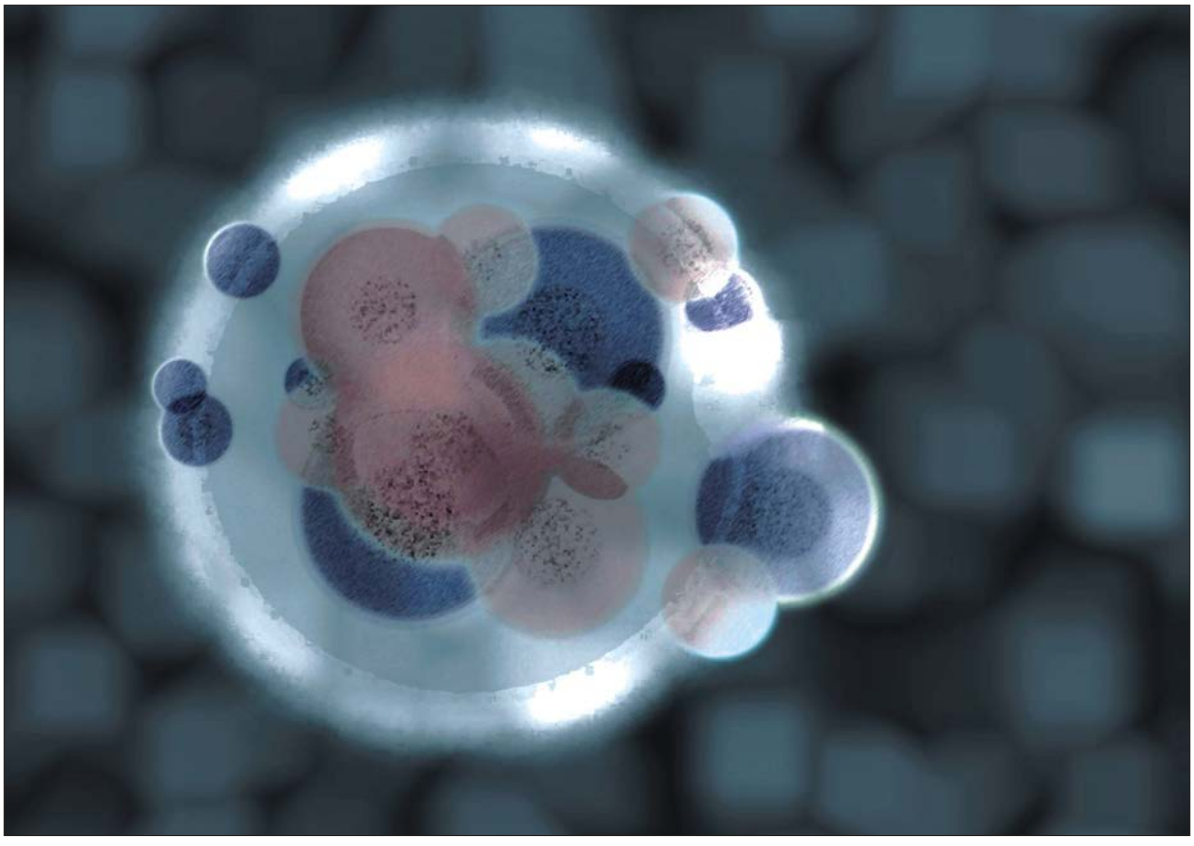

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Determination of Electrolytes (Na, K and Li) by Flame Photometry and Separation of Trace Metals (Ni, Co and Fe) Using Cation Exchange Resin in Selected Sweets of Different Shops of Karachi City

