ISSN 0975-2366
DOI:https://doi.org/10.31838/ijpr/2020.12.03.501

Research Article

Determination of Calcium Level in Milk Powder and Its Relation with Nutrition Rickets by Use FE- SEM With EDS

*RAFID A. DOULAB
EM-unit, Pharmacy College, Basra University
*Corresponding Author
Email: dolab2005@yahoo.com
Received: 08.10.20, Revised: 04.11.20, Accepted: 17.12.20

ABSTRACT
Milk powder is an essential nutrient for humans and mostly for children in developing countries like Iraq. This study highlights calcium sources intake on drying milk powder in the local market. The supreme symptom of calcium deficiency is rickets. Therefore, the 157 prominent sign children were selected to compare the signs and Ca levels in consummated milk powder brands. The results show the acceptable level of Ca in Dielac 1 6.71% and Al-Mudhish 1.10% brands compared with the others by using EDS, and it has matched with ricket children numbers. On the other hand, the FE-SEM images show some particle changes related to Ca inactivity on milk powder affected by its release.

Keywords: calcium, level, powder

INTRODUCTION

Milk products are vital for humans in recent years for being a good source of calcium (1). The content of minerals determines biological properties (2). The need for calcium in milk in mineral deficiency in food and milk will be the richer one depending on its rank (3, 4, 5). Milk powder is commonly used Due to its low humidity, lightweight, simple shipping, and outstanding conservation (6).

The element's level in milk powder brands varies compared with other diets (7). Also, those metals like calcium were reported in expired and unexpired dry milk cans, showing decreased levels compared with cow milk (8).

Dietary calcium deficiency plays an important role to induce rickets in children (9). Similar views on several cases report rickets in infants due to reduced dietary calcium intake (10, 11). The radiology, histology, and biochemical characteristics of rickets changed to a higher degree with calcium supplements (12).

Several reports highlighted the low dietary calcium intake and its relation with pathogenesis rickets (13, 14). On the other hand, nutrient rickets result from inadequate nutritional consumption instead of just vitamin D deficiency. It, therefore, can be balanced with calcium in addition to vitamin D (15).

For many years, the microscope is being used to monitor food adulteration based on powder and particle size morphology (16). Scanning the selected electron microscope (SEM) as energy – dispersive spectrometer (EDS) is the best technique for chemical characterization of samples (17); for example, calcium in milk will be the first organic can be detected due to a specific manner interlink (18, 19).

Calcium hydroxide has received more attention from wide applications of food additives and its effect on specific surface area characterization for power synthesis (20).

The microstructure of a selected powder was checked by an environmental scanning electron microscopy (ESEM) to compare and maybe find the contrast in the form of the examined powder particles in every drying system (21).

MATERIALS AND METHODS

According to a survey for ricket symptom children and consumed brands after checking the expiring date and bought from trusted sources, the milk powder was collected from the local Iraqi market.

For the SEM-EDS study, the specimens were first placed onto the specimen stub using double-sided carbon tape and sprayed with the powder and then, after blowing, all the stubs on the specimen holder to eliminate non-adherent objects. Formulated samples were placed onto FE-SEM from Zeiss Supra 55 V.P. through an airlock panel, relying on the low voltage to surpass the coating technique and prevent charging. Using a high resolution and a sharp picture secondary electron detector (SE2) with

fixed magnification and quite-well calibrated Bruker Quantax EDS XFlash for the components' qualitative and quantitative determination (3).

A group of 157 minors with leg deformities and radiographic ricket reports were enrolled for milk powder feeding, as shown in table (1). The calcium intake, according to daily requirements, depends on age (22).

RESULTS AND DISCUSSION

Table 1: show milk powder brands and survey for children range age

No	Milk powder brand	No. of children	No. of Rickets children	Age (Year)
1	Al Mudhish	20	1	1-3
2	Anchor	18	9	1-4
3	Nido	10	6	1-3
4	Dielac 1	25	0	1-2
5	Dielac 2	20	2	2-6
6	Guigoz	15	2	1-2
7	Liptomil	22	6	1
8	PediaSure	27	3	1-4

Scanning electron microscopes were applied on eight different milk powder particles to determine the EDS's calcium levels, and imaging of it depended on low voltage FE-SEM (17).

