



Sudan University of Science and Technology
College of Graduate Studies



**Detection of Some Elements in Kapo Powder milk
and Nedo powder milk by Using X-Ray
Fluorescence Device**

الكشف عن بعض العناصر في لبن البودرة كابو ولبن البودرة نيدو
بإستخدام جهاز الأشعة السينية المتوهجة

A dissertation Submitted as Partial Fulfillment of the Requirements for
the Degree of Master of Science in Physics.

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الآية

بسم الله الرحمن الرحيم

قال الله تعالى:

﴿ قُلْ هَلْ يَسْتَوِي الَّذِينَ يَعْلَمُونَ وَالَّذِينَ لَا يَعْلَمُونَ ﴾ صدق الله العظيم

[الزمر:9]

Dedication

To the spirit of my pure father

To my dear mother

To my brothers and sisters

To my husband

To all my teachers

To all my friends

I dedicate you this research

Acknowledgements

Firstly thanks to Allah for reconciling me

Thanks to my beautiful family and my teachers and especially the supervisor of this research; Dr. Nafeesa Bar Eldain.

Abstract

This study deals with the applications of spectroscopy, which is the detection of some elements of the Nido powder milk and Kapo powder milk and the concentration of these elements by X-Ray Fluorescence device and the comparison between them and to see the pH of the two samples, in milk Nido element chromium and its concentration 0.03%, manganese element, concentration 0.00%, Iron element and its concentration was 0.11%, nickel element, its concentration <0.001%, copper element, its concentration 0.00%, zinc element, its concentration 0.02 % and lead element and its concentration was 0.00%, and the PH of Nedo powder milk was 6.25.

In Kapo powder milk sample this elements were found also but in different concentrations; which found chromium element, its concentration was 0.02%, manganese element, concentration 0.02%, Iron element and its concentration was 0.02%, nickel element, its concentration <0.00%, copper element, its concentration 0.00%, zinc element, its concentration 0.00% and lead element and its concentration was 0.01%, and the PH of Kapo powder milk was 6.39.

المستخلص

يتناول هذا البحث إحدى تطبيقات علم الأطياف وهي الكشف عن بعض العناصر المكونة للبن البودرة نيدو ولبن البودرة كابو وتراكيز هذه العناصر بتقنية فلورة الأشعة السينية والمقارنة بينهم ومعرفة الرقم الهيدروجيني للعينتين ،حيث وجد في لبن نيدو عنصر الكروم وتركيزه 0.03% وعنصر المنجنيز وتركيزه 0.00% وعنصر الحديد وتركيزه 0.11% وعنصر النيكل وتركيزه > 0.01% وعنصر الزنك وتركيزه 0.02% وعنصر الرصاص وتركيزه 0.00% ، ووجد أن الرقم الهيدروجيني لهذه العينة هو 6.25.

أما في لبن كابو وجدت أيضاً هذه العناصر ولكن بتراكيز مختلفة حيث وجد عنصر الكروم وتركيزه 0.02% وعنصر المنجنيز وتركيزه 0.02% وعنصر الحديد وتركيزه 0.02% وعنصر النيكل وتركيزه 0.00% وعنصر الزنك وتركيزه 0.00% ووجد أن هذه العينة بها نسبة من عنصر الرصاص وتركيزه 0.01% ، ووجد أن الرقم الهيدروجيني لعينة لبن البودرة كابو هو 6.37 ولذلك فإن لبن نيدو هو الأفضل.

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Chapter one

Introduction & literature review

Chapter One

Introduction

1.1 Introduction

The analysis of milk is important because milk is an indicator of environmental contamination, a significant pathway for toxic metal intake by humans and a source of essential nutrients. Essential elements required by the human body include the four basic elements H, C, N and O; the quantity elements Na, Mg, K, Ca, P, S and Cl; and the essential trace elements Mn, Fe, Co, Ni, Cu, Zn, Mo, Se and I. Other trace elements include B, Ba, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Sb, Se, Sn, Sr, V, W and Zn. These elements were detected previously in liquid milk and powdered milk using neutron activation analysis. The importance of trace elements in nutrition is widely recognized as essential for growth and development of human beings, especially during infancy. On the other hand, several elemental deficiency syndromes have been reported over the past decades. Children in particular are susceptible to the effects of a trace element deficiency [1]. The determination of trace element levels in food-stuffs, especially milk [2], could play an important role in understanding a number of deficiency-related diseases.