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Abstract: Concentration of electrolytes (Na, K and Li) and trace metals (Fe, Ni and Co) was estimated in selected sweets purchased from eight famous shops of Karachi city. For the determination of electrolytes samples of eight different sweets (Gulab Jaman, Chumchum, Colored Chumchum, Bhashani Chumchum, Khopry ki Mithai, Qalaqand, Besin ka Luddo and Patisa) were analyzed through flame photometry. For the determination of Ni, Co and Fe solution of the sample of Gulab Jaman from six selected shops were passed through cation exchange resin. It was observed that all the three metals required 6 M HNO_3 for their complete elution through Dowex 50-X8 strong cation exchange resin, Ni required 30 mL of the acid, Co required 15 mL and Fe required 45 mL of 6 M HNO_3, while metals were determined through atomic absorption spectroscopy. The results for electrolytes were compared with recommended dietary allowances (RDA) values and it was observed that almost all the sweets contain high concentration of electrolytes. Therefore present study infer that the ingredients, which are used for the preparation of those sweets, are rich in electrolytes. Whereas results for iron and nickel are in accordance with RDA values. Results for cobalt are seemed to be higher than the recommended values of RDA.

Key words: Electrolytes, trace metals, cation exchange chromatography, flame photometer, flame atomic absorption spectrometer (FAAS)

INTRODUCTION

Heavy metals in traces are considered to be essential for plants and animals nutrition and serve some useful biological functions and hence formed the basis of life. Each living cell, including DNA, depends on metal for its structure and function. They are indispensable for their integrity, regulation and catalysis. Transition metals centered in proteins have a variety of functions i.e. binding and reduction of oxygen, they are also responsible for the functions like electron transfer reactions, group transfer reactions such as DNA transcription, control of macromolecular structure and molecular recognition among ATPases, nucleotides and their substrates[1,2].

Recent investigations indicate that metals may play a significant role against the variety of degenerative diseases and processes. However, all the heavy metals when present in excessive amounts have been found to be toxic for animals[3]. Too much of the trace element can lead to the imbalances resulting in diseases, rather than the absence of diseases[4].

Electrolytes are necessary for the formation of body fluids, cellular growth and healing, energy, muscle tone and nerve function. They are often vital in adsorption, function and effectiveness of certain vitamins[4]. Main groups of sodium, potassium, magnesium and calcium are main agents in signaling, triggering and control of cellular processes[5]. Lithium is essential for psychiatric disorder[6].

Humans may be exposed to environmental metals through different pathways. These contaminants could be consumed directly from drinking water or through the food. Food is also the main source of electrolytes for our body[7]. Although the food habits greatly differ from society to society hence the threat through these contaminants also varies. In Pakistani culture sweets have got the traditional importance while exchanging facilitations especially at the occasions of marriages and Eid. Owing to the high potential of sweets to contribute to the ailments from their use therefore present investigation was carried out for determining the presence of certain heavy metals and electrolytes in different selected and commonly used sweets. These are sweets are available in various famous sweet marts situated in Karachi city.

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These are composed of various ingredients such as milk, sugar, flavors, food colors and water. The origin of these ingredients may be synthetic or natural and hence their toxic effect on human health is not to be ignored.

MATERIALS AND METHODS

Sample collection: Samples of eight different and commonly used sweets such as Gulab Jaman, Chumchum, Colored Chumchum, Khopry ki Mithai, Bhashani, Qalaqand, Besin ka Luddo and Patisa were collected from the eight different famous sweet meet marts situated in the different regions of Karachi. Sampling was repeated thrice with the difference of two weeks.

Preparation of sample solution: Soon after the collection of samples, they were weighed and solutions of samples were prepared.

Digestion method: Ten g of each sweet was digested by using a mixture of 5 mL HNO_3 (65%) and 1 mL $HClO_4$(70%). Digestion was carried out in beakers and performed at 40-50°C for about 5 min on a hot plate. Digestion procedure was performed in fuming hood for safety purpose. After cooling each sample was filtered using watman-40 filter paper and then each sample was diluted with 5% HNO_3 (V/V) to 50 mL in 50 mL volumetric flask prior to analysis.

Storage of sample solution: Sample solutions were stored in washed and dried plastic bottles.

Reagents
HNO_3 (65%)
$HClO_4$ (70%)
Deionized water
Dowex 50-X8 strongly acidic cationic resin

Instruments
Perkins Elmer 2380
Hollow cathode lamps
Sherwood flame photometer 410
An electrical balance
Hot plate

Preparation of standards: Stick standards of 500 ppm of Fe, Co, Ni, Na, K and Li were prepared by dissolving specifically weighed quantity of nonhygroscopic, high quality salts such as $Fe(NH_4)_2(SO_4)_3.7H_2O$, $NiSO_4.7H_2O$, $CoCl_2.6H_2O$, NaCl, KCl and LiCl.