Table (2) represents the qualitative and quantitative concentration for calcium and phosphorus with the ZAF correction method, and Figure (2) illustrates the y-axis for x-ray count. At the same time, the x-axis, on the other side, shows energy in Kev. The detection lines, for energy matchup between sample levels and spectrum, peaks (23).

The estimated calcium in Dielac 1 was 6.71% compared with Al mudfish, 1.10 % higher than other brands, which did not exceed 0.71%. Simultaneously, the phosphorus percentage variable was generally higher than the calcium percentage equal to in Guigoz. Nevertheless, in Nido and Dielac 2, the phosphorus percentage were 0.09 and 0.05, respectively; zero calcium and phosphorus were observed for Liptomil.

On the other hand, the particle sizes and microstructure varied, too, depending on concentration and temperature (24).

Table 2: represents the qualitative and quantitative concentration for calcium and phosphorus with the ZAF correction method.

No	Milk powder brand	Calcium concentration %	Phosphorus concentration %	Particle size in μm
1	Al Mudhish	1.10	1.70	8-16
2	Anchor	0.09	0.13	35-65
3	Nido	0.68	0.09	40-60
4	Dielac 1	6.71	12.03	20-40
5	Dielac 2	0.07	0.05	5-40
6	Guigoz	0.01	0.01	10-50
7	Liptomil	0	0	5-30
8	PediaSure	0.71	0.81	10-40

Table (2) shows the milk powder brands and concentrations for Ca and P and particle sizes.

Figure (2) shows the shapes and particle size with the vast difference in microstructure like agglomerates of fine particles like in Nido, Guigoz, and Dielac 2 (25). Some other factors related to milk powder production procedure like calcium hydroxide concentration increase also with temperature and rotation show slightly increase the particle size as is evident in Liptomil and Pediasure and vice versa for the other brands with little spherical appearance some morphological changes, rectangular and crystal growth, also observe (20).

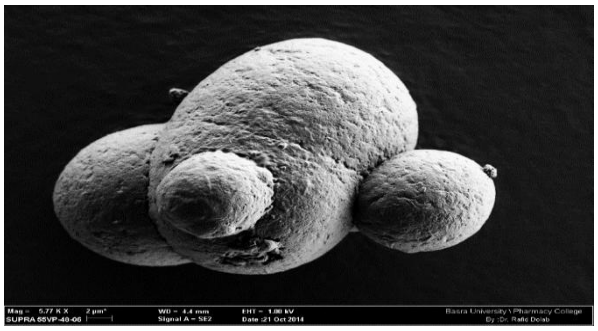
Sometimes the temperature during processing results in burnt particles, mainly by producing relics in examination (26). The particle size ranged from 2-52 μm or cracked; however, smooth surfaces were observed (27, 28).

Generally, the surface morphology could vary from smooth to moderate raisin-like depending on preparation composition (29).

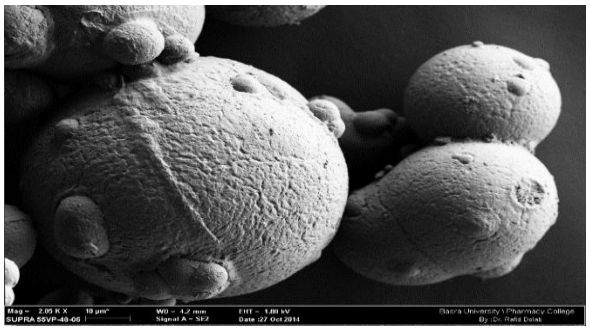
There is no difference between those who obtained calcium alone or others who received vitamin D with calcium (15). Children treated with

calcium that was given orally showed less rickets than those who did not finish their food completed with calcium (30). Otherwise, Vitamin D improves the healing of ricket in the minors with calcium deficiency, one it's not clear (31).