Diet quality is determined by the proportion of individual components, such as protein, fat, vitamins and hydrocarbons, and by the concentrations of major and essential trace elements. A certain number of elements are essential for life, while others are heavy metals or toxic elements, which demonstrate their toxic influence on human health and growth even at small concentrations. Some diseases are clearly correlated with an imbalance of trace elements in the human body. The excess or deficiency of some trace elements has a significant influence on human health. For example, a deficiency in Fe causes anaemia and a deficiency in Zn is

associated with depressed growth, sexual immaturity and skin lesions. The trace mineral selenium (Se) is an essential nutrient of fundamental importance to human health but is required only in small amounts. Selenium deficiency can lead to heart disease, hypothyroidism and a weakened immune system. In contrast, an excess of Mn may inhibit Fe absorption and an excess of Zn may cause a Cu deficiency. A large fraction of natural radiation exposure is due to ingestion of food containing natural radionuclides such as ^{40}K , ^{226}Ra , ^{210}Po and ^{210}Pb [3–5]. Among these, ^{40}K , which is an important radionuclide from a physical health point of view, is largely taken up by humans due to its widespread distribution in the environment and living organisms [1].

1.2 Literature review

Jolanta Borkowska-Burnecka*, Ewa Szmigiel and Wiesław Zyrnicki in 1996, their study was to Determination of Major and Trace Elements in Powdered Milk by Inductively Coupled Plasma

Atomic Emission Spectrometry they used inductively coupled plasma atomic emission spectrometry method to determine major (Ca, Mg, Na, K and P) and trace (Ba, B, Cu, Cd, Fe, Mn, Pb, Sr and Zn) elements in milk powder. Eight milk powder products have been studied. Samples have been digested in acids using a conventional hot plate technique and microwave heating high pressure procedure. Some matrix effects have been investigated and discussed.

UR-REHMAN, ISHRAT REHANA, WASIM YAWAR in 2012, their study was to determination of inorganic elements in milk powder using wavelength dispersive X-ray fluorescence spectrometer. They found the concentration of calcium, magnesium, phosphorus, potassium and trace elements, bromine, copper, iron, rubidium and zinc in different brands of

milk powder and infant formulas have been determined using a wavelength dispersive X-ray fluorescence spectrometer.

Lawal, N.S., Tajuddeen, N. and Garba, B.B. in (2015), their study was Assessment of some mineral elements in different brands of powdered milk sold in Samaru Zaria, Nigeria Powdered milk products contain important nutrient elements and are mostly consumed in developing countries. Good quality control is very essential in the production and processing of powdered milk products, also transportation and storage conditions might have effects on the composition of these products. The deficiency or excess of any of the mineral elements in powdered milk may create significant health problems for the consumer. In this study, the concentration levels of some mineral elements in different brands of powdered milk sold in Samaru market Zaria Nigeria were determined using Atomic Absorption Spectroscopy (A.A.S). The average concentrations of Ca, Mg, Fe, Cu, Zn and Mn in the five brands of powdered milk studied were 12183.16, 986.70, 108.88, 5.92, 42.97, and 8.44 mg/kg respectively. These concentrations of Ca, Mg, Fe, Cu, Zn and Mn contribute about 38, 9, 20, 13, 9 and 10% respectively to the recommended dietary allowance (RDA) per serving (25 g of powdered milk).

1.3 Research problem

Most of milk powder contains many contaminated elements that may differ from sample to another according to the company that made it from quality?

1.4 objective of research

The main objective of this research was to:

- 1/ Identification of elements in need powder milk
- 2/ Identification of elements in kappa powder milk
- 3/ Comparative between two results in two samples

1.5 Thesis Layout

This thesis is consist of four chapters, chapter one introduction and literature review, chapter two consists the basic concepts of spectroscopy, and its applications, powder milk, X-Ray Fluorescence , chapter three consist methodology (materials, device and method), chapter four consist of results and discussion and conclusion and recommendations, finally a list of references.

Chapter Two

Basic Concepts

Chapter Two

Basic Concepts

2.1 Spectroscopy

Spectroscopy is study of the interaction between matter and electromagnetic radiation. Historically, spectroscopy originated through the study of visible light dispersed according to its wavelength, by a prism. Later the concept was expanded greatly to include any interaction with radioactive energy as a function of its wavelength or frequency. Spectroscopic data is often represented by an emission spectrum, a plot of the response of interest as a function of wavelength or frequency.

