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Estimation of toxic, trace and essential metals (Pb, cd, Fe, Zn, Mn, Cu, Mg, K) in fruit and vegetable product (jam, ketchup, pickles) by atomic absorption spectrophotometer

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Abstract
With growing international trade, food safety has been emerged as an important global issue. In the context of current global challenges in food safety and security, the study was conducted to assess essential element (K, Mg), trace(Fe, Cu, Zn, Mn,) and toxic(Pb, Cd) in some commercial and local brand fruit and vegetable based food product like Jam, Ketchup and Pickle by using Atomic Absorption Spectrophotometer(AAS). Sample were prepared prior to measurement followed by wet ashing method. The result of Fe, Cu, Mn, Zn, Mg and K in the range of 2.073-6.428mg/100g, 0.103-0.297mg/100g, 0.208-0.970mg/100g, 0.325-2.015mg/100g, 18.016-25.666 mg/100g and 90.594-221.330 mg/100g respectively. Lead and Cadmium was not detected in any of Jam, Ketchup, Pickle samples. The essential element K, Mg found more rather than trace metal Fe, Cu, Mn, Zn. The obtained result of the sample were within the acceptable range approved by WHO and do not poses any health hazard.

Keywords: Essential element, trace element, toxic element, atomic absorption spectrophotometers (AAS)

Introduction
Fruits and vegetables are tremendous importance in the diet of human being; contain all the nutrients such as vitamins, minerals, fibers, phenolic compound, flavonoids and other antioxidant which is required for human health.

Fruit and vegetables are perishable in nature and seasonally available, needs to be preserved for lean/ off season by making products like Jam, Ketchup and pickles etc. to enhance the shelf life. Nowadays, a consumption fruit and vegetable based product are increased in all group of people and also served as Ready to Eat (RTE). It is well- known that a balanced diet is essential for maintaining good health. Hence, in respect of element, the nutritional value of foods is an important aspect. Importantly, it observed to positive and negative roles of essential, trace element in human health. Most of the fruit and vegetable based product consumed in the form of jam, ketchup and pickle. Products including jam, ketchup, pickle may give significant exposure routes to metal contamination (such from processing, water, raw material etc) and the effect of some metals such as lead and cadmium at low levels cause serious health problems such as cardiovascular, nervous, kidney as well as bone diseases (Godt *et al*. 2006) [4] Other elements such as aluminum, zinc, iron and copper are very important to human health at very low levels but can lead to toxicity once ingested in high doses. The essential metals potassium and magnesium can also have harmful effects when their intakes exceed the recommended quantities significantly. (National research council 1989) [13]

Exposure of consumers to metals and related health risks, DRI (Dietary Reference Intakes) [12] summaries recommended daily intake metal and there tolerable upper intake level. According to this maximum tolerable upper intake level of iron (40-45mg/d), copper 10mg/d, zinc 40mg/day, manganese 11mg/d, magnesium 350mg/d for normal adult. And if the dose larger than tolerable limit than stomach upset, constipation and blackened stools, intestinal problems or heart rhythm disorder etc.

It is however necessary to ensure the quality of these products, in order to ensure maintenance of proper quality, Indian Standard set the criteria for preparation of Jam (IS 5861-1993) [5], Ketchup (lS 3882-1966) [6], and Pickles (IS 3501- 1966) [7] and maximum permissible limit in Pb 2.5 mg/100g, Cu 30mg/100g, zinc 19 mg/100g.

So the study was conducted to assess essential (K, Mg), trace (Fe, Cu, Zn, Mn,) and toxic

(Pb, Cd) element in some commercial and local brand fruit and vegetable based food product like Jam, Ketchup and Pickle by using Atomic Absorption Spectrophotometer (AAS).

Atomic absorption spectroscopy (AAS) is a common technique used in many chemical measurements requiring a high degree of precision and accuracy, such as food & drug safety, clinical diagnostics and environmental sampling. In the present study AAS has been chosen for analysis because of its high sensitivity, wide dynamic range, relatively low risk of interference.