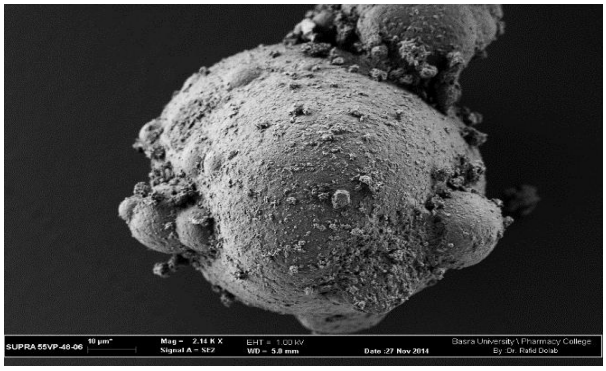
With few or no dairy products, the primary cause of rickets in minors with restraint for infants will be calcium deficiency intake (32).



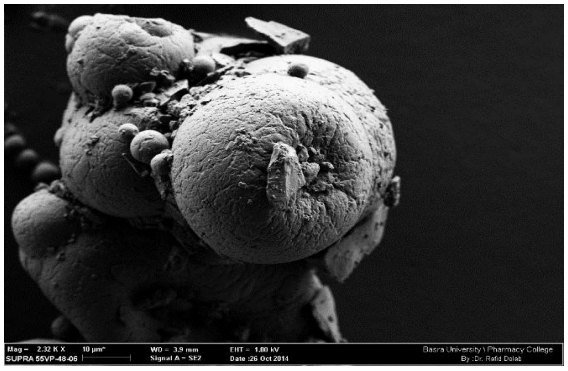
Al Mudhish



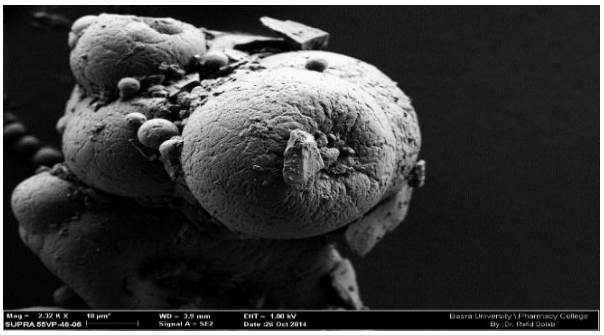
Anchor



Nido

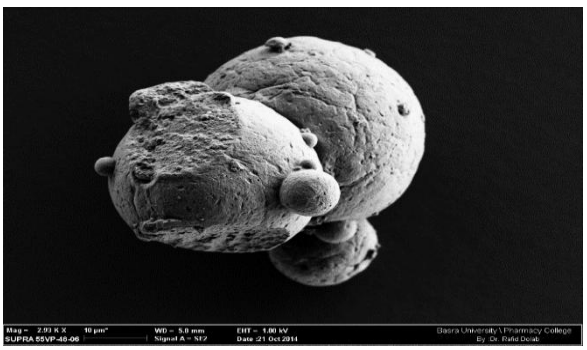


Dielac 1

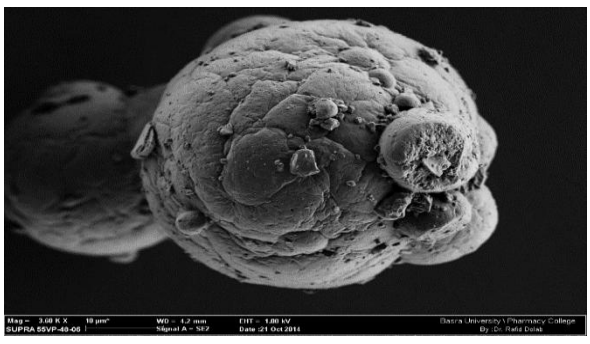


Dielac 2

Guigoz



Liptomil



PediaSure

Fig.2: FE-SEM image for different brands of milk powder scan at 10 µm

Spectrum: Al Mudhish

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Oxygen	K-series	7.33	72.32	82.61	2.94
Sodium	K-series	1.07	10.51	8.35	0.30
Sulfur	K-series	0.57	5.64	3.21	0.14
Potassium	K-series	0.42	4.13	1.93	0.12
Calcium	K-series	0.24	2.41	1.10	0.11
Phosphorus	K-series	0.31	3.03	1.79	0.12
Chlorine	K-series	0.20	1.97	1.01	0.10
Total:		10.14	100.00	100.00	