Working standards: Working standards were prepared by diluting stock solutions from 500 to 100 ppm and then from 100 ppm to required concentration. The standards, samples and blank were aspirated into a fuel lean air-acetylene flame under optimum working conditions (Table 1).

Table 1: Working parameters for atomic absorption spectrometer

Working parameters (AAS)	Ni	Co	Fe
Wave length (nm)	232.00	240.70	248.30
Lamp current (mA)	26.00	26.00	26.00
Slit width	0.70	0.70	0.70
Acetylene flow rate (mL/min)	20.00	20.00	20.00
Air flow rate (mL/min)	40-50	40-50	40-50
Air pressure (Psi)	60-80	60-80	60-80
Acetylene pressure (Psi)	80.00	80.00	80.00

Preparation of column: Dowex 50-X8 strongly acidic cation exchange resin was agitated with hot 6 M HCl to remove any impurity. The excess acid was washed off with deionized water and then slurry of the resin was prepared. Column already plugged with glass wool was filled with water and then slurry of resin was introduced into the column. At the same time the water was drained off from the column. The water level was maintained 2 cm above the resin.

Analytical method: A standard solution of Ni, Fe and Co was passed through the column at the linear flow rate of 1 mL/min. the elution process was carried out with 1 M 10 M HNO_3. The effluents were collected in a batch of 5 mL in each calibrated vials. Sample solutions that were eluted with 6 M HNO_3 were also passed through resin and collected as standard solution was collected.

RESULTS AND DISCUSSION

Sodium: It was observed from Table 2 that sweets of United King contain sodium within the range of 479-3252 ppm. Such a high concentration of sodium in sweets was due to the ingredients used for their preparation mainly milk and water. Table 2 also shows that Khopre ki Mithai contains higher concentration of sodium that may be due to its main constituent coconut. Almost all values are higher than RDA values. Concentration of sodium in Qalaqand was exceptionally high i.e. beyond the range of 3000 ppm, which may be due to the use of large quantity of milk. Concentration of sodium (Table 3) in Nirala sweets was found within the range of 230-3331 ppm. Table 4 shows that the concentration of sodium in the sweets of S. Abdul Khaliq ranges between 285-3625 ppm. Sweets from Fresh Well contain sodium in the range of 230-3653 ppm (Table 5). It is observed that these sweets contain sodium in the range of 330-3716 ppm (Table 6). Results from the

Table 2: Concentration of electrolytes in sweets of United King Sweet Meet Mart

Samples	Na (ppm)	K (ppm)	Li (ppm)
Gulab Jaman	1945.80	2168.70	114.750
Chumchum	479.10	233.60	80.260
Colored Chumchum	515.69	356.21	81.800
Khopre Ki Mithai	1970.06	2356.44	150.160
Bhashani Chumchum	801.72	2071.95	0.000
Qalaqand	3251.91	606.60	191.130
Besin Ka Ludoo	934.47	3706.70	121.006
Patisa	1388.96	2783.74	160.840

Table 3: Concentration of electrolytes in sweets of Nirala Sweet Meet Mart

Samples	Na (ppm)	K (ppm)	Li (ppm)
Gulab Jaman	1783.70	2101.33	91.63
Chumchum	232.95	174.71	0.00
Colored Chumchum	276.91	346.13	76.84
Khopre Ki Mithai	-------	--------	-----
Bhashani Chumchum	2085.60	2799.50	119.18
Qalaqand	3330.50	4585.50	199.11
Besin Ka Ludoo	-------	--------	-----
Patisa	845.12	1835.95	87.43

Table 4: Concentration of electrolytes in sweets of S.Abdul Khaliq Sweet Meet Mart