Material and method

Chemicals and instrumentation

We used Atomic Absorption Spectrophotometer of AAnalyte800 (Producer company Perkin Elmer Inc. USA; year of producer-2003) for determination of metal concentration. The spectrometer fully operated via computer, using software support winlab32. The stock solutions (1 mg/L and 0.1 mg/L) of the metals were prepared from High Purity Single Element Standard Solutions (1000 mg/L) were purchased from Merck. And further diluted with deionized water (>18.2 MΩ/cm) obtained from a Millipore MilliQ. Suprapur HNO_3 (65 %, w/w) were purchased from Merck.

Sampling

Sample was collected randomly from local market. Out of five samples three are commercial (coded as1, 2 and 3) and two local branded (4, 5).Sample were coded to overcome the sampling biases.

Destruction of organic matter

Wet ashing and preparation of sample

In order to obtain homogenous mixture, sample mixed in high speed blender. Transfer suitable quantity of sample (3g) into a macro-kjeldahl digestion flask. Add 20 ml concentrated nitric acid and makeup to 20 ml water (depending on the water content of sample). Boil the contents of the flask to reduce the volume to 20 ml. Cool the solution, add 10 ml of conc. sulphuric acid (H_2SO_4) and boil again.

Add further small quantities of nitric acid whenever the contents begin to blacken. When the addition of nitric acid is no longer necessary (i.e. when the liquid no longer blackens) continue heating till white fumes are evolved. At this stage, cool the solution and add 10 ml of saturated ammonium oxalate solution and again boil until copious white fumes are again produced. The solution was left to cool and then rinsed with 10% nitric acid. The solution suspension was filtered and the filtrate made up to the volume of100 ml with 10M nitric acid. (FSSAI, 2015) [2].

Determination

Flame procedures,

We used the hollow cathode lamp to assays of K, Mg, Mn, Zn, Cu, and Fe at 766.5nm, 202.2nm, 279.8nm, 213.9nm, 248.3nm and 324.7nm respectively.

Graphite furnace program

We used the graphite furnace program to assays Pb and Cd at 283.3nm and 228.8nm respectively.

Result and discussion

The concentration of metals in Jam, Ketchup and Pickle, reported in (Table 1 and fig1), (Table 2 and fig2) and (Table3 and fig3) respectively.

Table 1: Average concentration of metals in (Mean ± SD, n = 3, mg/ 100g) in Jam. (ND-Not detected).

Sample	Pb	Cd	Fe	Mn	Cu	Zn	K	Mg
1	ND	ND	6.428±0.299	0.268±0.003	0.114±0.001	1.734±0.021	220.973±24.87	20.001±0.61
2	ND	ND	5.217±0.2	0.229±0.007	0.103±0.005	2.015±0.048	217.215±20	19.021±0.70
3	ND	ND	4.012±0.134	0.251±0.001	0.108±0.001	1.116±0.028	221.330±25.31	20.471±0.42
4	ND	ND	3.877±0.317	0.220±0.01	0.112±0.001	1.567±0.036	219.536±10.74	21.333±0.28
5	ND	ND	5.625±0.213	0.208±0.001	0.107±0.001	1.144±0.051	220.768±15.46	22.721±0.67

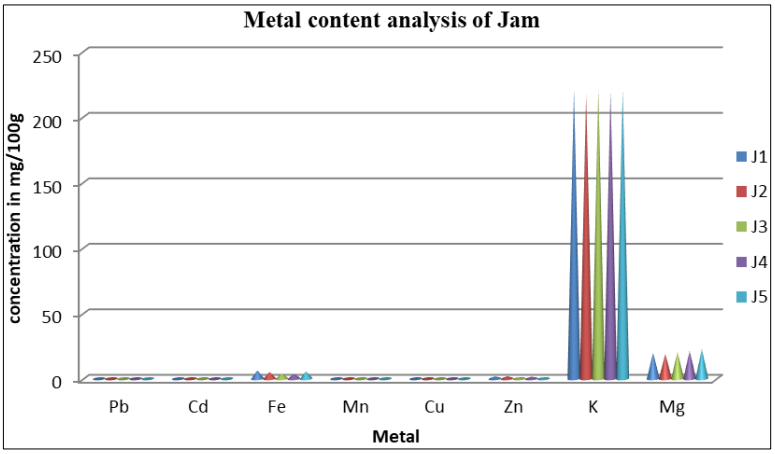

Fig 1: Concentration of metals in Jam.