Spectroscopy and spectrograph are terms used to refer to the measurement of radiation intensity as a function of wavelength and are often used to describe experimental spectroscopic methods. Spectral measurement devices are referred to as spectrometers, spectrophotometers, spectrographs or spectral analyzers.

Daily observations of color can be related to spectroscopy. Neon lighting is a direct application of atomic spectroscopy. Neon and other noble gases have characteristic emission frequencies (colors). Neon lamps use collision of electrons with the gas to excite these emissions. Inks, dyes and paints include chemical compounds selected for their spectral characteristics in order to generate specific colors and hues. A commonly encountered molecular spectrum is that of nitrogen dioxide. Gaseous nitrogen dioxide has a characteristic red absorption feature, and this gives air polluted with nitrogen dioxide a reddish-brown color. Rayleigh scattering is a spectroscopic scattering phenomenon that accounts for the color of the sky.

Spectroscopic studies were central to the development of quantum mechanics and included Max Planck's explanation of blackbody radiation, Albert Einstein's explanation of the photoelectric effect and Neil's Bohr's explanation of atomic structure and spectra. Spectroscopy is used in physical and analytical chemistry because atoms and molecules have unique spectra. As a result, these spectra can be used to detect, identify and quantify information about the atoms and molecules. Spectroscopy is also used in astronomy and remote sensing on Earth. Most research telescopes have spectrographs. The measured spectra are used to determine the chemical composition and physical properties of astronomical objects (such as their temperature and velocity).

One of the central concepts in spectroscopy is a resonance and its corresponding resonant frequency. Resonances were first characterized in mechanical systems such as pendulums. Mechanical systems that vibrate or oscillate will experience large amplitude oscillations when they are driven at their resonant frequency. A plot of amplitude vs. excitation frequency will have a peak centered at the resonance frequency. This plot is one type of spectrum, with the peak often referred to as a spectral line, and most spectral lines have a similar appearance.

In quantum mechanical systems, the analogous resonance is a coupling of two quantum mechanical stationary states of one system, such as an atom, via an oscillatory source of energy such as a photon. The coupling of the two states is strongest when the energy of the source matches the energy difference between the two states. The energy E of a photon is related to its frequency (ν) by $E=h\nu$ where h is Planck's constant, and so a spectrum of the system response vs. photon frequency will peak at the resonant frequency or energy. Particles such as electrons and neutrons have a comparable relationship, the de Broglie relations, between their kinetic

energy and their wavelength and frequency and therefore can also excite resonant interactions.

Spectra of atoms and molecules often consist of a series of spectral lines, each one representing a resonance between two different quantum states. The explanation of these series, and the spectral patterns associated with them, were one of the experimental enigmas that drove the development and acceptance of quantum mechanics. The hydrogen spectral series in particular was first successfully explained by the Rutherford-Bohr quantum model of the hydrogen atom. In some cases spectral lines are well separated and distinguishable, but spectral lines can also overlap and appear to be a single transition if the density of energy states is high enough. Named series of lines include the principal, sharp, diffuse and fundamental series.

2.2 Classification

Spectroscopy is a sufficiently broad field that many sub-disciplines exist, each with numerous implementations of specific spectroscopic techniques. The various implementations and techniques can be classified in several ways.

2.2.1 Type of radioactive energy:

Types of spectroscopy are distinguished by the type of radiative energy involved in the interaction. In many applications, the spectrum is determined by measuring changes in the intensity or frequency of this energy. The types of radiative energy studied include:

- Electromagnetic radiation was the first source of energy used for spectroscopic studies. Techniques that employ electromagnetic

radiation are typically classified by the wavelength region of the spectrum and include microwave, terahertz, infrared, near infrared, visible and ultraviolet, x-ray and gamma spectroscopy.

- Particles, due to their de Broglie wavelength, can also be a source of radiative energy and both electrons and neutrons are commonly used. For a particle, its kinetic energy determines its wavelength.
- Acoustic spectroscopy involves radiated pressure waves.
- Mechanical methods can be employed to impart radiating energy, similar to acoustic waves, to solid materials.

2.2.2 Nature of the interaction

Types of spectroscopy can also be distinguished by the nature of the interaction between the energy and the material. These interactions include: [1]

- Absorption occurs when energy from the radiative source is absorbed by the material. Absorption is often determined by measuring the fraction of energy transmitted through the material; absorption will decrease the transmitted portion.
- Emission indicates that radiative energy is released by the material. A material's blackbody spectrum is a spontaneous emission spectrum determined by its temperature; this feature can be measured in the infrared by instruments such as the Atmospheric Emitted Radiance Interferometer (AERI).[4] Emission can also be induced by other sources of energy such as flames or sparks or electromagnetic radiation in the case of fluorescence.