Samples	Na (ppm)	K (ppm)	Li (ppm)
Gulab Jaman	1631.97	2654.70	83.83
Chumchum	285.56	237.96	0.00
Colored Chumchum	493.70	263.30	0.00
Khopre Ki Mithai	2755.65	3726.12	89.86
Bhashani Chumchum	436.50	1926.50	61.13
Qalaqand	3624.70	3828.76	159.73
Besin Ka Ludoo	1191.73	3712.70	148.96
Patisa	792.36	1421.60	116.52

Table 5: Concentration of electrolytes in sweets of Fresh Well Sweet Meet Mart

Samples	Na (ppm)	K (ppm)	Li (ppm)
Gulab Jaman	1367.73	1396.80	77.52
Chumchum	289.20	241.02	0.00
Colored Chumchum	299.17	241.64	0.00
Khopre Ki Mithai	2382.20	3394.67	156.66
Bhashani Chumchum	880.17	997.50	118.30
Qalaqand	3652.60	3181.81	197.60
Besin Ka Ludoo	1169.90	1462.40	81.63
Patisa	229.89	362.99	76.91

Table 6: Concentration of electrolytes in sweets of Darbar-e-Shirin Sweet Meet Mart

Samples	Na (ppm)	K (ppm)	Li (ppm)
Gulab Jaman	1963.80	2296.13	120.85
Chumchum	330.35	117.98	0.00
Colored Chumchum	363.60	181.80	0.00
Khopre Ki Mithai	3016.80	3272.07	150.84
Bhashani Chumchum	486.44	370.60	0.00
Qalaqand	3715.4	3709.71	188.92
Besin Ka Ludoo	877.87	877.87	117.05
Patisa	325.4	522.94	0.00

shops of Mahmood, Dilpasand and Fresco are listed in Table 7, 8 and 9, respectively. The data shows that the sodium contents of the sweets of these three shops was in the range of 325-3084, 451-3025 and 633-3251 ppm, respectively.

When results were compared with RDA values (Table 16) observed that almost all sweets contain higher concentration of sodium.

Table 7: Concentration of electrolytes in sweets of Mahmood Sweet Meet Mart

Samples	Na (ppm)	K (ppm)	Li (ppm)
Gulab Jaman	1912.20	3013.20	115.90
Chumchum	426.26	292.30	0.00
Colored Chumchum	500.40	381.26	0.00
Khopre Ki Mithai	1643.11	3624.16	81.11
Bhashani Chumchum	324.70	417.49	57.98
Qalaqand	3083.40	4534.37	199.51
Besin Ka Ludoo	1102.30	3169.80	119.16
Patisa	524.36	686.67	0.00

A general trend is observed for sodium that Khopry ki Mithai and Qalaqand contain highest concentration of sodium independent of the shop because they are mainly prepared from milk and milk contains a considerable amount of sodium[8,9]. Coconut in Khopry ki Mithai may also responsible for its high sodium contents.

Potassium: It can be seen from Table 2 that the sweets of United King contain 233-3707 ppm of potassium. Concentration of potassium in Nirala sweets was found within the range of 174-2799.5 ppm for almost all the sweets but for Qalaqand the concentration of potassium was found to be extremely high i.e. beyond 45000 ppm, (Table 3).

Table 4 shows that the concentration of potassium in S. Abdul Khaliq's sweets is ranges between 238-3829 ppm. In the sweets of Fresh Well concentration of potassium is found in the range of 241-3395 ppm (Table 5). In Table 6 values for the potassium contents of the sweets of Darbar-e-Shirin shows that the concentration of potassium in those sweets ranges between 117-3710 ppm. In Table 7, 8 and 9 concentration of potassium in the sweets of Mahmood, Dilpasand and Fresco sweet marts showed that the sweets contain potassium within the range of 291-4535, 281-39467 and 238-4540 ppm, respectively.

When the results for potassium concentration in sweets were compared with RDA (Table 16) values it was observed that all the sweets contained higher than the values proposed by RDA.

It can be observed from Table 2-7 that Khopry ki Mithai and Qalaqand from almost all the shops contain highest concentration of potassium. The reason is same as that for sodium i.e. the use of high quantity of milk for their preparation, which contains considerable amount of potassium also[8,9].