Table 2: Average concentration of metals (Mean±SD, n = 3, mg/ 100g) in Ketchup. (ND-Not detected).

Sample	Pb	Cd	Fe	Mn	Cu	Zn	K	Mg
1	ND	ND	3.156±0.084	0.97±0.014	0.215±0.008	0.387±0.006	141.661±5.214	24.332±0.91
2	ND	ND	2.703±0.061	0.100±0.027	0.217±0.001	0.468±0.004	134±5	21.338±0.62
3	ND	ND	3.473±0.058	0.116±0.01	0.200±0.007	0.442±0.004	129.333±6.176	25.666±0.81
4	ND	ND	2.736±0.021	0.95±0.021	0.204±0.002	0.325±0.001	159.531±4.883	22.665±0.3
5	ND	ND	2.703±0.034	0.92±0.018	0.198±0.005	0.378±0.005	139.547±5.114	22.376±0.76

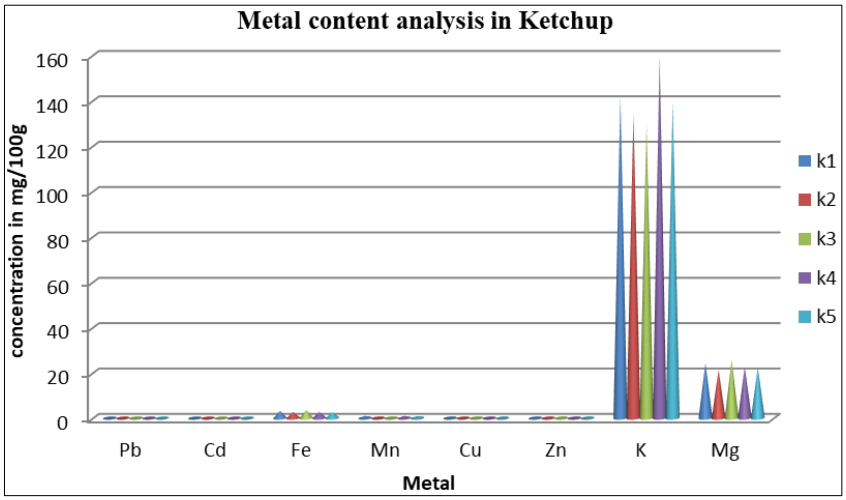

Fig 2: Concentration of metals in Ketchup.

Table 3: Average concentration of metals (Mean±SD, n = 3, mg/ 100g) in pickle samples. (ND-Not detected).

Sample	Pb	Cd	Fe	Mn	Cu	Zn	K	Mg
1	ND	ND	4.532±0.05	0.373±0.01	0.297±0.007	0.904±0.1	126.322±10.96	21.916±0.67
2	ND	ND	4.997±0.047	0.381±0.005	0.243±0.014	0.830±0.1	115.857±5.321	18.453±0.81
3	ND	ND	4.211±0.081	0.343±0.003	0.240±0.002	0.934±0.007	130.451±5.114	19.763±0.931
4	ND	ND	5.018±0.01	0.321±0.007	0.210±0.01	0.812±0.004	128.746±8.413	18.016±0.9
5	ND	ND	5.001±0.069	0.301±0.01	0.228±0.004	0.747±0.01	90.594±10.731	19.752±0.542

