. (Pogacean., *et al.* 2014 and Podbielska., *et al.* 2017) mentioned that, application of recommended dose and harvest time has a great impact on initial concentrations in raw agricultural commodity and the Maximum Residue Limit (MRL) [38,37]. Our results were in accordance with (Sohair, *et al.* 2015) observed that, Chlorpyrifos residues at 0.02 mg/kg, Carbendazim residues at 0.05 mg/kg and Cypermythrin residues at 0.05 mg/kg but not observed Boscalid and Lambada-Cyhalothrin residues in mango fruit sample [47]. Also, Our results were in accordance with (Sohair, *et al.* 2015) who observed that, Acteamiprid residues at 0.11 mg/kg, Chlorpyrifos residues at 0.04 mg/kg, Carbendazim residues at 0.05 mg/kg and Cypermythrin residues at 0.05 mg/kg in guava fruit sample [47].

Pesticide residues		Process step		LOQ (mg/kg)	MRL (mg/kg) (Codex, 2019a) [11]
		Mango and guava fruits Before Rinsing at 80c /60sec (mg/kg)	Mango and guava pulp After pasteurization processes(mg/kg)		
A) Mango samples					
Insecticides	Cypermethrin	0.01	N.D	0.01	0.7
	Lambda-Cyhalothrin	< LOQ	N.D	0.01	0.2
	Chlorpyrifos	0.01	N.D	0.01	0.01
Fungicides	Boscalid	< LOQ	N.D	0.01	0.01
	Carbendazim	< LOQ	< LOQ	0.01	5.0
B) Guava samples					
Insecticides	Cypermethrin	0.05	0.029	0.01	0.05
	Acetamiprid	< LOQ	< LOQ	0.01	0.01
	Chlorpyrifos	0.01	0.01	0.01	0.01
Fungicides	Carbendazim	< LOQ	N.D	0.01	0.1

**Table 1:** Determination of pesticide residues in mango and guava fruits before rinsing and after pasteurization processes of mango and guava pulp.

LOQ: Limit of Quantification; N.D: Not Detected; MRL: Maximum Residue Limit

The results shown also, rinsing by hot water at 80 C°/60 sec for mango and guava fruits and pasteurization processes of pulp at 110 C°/40sec had a great impact processes to removal a most of pesticide residues. Our results were in accordance with (Rodrigues, *et al.* 2017; Polat and Tiryaki, 2019) mentioned that, rinsing is the first step in food processing to remove dust, soil and pesticide residues [42, 39]. Also, our results also were accordance with Holland, *et al.* (1994) mentioned that, pesticides molecules undergo volatilization, hydrolysis or degradation reactions during thermal processing [23]. Many researchers have found that thermal processing is effective in removing pesticide residues from different fruit commodities such as apples [16] grapes [45, 22], apricots, peaches and oranges [8].

The results shown also, Cypermethrin pesticide not found after pasteurization processes of mango pulp while it's decreased after pasteurization processes from 0.05 to 0.029 mg/kg in guava pulp, its mean that, pasteurization processes of mango and guava was effect positively on its concentration and it's in legal limit which authorized by Codex Alimentarius commission (2019a) [11]. Our results in accordance with Yuncheng, *et al.* (2012) who observed that, Cypermethrin, thermally unstable pesticide, and the residue levels in concentrated orange juice was reduced by 63.6% [51].

No effect of rinsing by hot water and the pasteurization processes of mango pulp on Carbendazim concentration and its presence at the same concentration at less than limit of quantification before rinsing by hot water while not observed it in guava pulp after

pasteurization processes. Despite that, Carbendazim present in the legal limit which authorized by Codex Alimentarius commission (2019a) [11]. Our results were in accordance with Yuncheng, *et al.* (2012) who observed that, carbendazim, thermally stable pesticide, and the residue level was increased 1.1 times in orange concentrated [51].