- Elastic scattering and reflection spectroscopy determine how incident radiation is reflected or scattered by a material.
- Crystallography employs the scattering of high energy radiation, such as x-rays and electrons, to examine the arrangement of atoms in proteins and solid crystals.
- Impedance spectroscopy studies the ability of a medium to impede or slow the transmittance of energy. For optical applications, this is characterized by the index of refraction.
- Inelastic scattering phenomena involve an exchange of energy between the radiation and the matter that shifts the wavelength of the scattered radiation. These include Raman and Compton scattering.
- Coherent or resonance spectroscopy are techniques where the radiative energy couples two quantum states of the material in a coherent interaction that is sustained by the radiating field. The coherence can be disrupted by other interactions, such as particle collisions and energy transfer, and so often require high intensity radiation to be sustained. Nuclear magnetic resonance (NMR) spectroscopy is a widely used resonance method and ultrafast laser methods are also now possible in the infrared and visible spectral regions.

2.2.3 Types of material

Spectroscopic studies are designed so that the radiant energy interacts with specific types of matter.

1- Atomic Spectroscopy

Atomic spectroscopy was the first application of spectroscopy developed. Atomic absorption spectroscopy (AAS) and atomic emission spectroscopy (AES) involve visible and ultraviolet light. These absorptions and emissions, often referred to as atomic spectral lines, are due to electronic transitions of outer shell electrons as they rise and fall from one electron orbit to another. Atoms also have distinct x-ray spectra that are attributable to the excitation of inner shell electrons to excited states.

Atoms of different elements have distinct spectra and therefore atomic spectroscopy allows for the identification and quantitation of a sample's elemental composition. Robert Bunsen and Gustav Kirchhoff discovered new elements by observing their emission spectra. Atomic absorption lines are observed in the solar spectrum and referred to as Fraunhofer lines after their discoverer. A comprehensive explanation of the hydrogen spectrum was an early success of quantum mechanics and explained the Lamb shift observed in the hydrogen spectrum, which further led to the development of quantum electrodynamics.

Modern implementations of atomic spectroscopy for studying visible and ultraviolet transitions include flame emission spectroscopy, inductively coupled plasma atomic emission spectroscopy, glow discharge spectroscopy, microwave induced plasma spectroscopy, and spark or arc emission spectroscopy. Techniques for studying x-ray spectra include X-ray spectroscopy and X-ray fluorescence (XRF).

2- Molecules:

The combination of atoms into molecules leads to the creation of unique types of energetic states and therefore unique spectra of the transitions betn

these states. Molecular spectra can be obtained due to electron spin states (electron paramagnetic resonance), molecular rotations, molecular vibration and electronic states.

Rotations are collective motions of the atomic nuclei and typically lead to spectra in the microwave and millimeter-wave spectral regions; rotational spectroscopy and microwave spectroscopy are synonymous. Vibrations are relative motions of the atomic nuclei and are studied by both infrared and Raman spectroscopy. Electronic excitations are studied using visible and ultraviolet spectroscopy as well as fluorescence.

Studies in molecular spectroscopy led to the development of the first maser and contributed to the subsequent development of the laser.

3- Crystals and extended materials:

The combination of atoms or molecules into crystals or other extended forms leads to the creation of additional energetic states. These states are numerous and therefore have a high density of states. This high density often makes the spectra weaker and less distinct, i.e., broader. For instance, blackbody radiation is due to the thermal motions of atoms and molecules within a material. Acoustic and mechanical responses are due to collective motions as well. Pure crystals, though, can have distinct spectral transitions, and the crystal arrangement also has an effect on the observed molecular spectra. The regular lattice structure of crystals also scatters x-rays, electrons or neutrons allowing for crystallographic studies.

4- Nuclei

Nuclei also have distinct energy states that are widely separated and lead to gamma ray spectra. Distinct nuclear spin states can have their

energy separated by a magnetic field, and this allows for NMR spectroscopy.