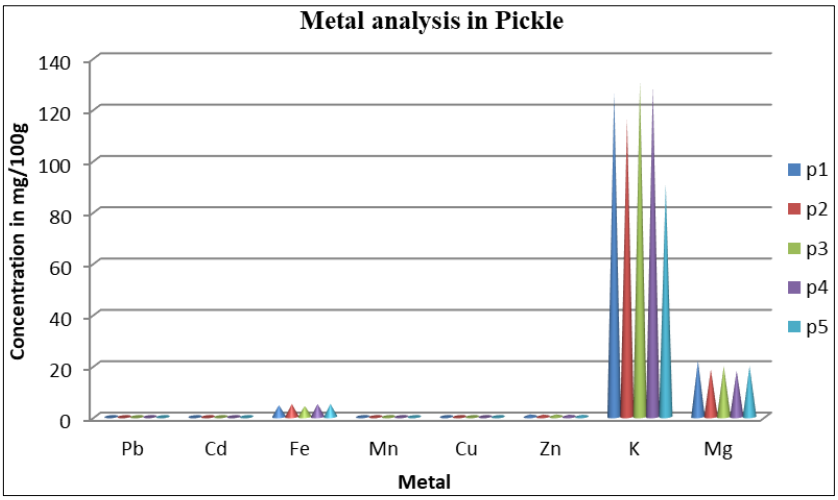

Fig 3: Concentration of metals in Pickle.

Lead (Pb)

WHO (1972) [18], recommended the weekly intake of Pb from all sources should not exceed 0.05 mg.kg-1 body weight for lead. Airborne lead can be deposited on soil, water and plants thus reaching human via the food chain. Lead is accumulated in the skeleton and cause renal tubular damage and may also give rise to kidney damage, nervous disorder, high blood pressure and muscle pain (Lokeshappa *et al.* 2012) [11]. Similarly Indian standard (5861, 3882 and 3501) [5, 6, 7] set the maximum permissible level in jam, ketchup, pickle for Pb was 2.5 mg/kg. From Table 1, fig1, Table 2, fig2 and Table 3, fig3 lead(Pb) was not detected(ND) in any brand of Jam, Ketchup and Pickle. Comparing the result with the heavy metal limitation set by WHO, none of the samples exceeded the permissible level.

Cadmium (Cd)

Table 1, fig1, Table 2, fig2 and Table 3, fig3 cadmium (Cd) was not detected in any brand of Jam, Ketchup and Pickle. Nordic Council of Ministers (2003) [14]. Suggested the weekly intake of metals from all sources should not exceed 0.075 mg.kg-1 body weight for cadmium. It does not have a role in biological process in living organisms (Godt *et al.* 2006) [4]. Thus, even in low concentration, cadmium could be harmful to living organisms. Cd poisoning in man could lead to cancer of the lungs, renal damage, bone disorder and osteoporosis etc. (Järup *et al.* 1998) [9]. Comparing the result with the heavy metal limitation set by Nordic Council of Ministers [14], none of the samples exceeded the permissible level.

Copper-(Cu)

DRI (Dietary reference Intake) [12] gives the tolerable upper intake level, 10mg/d for copper for normal adult. Indian standard (5861, 3882 and 3501) [5, 6, 7] also set the maximum permissible level in jam, ketchup, pickle for Cu is 30mg/kg. In human body, copper is maintained in homeostasis (Jesse and Mary, 2004) [10]. If the intake of copper exceeds the range of the human tolerance, it would cause toxic effects such as

hemolysis, jaundice and even death. Most recently, the study indicates that the overload of common copper changes in lipid profile, oxidative stress, renal dysfunction (Galhardi *et al*., 2004) [3] Concentration of copper in Jam was 0.103-0.114mg/100g (Table 1and fig1), Ketchup 0.198-0.217mg/100g (Table2 and fig 2), Pickle 0.210-0.297mg/100g (Table3 and fig.3) was estimated. Comparing the result of copper with the metal limitation set by IS standard and DRI, concentration of Cu was below the permissible limit.

Zinc

Concentration of Zn in Jam was 1.144-2.015mg/100g (Table 1, fig1), Ketchup 0.325-0.468mg/100g (Table2, fig2), Pickle 0.747-0.934mg/100g (Table 3, fig3) was estimated. The tolerable upper intake level set by DRI [12] was, 40mg/d for zinc for normal adult. Zinc is essential to all organisms and has an important role in metabolism, growth, development. It is an essential co-factor for a large number of enzymes in the body. Excessive intake of zinc cause lethargy, focal neuronal deficits, respiratory disorder, nausea, vomiting, epigastric pain diarrhea, elevated risk of prostate cancer (Plum *et al*. 2010) [15]. Indian standard (5861, 3882 and 3501) [5, 6, 7], also set the maximum permissible level in jam, ketchup, pickle for Zn is 50mg/kg. The amount of zinc concentration was below the permissible limit in all Jam, Ketchup and Pickle samples.