Results also showed no effect of rinsing by hot water and pasteurization processes of guava pulp on Chlorpyrifos concentration and its presence at the same concentration while not found it in mango pulp after pasteurization processes. Despite that, chloropyrifos present in the legal limit which authorized by Codex Alimentarius commission (2019a) [11]. We can Interpretation it that, a mango peel was removed after rinsing by hot water but in guava fruit peel is not removed. This can explain presence of Chlorpyrifos residues in guava fruit after pasteurization. These result accordance with Marudov, *et al.* (1999) who mentioned that, pasteurization had a few effect on chlorpyrifos residues reduction in peach nectar [30].

In the same table, the amount of acetamiprid remained the same trend after rinsing by hot water and after pasteurization processes of guava pulp at less than limit of quantification (LOQ). Despite that, acetamiprid present in the legal limit which authorized by Codex Alimentarius commission (2019a) [11]. Our results in accordance with Holland, *et al.* (1994) who observed that, Acetamiprid is a systemic insecticide, which means that the substance



penetrates into deeper layers of the plant and is more difficult to remove, thus high temperature caused concentration of residues [23].

**Determination of heavy metals in mango and guava fruits before rinsing and after pasteurization processes of pulp**

Fruits contamination by heavy metals is one of the major problems that arise from the huge uses of fertilizers, pesticides and oth-

er chemicals. Lead, cadmium, mercury and arsenic are among the most risky heavy metals when consumed through contaminated food [34,40].

The concentration of heavy metals in mango and guava fruits before rinsing by hot water and after pasteurization processes of mango and guava pulp are presented in table 2.

Heavy metals	Process step		LOQ (mg/kg)	MRL (mg/kg) (Codex, 2019b)[12]
	Before rinsing (mg/kg)	After pasteurization processes (mg/kg)		
A) Mango samples				
Lead (Pb)	N.D	N.D	0.02	0.1
Mercury (Hg)	N.D	N.D	0.05	0.1
Cadmium (Cd)	< LOQ	< LOQ	0.02	0.05
Arsenic (As)	N.D	N.D	0.02	0.1
B) Guava samples				
Lead (Pb)	0.071	0.04	0.02	0.1
Mercury (Hg)	N.D	N.D	0.05	0.1
Cadmium (Cd)	< LOQ	N.D	0.02	0.05
Arsenic (As)	N.D	N.D	0.02	0.1

**Table 2:** Determination of heavy metals in mango and guava fruits before rinsing and after pasteurization processes of mango and guava pulp.

LOQ: Limit of Quantification; N.D: Not Detected; MRL: Maximum Residue Limit

As shown in table (2) Lead, mercury and arsenic were not detected in raw mango fruits before rinsing by hot water and after pasteurization processes of mango pulp. Cadmium was only detected in raw mango fruits before rinsing by hot water and after pasteurization processes of mango pulp, and it was concentrated less than limit of quantification (LOQ), and it was not exceeded the legal limit (0.05 mg/kg) which authorized by Codex Alimentarius commission (2019b) [12].

Results also showed that, raw guava fruits before rinsing by hot water contained lead at a concentration of 0.071 mg/kg, while after pasteurization processes guava pulp, Lead decreased into a concentration of 0.04 mg/kg which is considered below the maximum permissible limit which authorized by Codex Alimentarius commission (2019b) [12]. Cadmium was only detected in raw guava fruits before rinsing by hot water and it was concentrated less than limit of quantification (LOQ), and it was not exceeded the legal limit (0.05 mg/kg) which authorized by Codex Alimentarius commission (2019b) [12], while after pasteurization processes guava pulp, cadmium was not detected. On the other hand, mercury and arsenic were not detected before rinsing by hot water and after pasteurization processes guava pulp.

Heavy metal analysis showed the absence of lead, mercury and arsenic while cadmium is the only element detected in raw mango before rinsing by hot water and after pasteurization processes mango pulp but it less than these limit of quantification (LOQ) and not exceeded the maximum permissible limit (0.05 mg/kg) authorized by Codex Alimentarius commission (2019b) [12]. Our results also were in accordance with (May, *et al.* 2019) who reported that absence of Lead in all fruit sample in Egypt [31]. Cadmium is spread in environment and can be source it fertilizers and pesticides which used to in agricultural sector and various industrial uses such as NiCd batteries [2].