2.2.4 Other types

Other types of spectroscopy are distinguished by specific applications or implementations:

- Acoustic resonance spectroscopy is based on sound waves primarily in the audible and ultrasonic regions
- Auger spectroscopy is a method used to study surfaces of materials on a micro-scale. It is often used in connection with electron microscopy.
- Cavity ring down spectroscopy
- Circular Dichroism spectroscopy
- Coherent anti-Stokes Raman spectroscopy (CARS) is a recent technique that has high sensitivity and powerful applications for *in vivo* spectroscopy and imaging.[5]
- Cold vapour atomic fluorescence spectroscopy
- Correlation spectroscopy encompasses several types of two-dimensional NMR spectroscopy.
- Deep-level transient spectroscopy measures concentration and analyzes parameters of electrically active defects in semiconducting materials
- Dual polarisation interferometry measures the real and imaginary components of the complex refractive index
- Electron phenomenological spectroscopy measures physicochemical properties and characteristics of electronic structure of multicomponent and complex molecular systems.
- EPR spectroscopy

- Force spectroscopy
- Fourier transform spectroscopy is an efficient method for processing spectra data obtained using interferometers. Fourier transform infrared spectroscopy (FTIR) is a common implementation of infrared spectroscopy. NMR also employs Fourier transforms.
- Hadron spectroscopy studies the energy/mass spectrum of hadrons according to spin, parity, and other particle properties. Baryon spectroscopy and meson spectroscopy are both types of hadron spectroscopy.
- Hyperspectral imaging is a method to create a complete picture of the environment or various objects, each pixel containing a full visible, VNIR, NIR, or infrared spectrum.
- Inelastic electron tunneling spectroscopy (IETS) uses the changes in current due to inelastic electron-vibration interaction at specific energies that can also measure optically forbidden transitions.
- Inelastic neutron scattering is similar to Raman spectroscopy, but uses neutrons instead of photons.
- Laser-Induced Breakdown Spectroscopy (LIBS), also called Laser-induced plasma spectrometry (LIPS)
- Laser spectroscopy uses tunable lasers [6] and other types of coherent emission sources, such as optical parametric oscillators, [7] for selective excitation of atomic or molecular species.
- Mass spectroscopy is an historical term used to refer to mass spectrometry. Current recommendations [8] are to use the latter term. Use of the term mass spectroscopy originated in the use of phosphor screens to detect ions.
- Mössbauer spectroscopy probes the properties of specific isotopic nuclei in different atomic environments by

analyzing the resonant absorption of gamma-rays. See also Mössbauer effect.

- Neutron spin echo spectroscopy measures internal dynamics in proteins and other soft matter systems
- Photoacoustic spectroscopy measures the sound waves produced upon the absorption of radiation.
- Photoemission spectroscopy
- Photothermal spectroscopy measures heat evolved upon absorption of radiation.
- Pump-probe spectroscopy can use ultrafast laser pulses to measure reaction intermediates in the femtosecond timescale.
- Raman optical activity spectroscopy exploits Raman scattering and optical activity effects to reveal detailed information on chiral centers in molecules.
- Raman spectroscopy
- Saturated spectroscopy
- Scanning tunneling spectroscopy
- Spectrophotometry
- Spin noise spectroscopy traces spontaneous fluctuations of electronic and nuclear spins.[9]
- Time-resolved spectroscopy measures the decay rate (s) of excited states using various spectroscopic methods.
- Time-Stretch Spectroscopy[10][11]
- Thermal infrared spectroscopy measures thermal radiation emitted from materials and surfaces and is used to determine the type of bonds present in a sample as well as their lattice environment. The techniques are widely used by organic chemists, mineralogists, and planetary scientists.

- Transient grating spectroscopy measures quasiparticle propagation. It can track changes in metallic materials as they are irradiated.
- Ultraviolet photoelectron spectroscopy(UPS)
- Ultraviolet–visible spectroscopy
- Vibrational circular dichroism spectroscopy
- Video spectroscopy
- X-ray photoelectron spectroscopy (XPS)

2.3 Applications

- Cure monitoring of composites using optical fibers.
- Estimate weathered wood exposure times using near infrared spectroscopy.[13]
- Measurement of different compounds in food samples by absorption spectroscopy both in visible and infrared spectrum.

Measurement of toxic compounds in blood samples

2.4 Powdered milk

Powdered milk or dried milk is a manufactured dairy product made by evaporating milk to dryness. One purpose of drying milk is to preserve it; milk powder has a far longer shelf life than liquid milk and does not need to be refrigerated, due to its low moisture content. Another purpose is to reduce its bulk for economy of transportation. Powdered milk and dairy products include such items as dry whole milk, nonfat (skimmed) dry milk, dry buttermilk, dry whey products and dry dairy blends. Many dairy products exported conform to standards laid out in Codex Alimentarius. Many forms of milk powder are traded on exchanges.