Lithium: Table 2 shows that the sweets of United King sweet meet mart contain 80-191 ppm of lithium. Sweets of Nirala sweet meet mart contain lithium within the range of 76.8-199.12 ppm (Table 3). Results of the sweets purchased from S. Abdul Khaliq's shows that the concentration of lithium in these sweets ranges between 61-159.8 ppm (Table 4). It can be seen from Table 5 that

Table 8: Concentration of electrolytes in sweets of Dilpasand Sweet Meet Mart

Samples	Na (ppm)	K (ppm)	Li (ppm)
Gulab Jaman	2148.90	2387.70	89.54
Chumchum	451.27	281.78	0.00
Colored Chumchum	476.37	297.73	0.00
Khopre Ki Mithai	2953.89	3945.60	148.30
Bhashani Chumchum	1068.80	519.95	57.80
Qalaqand	3026.94	3545.18	177.80
Besin Ka Ludoo	1019.76	2222.85	148.95
Patisa	459.04	753.30	58.85

Table 9: Concentration of electrolytes in sweets of Fresco Sweet Meet Mart

Samples	Na (ppm)	K (ppm)	Li (ppm)
Gulab Jaman	1887.96	2840.40	84.54
Chumchum	632.99	238.25	0.00
Colored Chumchum	1317.10	581.90	61.26
Khopre Ki Mithai	3250.80	4539.63	174.15
Bhashani Chumchum	1237.40	785.28	178.50
Qalaqand	3102.20	3933.35	117.06
Besin Ka Ludoo	1278.05	2318.30	79.18
Patisa	914.31	1086.40	71.64

Table 10: Separation of trace metals from sample solution of Gulab Jaman of United King Sweet Meet Mart

Vol. 6 M HNO_3 (mL)	Fe (ppm)	Ni (ppm)	Co (ppm)
5	3.75	0.92	0.39
10	2.30	0.83	0.40
15	1.00	0.05	7.42
20	0.50	0.42	2.81
25	4.67	0.37	1.10
30	13.57	2.14	0.85
35	1.81	4.18	0.10
40	1.16	1.14	0.05
45	2.45	9.55	0.05
50	0.00	0.50	0.02

Table 11: Separation of trace metal from sample solution of Gulab Jaman of Nirala Sweet Meet Mart

Vol. 6 M HNO_3 (mL)	Fe (ppm)	Ni (ppm)	Co (ppm)
5	2.22	0.09	0.47
10	2.27	0.14	0.47
15	1.95	0.05	7.47
20	1.20	0.05	2.55
25	0.56	1.73	1.15
30	12.14	7.46	0.49
35	0.74	7.75	0.44
40	0.71	7.75	0.11
45	0.50	9.64	0.04
50	0.20	0.05	0.01

Table 12: Separation of trace metals from sample solution of Gulab Jaman of Mahmood Sweet Meet Mart

Vol. 6 M HNO_3 (mL)	Fe (ppm)	Ni (ppm)	Co (ppm)
5	1.79	0.96	0.80
10	3.09	0.76	0.90
15	2.75	0.81	7.80
20	2.27	0.96	0.81
25	1.16	0.99	0.81
30	13.63	1.05	0.81
35	1.84	4.81	0.77
40	0.77	6.76	0.58
45	0.65	9.62	0.03
50	0.42	1.71	0.01

Fresh Well's sweets contain 76.9-197.6 ppm lithium. Sweets select from Darbar-e-Shirin sweet mart contain 117-189 ppm of lithium (Table 6). Table 7 and 8 show the

Table 13: Separation of trace metals from sample solution of Gulab Jaman of Darbar-e-Shirin Sweet Meet Mart

Vol. 6 M HNO_3 (mL)	Fe (ppm)	Ni (ppm)	Co (ppm)
5	0.23	0.41	0.51
10	0.33	0.22	0.54
15	0.37	0.26	7.51
20	0.28	0.36	4.50
25	9.68	1.59	0.52
30	10.29	1.05	0.52
35	2.14	1.05	0.44
40	0.42	5.04	0.25
45	0.33	9.36	0.07
50	0.14	0.03	0.07