The results revealed that lead concentration decreased after pasteurization processes guava pulp from 0.071 mg/kg into 0.04 mg/kg also, cadmium detected only before rinsing by hot water, while not detected it after pasteurization processes guava pulp. Our results in accordance with (Chinazo, *et al.* 2020) who mentioned that found lead and cadmium in a different concentrations in sample of fruits [9]. Also, our results are in agreement with those of (Igwegbe., *et al.* 1992) who found that, rinsing of crops could

lead to remove of high amounts of the heavy metals that may be present as surface contaminants [26].

**Determination of mycotoxin in mango and guava fruits before rinsing and after pasteurization processes of pulp**

Mycotoxins are less volatile, low molecular weight compounds and highly toxic effect and intake it can affect acute on human health [14,28]. Patulin is one of mycotoxin which produced by certain fungal species such as *Penicillium*, *Aspergillus* and *Byssoschlamys* which

grow on fruit [41]. Also Ochratoxin-A is one of mycotoxin which produced by species of *Penicillium* and *Aspergillus* [3]. Presence of Ochratoxin A in fruit juice point to poor agriculture and harvesting practice, when lead to a physiological damage in fruit tissue. Patulin and Ochratoxin A are the mycotoxin which associated more frequently with fruit juice [15,44].

The concentration of mycotoxin (Patulin and Ochratoxin-A) in raw mango and guava fruits before rinsing by hot water and after pasteurization processes mango and guava pulp are presented in table 3.

Mycotoxins	Process step		LOQ (µg /kg)	ML (µg /kg) (Codex, 2019b) [12]
	Before rinsing (µg /kg)	After pasteurization processes (µg /kg)		
A) Mango samples				
Patulin	N.D	N.D	10	50
Ochratoxine A	N.D	N.D	0.5	5
B) Guava samples				
Patulin	N.D	N.D	10	50
Ochratoxine A	N.D	N.D	0.5	5

**Table 3:** Determination of mycotoxin in mango and guava fruit before rinsing and after pasteurization processes of pulp.

LOQ: Limit of Quantification; N.D: Not Detected; M.L: Maximum Level

As shown in table (3), Patuline and Ochratoxin A were not detected in all samples of mango and guava fruits before rinsing by hot water and after pasteurization processes mango and guava pulp. These results are interesting as several studies have cited detected Patulin in apple from Pakistan at 396 µg/kg [25]. Also, (Abu-Bakar, *et al.* 2014) mentioned that, Patulin were detected in apple, grape and mango juice in Malaysia at 0.5-100 µg/kg [1]. Also, these results are interesting as several studies have cited occurrence of Ochratoxin A in cherries at 2.71 µg/kg (Engelhardt, *et al.* 1999) and also detected that, peaches and apples were contaminated with Ochratoxin A [18]. The absence of Patulin and Ochratoxin A might be an indication of good fruit because the process of receiving fruits is carried out with high efficiency and according to specific specifications, and it is not allowed to receive fruits that contain mechanical damage, insect or fungal infections. Another probable explanation could be use of fungicide by farmers. As mentioned above, at (Table 1) fungicides were detected in mango and guava fruits.

**Conclusion**

Finally, it can be concluded that pesticide residues, heavy metals (lead, cadmium, mercury, and arsenic) and mycotoxins (Patulin and Ochratoxin A) in mango and guava fruits before rinsing by hot

water and after pasteurization processes of mango and guava pulp not exceeded the permissible limit which recommended by Codex, 2019 also observed, a decrease in the concentration of some pesticide residues and heavy metals after pasteurization process mango and guava pulp, while the presence of mycotoxins (Patulin and Ochratoxin A) was not indicated before rinsing or after pasteurization. The results showed that, the fruit supplied to the company is high quality, thus ensuring the production of a safe product in which pesticide residues, heavy metals (lead, cadmium, mercury, and arsenic) and mycotoxins (Patulin and Ochratoxin A) do not exceed the maximum limits that have been recommended by the Codex Authority, 2019.

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