Spectrum: Anchor

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Carbon	K-series	56.05	56.05	63.49	18.85
Oxygen	K-series	41.49	41.49	35.28	14.68
Fluorine	K-series	0.85	0.85	0.61	0.65
Potassium	K-series	0.53	0.53	0.18	0.14
Phosphorus	K-series	0.29	0.29	0.13	0.12
Chlorine	K-series	0.29	0.29	0.11	0.11
Calcium	K-series	0.26	0.26	0.09	0.11
Magnesium	K-series	0.18	0.18	0.10	0.11
Zinc	K-series	0.07	0.07	0.01	0.11
Total:		100.00	100.00	100.00	

Spectrum: Nido

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Carbon	K-series	61.85	61.85	69.55	20.97
Oxygen	K-series	33.84	33.84	28.57	12.69
Calcium	K-series	2.03	2.03	0.68	0.26
Fluorine	K-series	0.56	0.56	0.40	0.55
Sodium	K-series	0.35	0.35	0.21	0.16
Magnesium	K-series	0.37	0.37	0.21	0.15
Phosphorus	K-series	0.20	0.20	0.09	0.10
Sulfur	K-series	0.19	0.19	0.08	0.10
Chlorine	K-series	0.18	0.18	0.07	0.10
Potassium	K-series	0.41	0.41	0.14	0.12
Total:		100.00	100.00	100.00	

Spectrum: Dielac 1

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Fluorine	K-series	1.36	15.13	22.57	1.04
Sodium	K-series	1.40	15.63	19.28	0.38
Magnesium	K-series	0.80	8.89	10.37	0.23
Phosphorus	K-series	1.18	13.14	12.03	0.24
Sulfur	K-series	0.35	3.94	3.48	0.14
Chlorine	K-series	1.29	14.32	11.45	0.24
Potassium	K-series	1.75	19.45	14.10	0.27
Calcium	K-series	0.85	9.49	6.71	0.20
Total:		8.97	100.00	100.00	

Spectrum: Dielac 2

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Carbon	K-series	57.73	57.73	64.81	19.52
Oxygen	K-series	41.19	41.19	34.71	14.80
Sodium	K-series	0.25	0.25	0.15	0.13
Aluminium	K-series	0.19	0.19	0.09	0.11
Silicon	K-series	0.01	0.01	0.01	0.08
Phosphorus	K-series	0.11	0.11	0.05	0.10
Sulfur	K-series	0.02	0.02	0.01	0.08
Chlorine	K-series	0.10	0.10	0.04	0.09
Potassium	K-series	0.20	0.20	0.07	0.11
Calcium	K-series	0.20	0.20	0.07	0.11
Total:		100.00	100.00	100.00	

Spectrum: Guigoz

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Carbon	K-series	74.88	74.88	80.05	24.92
Oxygen	K-series	24.44	24.44	19.62	9.66
Sodium	K-series	0.37	0.37	0.21	0.16
Sulfur	K-series	0.23	0.23	0.09	0.11
Potassium	K-series	0.01	0.01	0.00	0.08
Calcium	K-series	0.05	0.05	0.01	0.09
Phosphorus	K-series	0.02	0.02	0.01	0.08
Total:		100.00	100.00	100.00	