Manganese (Mn)

Manganese is essential element required for various biochemical processes. The kidney and liver are the main storage places for the manganese in the body. Mn can improve immunity and stimulate anti-toxin synthesis (Qing-hua *et al*. 2012) [16]. Excessive intake of manganese hinders the iron absorption. Excess Mn in body can cause serious side effect, including symptoms resembling Parkinson disease such as shaking (tremors). DRI (Dietary reference Intake) [12] gives the tolerable upper intake level, 11mg/d for manganese for normal adult. Concentration of Mn in Jam was 0.208-0.268mg/100g (Table 1, fig1), Ketchup 0.9-0.970mg/100g (Table 2, fig2), Pickle 0.301-0.381mg/100g Table3, fig3 was estimated. By comparing the concentration of Mn with tolerable upper intake level set by DRI, none of sample above the permissible limit.

Iron (Fe)

Concentration of Fe in Jam was3.877-6.428 mg/100g (Table 1 andfig1), Ketchup 2.073-3.473mg/100g (Table2 and fig2), Pickle 4.211-5.018mg/100g (Table 3 andfig3) was estimated. DRI (Dietary reference Intake) [12] gives the tolerable upper intake level, 40-45 mg/d for iron for normal adult. Iron (Fe) is an essential element in man and plays a vital role in the formation of hemoglobin, oxygen and electron transport in human body. Excessive intake of iron can cause stomach upset, constipation and blacked stool. Excessive iron can be damaging to the gastrointestinal system. Symptoms of iron toxicity include nausea, vomiting, diarrhea and stomach pain. Over time, iron can accumulate in the organs, and cause fatal damage to the liver or brain. (Catherine S, 2018) [1]. Comparing the result of iron with the metal limitation set Dietary Reference Intakes none of the sample exceeded the permissible level hence they are safe for human consumption.

Magnesium-(Mg)

Concentration of Mg in Jam was 19.021-22.721mg/100g (Table 1 andfig1), Ketchup 21.338-25.666mg/100g (Table2 and fig2), Pickle 18.016-21.916mg/100g (Table3 andfig3). DRI (Dietary reference Intake) [12] recommended daily intake of metal from all sources should not exceed 350 mg/d for magnesium. Mg is present in small concentration in all cell and is requirement for cellular metabolism. It is also present in bone along with calcium. Mg is also important to have a role in cardiovascular disease. Excessive intake causes stomach problem and diarrhea, lethargy, nausea and vomiting, irregular heartbeat, cardiac assets etc. (National academic press, 1989) [13]. Comparing the result with the metal limitation set by DRI, none of the samples exceeded the permissible level.

Potassium-(K)

DRI (Dietary reference Intake) [12] recommended daily intake of metal from all sources should not exceed 3500 mg for potassium. Potassium (K) is an essential nutrient needed for maintenance of total body fluid volume, acid and electrolyte balance, and normal cell function. (Young, 2001) [19]. Potassium toxicity has been studied in relation to the use of high doses of salt substitutes. The symptoms described have been chest tightness, nausea and vomiting, diarrhoea, hyperkalaemia, shortness of breath and heart failure (Restuccio, 1992) [17]. Concentration of K in Jam was 217.215-221.330 mg/100g (Table1, fig1), Ketchup 129.333-159.531mg/100g (Table2, fi2), Pickle 90.594-129.322mg/100g (Table3, fig3) estimated. By comparing the concentration of Potassium (K) with the metal limitation set Dietary Reference Intakes none of jam, ketchup and pickle sample exceeded the permissible level.

Conclusion

The study shows that the levels of toxic, trace and essential element determined in fruit and vegetable based samples are within the acceptable range and do not poses any health hazard to the their consumers; hence they are safe for human consumption and there was no adverse effect on human health.

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