Table 14: Separation of trace metals from sample solution of Gulab Jaman of FreshWell Sweet Meet Mart

Vol. 6 M HNO_3 (mL)	Fe (ppm)	Ni (ppm)	Co (ppm)
5	0.90	0.31	0.70
10	0.85	0.53	0.72
15	0.71	0.77	7.71
20	8.47	0.98	3.73
25	0.37	2.59	0.71
30	10.88	2.59	0.71
35	1.04	1.67	0.68
40	0.85	6.41	0.66
45	0.76	9.31	0.05
50	0.27	0.55	0.05

Table 15: Separation of trace metals from sample solution of Gulab Jaman of Dilpasand Sweet Meet Mart

Vol. 6 M HNO_3 (mL)	Fe (ppm)	Ni (ppm)	Co (ppm)
5	0.87	0.48	0.89
10	0.88	0.63	1.87
15	0.83	0.77	7.87
20	0.06	0.77	6.88
25	8.89	1.53	1.57
30	10.95	2.58	0.89
35	0.35	1.63	0.89
40	0.64	5.58	0.89
45	0.54	9.77	0.01
50	0.50	0.43	0.01

Table 16: RDA values

Elements	RDA values
Na	500 mg/day
K	200 mg/day
Li	2-3 mg/day
Fe	50-400 mg/day
Ni	300-600 mg/day
Co	0.1 mg/day

results from the shops of Mahmood and Dilpasand, sweets from these shops contained lithium within the range of 57.9-199.52 and 57.8-177.8 ppm, respectively. Results shows that Fresco's sweets contain lithium in the range of 61.2-178.5 ppm (Table 9).

The way through which lithium can enter the sweets may be the ingredients use for the preparation of these sweets especially water which contains lithium[10]. Lithium is also use in alloys[11], hence those utensils which are used during the preparation of sweets if contains lithium are also responsible for the lithium contents of sweets.

Results for lithium are also observed higher than the RDA recommended values (Table 16).

Iron: It can be seen from Table 10-15 that iron in the samples of Gulab Jaman purchased from different shops required 30 mL of 6 M HNO_3 to be eluted completely, when eluted from Dowex50-X8 cation exchange resin. It has also been observed that the concentration of iron in those samples ranges between 10-14 ppm.

The way through which iron gets entry to the sweets is the use of "Karhai" for frying the sweets because it is made-up of iron.

Results for iron are in accordance with RDA values listed in Table 16.

Nickel: Table 10-15 show that the concentration of nickel in the samples of Gulab Jaman of different sweet shops ranges between 9-10 ppm. Table 10-15 also show that nickel in these samples required 45 mL of 6 M HNO_3 for its complete elution through Dowex 50-X8 cation exchange resin. Since sweets are prepared in "ghee" and we are not sure about the quality of that ghee, which becomes a main source of nickel in the sweets because nickel is used as a catalyst for the hydrogenation of oil to convert it into ghee. Nickel is also used greatly in making alloys including stainless steel[12] therefore the utensils used during the preparation of sweets are also responsible for the nickel contents of the sweets. Sugar the main ingredient of the sweets is also responsible for the nickel contents of the sweets[13]. Results for the nickel are also in accordance with RDA values reported in Table 16.

Cobalt: It can be seen from Table 10-15 that cobalt in samples of Gulab Jaman selected from different shops required 15 mL of 6 M HNO_3 for its complete elution through Dowex 50-X8 cation exchange resin. It has been also observed that Gulab Jaman of the different sweet marts contain 7-8 ppm of cobalt. The values for cobalt are found to be higher than the recommended values of RDA listed in Table 16.

Cobalt is also greatly used in alloys[14], hence it may also enter the sweets through utensils. Milk and other ingredients mainly sugar[13] are also responsible for the entrance of cobalt in sweets.

This study was limited to the famous shops but it is necessary that rest of the shops are also monitored for their metal load and the presence of electrolytes in order to assure the quality of the sweets.

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