Spectrum: Liptomil

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Oxygen	K-series	59.73	59.73	57.32	21.35
Nitrogen	K-series	36.55	36.55	40.07	12.88
Sodium	K-series	1.80	1.80	1.20	0.45
Fluorine	K-series	1.50	1.50	1.21	1.17
Sulfur	K-series	0.42	0.42	0.20	0.13
Total:		100.00	100.00	100.00	

Spectrum: PediaSure

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Oxygen	K-series	18.31	87.25	92.60	6.50
Sodium	K-series	1.05	5.01	3.70	0.30
Calcium	K-series	0.35	1.68	0.71	0.12
Potassium	K-series	0.41	1.94	0.84	0.12
Phosphorus	K-series	0.31	1.48	0.81	0.12
Sulfur	K-series	0.28	1.34	0.71	0.11
Chlorine	K-series	0.27	1.30	0.62	0.11
Total:		20.99	100.00	100.00	

Table (3): EDX acquisition tables of the element distribution of surface for different brands of milk powder scan at 10 µm

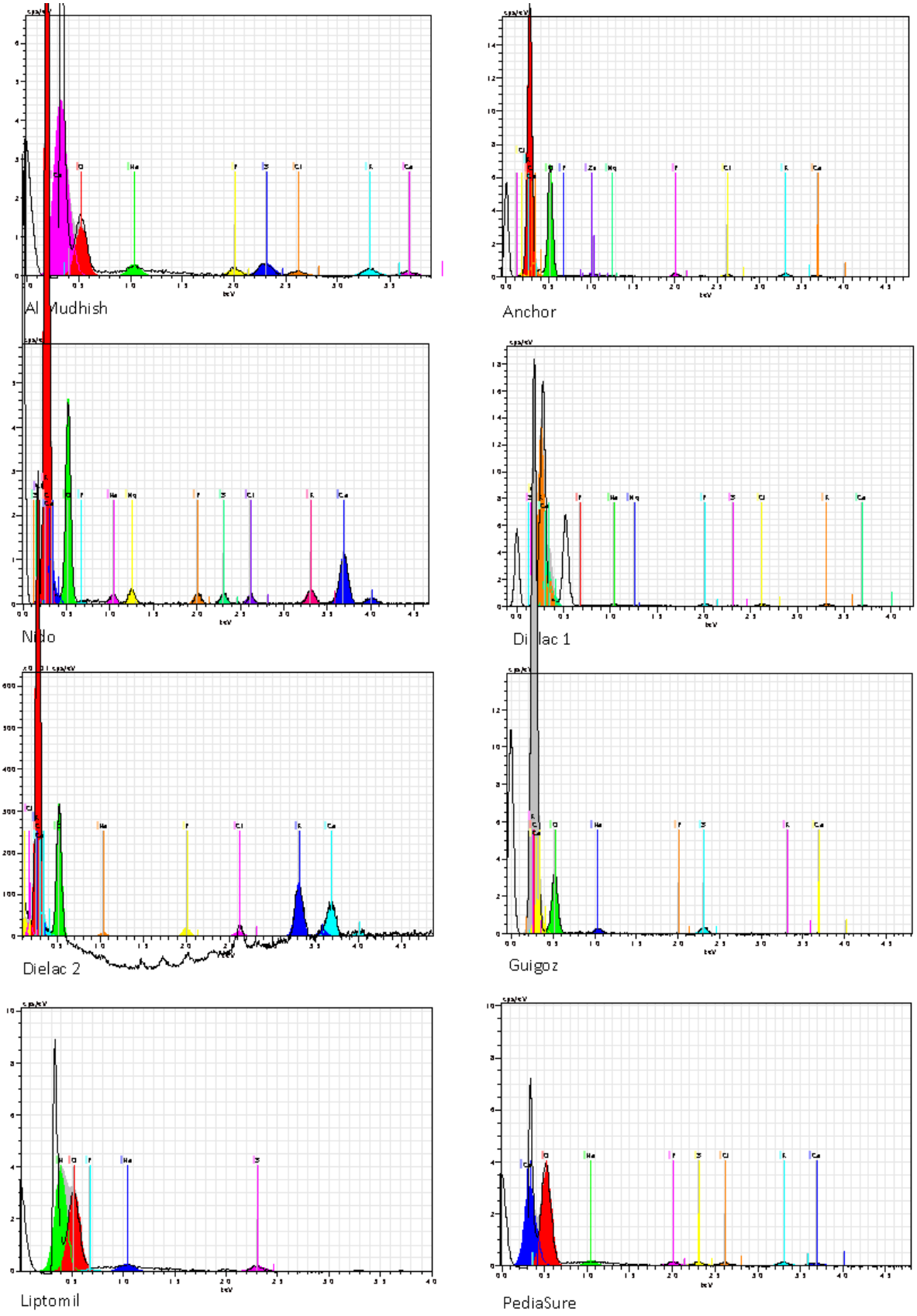


Figure (2): EDX spectra of the element distribution of surface for different brands of milk powder scan at 10 µm

REFERENCES

1. Semaghuil Birghila, Simona Dobrinas, Gabriela Stanciu. , Determination Of Major And Minor Elements In Milk Through ICP-AES. Environment Engineering And Management Journal.VOL 7; No.6, 2008, Pp805-808.
2. Dobrazanski, R. Kolacz, H. Gorecka, Et Al. , The Content Of Microelements And Trace Elements In Raw Milk From Cows In The Silesian Region. Polish Journal Of Environmental Studies, VOL. 14,No 5, 2005, 685-689.
3. WNUK W., ODOJ J., BIS-WENCEL H., SABA L., NOWAKOWICZ-DEBEK B., Et Al. , Milk And Hair Coat As Indicators Of Macroelements Content In Cows At Different Stages Of Lactation. Annales Univ. M. Curie-Sk3odowska Lublin-Polonia, Vol. XXI, N2, 96, Sectio EE, 2003, 333.
4. FUNDORA, O., GONZALEZ, M.E., LEZCANO, O., Et Al. , A Comparative Study Of Milk Composition And Stability Of Murrah River Buffaloes And Holstein Cows Grazing Star Grass. Cub. J. Agric. Sci., 35: 2001, 219-222.
5. AHMAD, S., GAUCHER, I., ROUSSEAU, F., Et Al. , Effects Of Acidification On Physico-Chemical Characteristics Of Buffalo Milk: A Comparison With Cow's Milk. Fd. Chem., 106: 2008, 11-17.
6. SHIRATSUCHI, H., SHIMODA, M., IMAYOSHI, K., Et Al. , Volatile Flavor Compounds In Spray-Dried Skim Milk Powder. J. Agric.Fd. Chem., 42:1994, 984-988.
7. Ikem A., Nwankwoala A., Odueyungbo S., Et Al., Levels Of 26 Elements In Infant Formula From USA, UK, And Nigeria By Microwave Digestion And ICP–OES, Food Chemistry, 77, 2002, 439–447.
8. Jaffar, M., Shah, M.H., Shaheen, N., Et Al. , Pre- And Post-Expiry Metal Levels In Canned Dry Milk. Nut. Food Sci., 34(2): 2004, 65-71.
9. 9. John M. Pettifor , Nutritional Rickets: Deficiency Of Vitamin D, Calcium, Or Both. Am J Clin Nutr., 2004, 1725S-9S. .9.
10. Kooh SW, Fraser D, Reilly BJ, Et Al. , Rickets Due To Calcium Deficiency. N Engl J Med., 297:1997, 1264–6
11. Legius E, Proesmans W, Eggermont E, Et Al. , Rickets Due To Dietary Calcium Deficiency. Eur J Pediatr.,148: 1989, 784–785.
12. Marie PJ, Pettifor JM, Ross FP. , Histological Osteomalacia Due To Dietary Calcium Deficiency In Children. N Engl J Med., 307:1982, 584-8.
13. Oginni LM, Worsfold M, Oyelami OA, Et Al. , Etiology Of Rickets In Nigerian Children. J Pediatr., 128: 1969, 692– 4.
14. Okonofua F, Gill DS, Alabi ZO, Et Al. , Rickets In Nigerian Children: A Consequence Of Calcium Malnutrition. Metabolism, 40: 1991, 209-13.
15. Thacher TD, Fischer PR, Pettifor JM, Et Al., A Composition Of Calcium, Vitamin D, Or Both For Nutritional Rickets In Nigerian Children. N Engl J Med., 341;1999, 563-8.
16. Brennan, J.G. And Grandison, A.S. , Food Processing Handbook. 2nd Ed. Vol.2, Wiley-VCH Verlag And Co. Kgaa, Boschstr, Weinheim, Germany, 2012, P 494.
17. Rubina Parween, Darakhshan Ara, Muhammad Shahid Et Al. , FUUAST J. BIOL., 6(2): 2016, 161-164.
18. Fennema, O.R. , Food Chemistry, 3rd Ed. Owen R. Fennema, Chapter No. 14.1969
19. Whitney, E. And Rolfes, S.R. , Understanding Nutrition. 10th Ed., Thompson Wadsworth Australia, 2005, 458.
20. Moon-Kwan Choi, Jin-Sang Cho, Sung-Min Joo, Et Al., Effect Of Several Factors On The Synthesis Of Calcium Hydroxide Prepared By Spray Drying Method Using Limestone. Material Science Forum. Vol.544-545, 2007, Pp 733-736.
21. Ronald I. Thompson. , Nutrient Profile, Functional Properties, Andmicrostructure Of Dried Waste Milk Products For Use As Apotential ANIMAL FEED. Faculty Of The Louisiana State University And Agricultural And Mechanical College. 2002, P43.
22. Institute Of Medicine. , Dietary Reference Intakes For Calcium And Vitamin D. Washington, DC : The National Academic Press, 2011.
23. Trincavelli, J., Silvina L. And Rita B. , Standardless Quantification Methods In Electron Probe Microanalysis. Spectrochimica Acta Part B: Atomic Spectroscopy, 101, 2014. 76-85.
24. Mian Anjum Murtaza, Nuzhat Huma, Irfan Mustafa Et Al. , Composition And Microstructure Of Buffalo Skim Milk Powder As Influenced By Concentration And Drying Tempretures. Pakistan J. Zool., Vol. 47 (4), 2015, Pp 1157-1161.
25. Y. Wen , L Xiang, Y. Jin. , Materials Letters Vol. 57 , 2003, P.2565-2571.

26. PATEL, R.S. AND MISTRY, V.V., , Physicochemical And Structural Properties Of Ultrafiltered Buffalo Milk And Milk Powder. J. Dairy Sci., 80: 1997, 812-817.
27. CARIC, M. AND KALAB, M. , Effects Of Drying Techniques On Milk Powder Quality And Microstructure: A Review. Fd. Microstr., 6: 1987, 171.
28. MISTRY, V.V., HASSAN, H.N. AND ROBISON, D.J. , Effect Of Lactose And Protein On The Microstructure Of Dried Milk. Fd. Struct., 11: 1992, 73.
29. Vehring R. , Pharmaceutical Particle Engineering Via Spray Drying. Pharma Res., 25: 2008, 999-1022.
30. Tom D Thacher, Philip R Fischer, John M Pettifor. , Vitamin D Treatment In Calcium-Deficiency Rickets: A Randomized Controlled Trail. Arch Dis Child., 2013, 99-807-811.
31. Thacher TD, Obadofin MO, O Brien KO, Et Al. , The Effect Of Vitamin D2 And Vitamin D3 On Intestinal Calcium Absorption In Nigerian Children With Rickets. J Clin Endocrinol Metab., 94: 2009, 3314-21.
32. Delucia MC, Mitnick ME, And Carpenter TO. (2003). Nutritional Rickets With Normal Circulating 25-Hydroxyvitamin D: A Call For Reexamining The Role Of Dietary Calcium Intake In North American Infants. J Clin Endocrinol Metab , 88: 2003, 3539–45.