Powdered milk is used for food and health (nutrition), and also in biotechnology.

2.4.1 Milk Powder Production

Raw milk on arrival at the factory is rapid tested for temperature, hygiene, antibiotics, water addition and adulteration.

On acceptance the milk is pumped into a silo storage tank at the processing plant and held at temperatures below 7 C and usually below 5 C.

Raw whole milk has varying Fat & Solids Not Fat (SNF) content and typically between 3.5% and 4.5% fat and 8% to 9%

SNF giving typical Total Solids of 12.5%. Developing countries are milk solids usually lower and care should be taken in ensuring that costs / yields are calculated accurately for any planned high volume dairy manufacturing plant and we suggest you prepare a very detailed yield spreadsheet. Use the calculator on the link above to view the effect of 1.0 %protein or moisture variation on annual milk powder production yield and milk powder profitability.

Milk is mostly collected in insulated tankers from dairy farms or milk collection stations. Prior to collection the driver will carry out quality and measurement tests to ensure the milk is of the required quality.

Adulteration of raw milk in some countries is prolific and a real barrier to globally operating companies establishing facilities due to the high brand risk involved.

When the raw milk arrives at the manufacturing plant it is usually separated into cream and skimmed milk to enable standardization of the fat content prior to spray drying. High volume manufacturers will automate this cream fat standardization using an inline “standomat” which doses cream back into the skim to give the correct fat % in the milk to be processed.

Some high volume plants particularly in the US also standardise the SNF content to maximise yields and give a consistent quality.

The microbial quality of milk powders is very important and it is possible at this early stage of processing to take out 99.9% of the spore-forming bacteria by either bacto-fugation or microfiltration prior to heat treatment.

This is an ideal next stage but many processors primarily due to cost do not include this stage.

Both processes require that the milk is first separated and then the skimmed milk portion has the bacteria removed and the cream is then high temperature pasteurised and returned to the milk (if required) This is the method used for ESL milk (Extended Shelf Life). Infant Formula manufacturers operating in developing countries should use a bactofuge or microfiltration plant to ensure finished product quality.

The milk is high temperature short-time pasteurized (HTST) by heating to at least 72 C, and holding at or above this temperature for at least 15 seconds, (An equivalent temperature / time combination can be used). Most high volume liquid milk plants now operate on a higher holding time of 25 to 35 as a precaution over the possible survival of MAP which can cause Crohn's disease in humans. *Mycobacterium avium* subspecies *paratuberculosis* may be capable of surviving pasteurization(.

Heat treatment affects the functional properties of skim milk powder and the keeping quality of whole milk powder, so the temperature and time combinations used by manufacturers can vary widely depending on the required properties required of the finished powder.

In skimmed milk powder, the extent of heat treatment (and holding time) can be measured by the whey protein nitrogen index (WPNI), which measures the amount of un-denatured whey protein.

Skimmed milk powder processing differs from whole milk and buttermilk processing by the heat treatment given to skim milk before evaporation.

The skimmed milk heat treatment temperature coupled with the holding time determines the heat classification of the powder produced. For skim milk powder classified as “low-heat”, the milk is low temperature pasteurized with little or no holding required, while heat treatment for a “high-heat” method requires heating milk to 85-88 C and holding at this temperature for 15 to 30 seconds..There is no requirement to homogenize skimmed milk for powder production because of its low fat content. High heat, heat stable powders are also produced by varying the evaporation conditions.

Homogenisation is not a mandatory step in whole milk or buttermilk processing, but is usually applied in order to decrease the free fat content.

2.5 X ray Fluorescence

XRF is an analytical method to determine the chemical composition of all kinds of materials. The materials can be in solid, liquid, powder, filtered or other form.

XRF can also sometimes be used to determine the thickness and composition of Layers and coatings. The method is fast, accurate and non-destructive, and usually requires only a minimum of sample preparation. Applications are very broad and include the metal, cement, oil, polymer, plastic and food industries, along with mining, mineralogy and geology,

and environmental analysis of water and waste materials. XRF is also a very useful analysis technique for research and pharmacy.

Spectrometer systems can be divided into two main groups: energy dispersive systems (EDXRF) and wavelength dispersive systems (WDXRF), explained in more detail later. The elements that can be analyzed and their detection levels mainly depend on the spectrometer system used. The elemental range for EDXRF goes from sodium to uranium (Na to U). For WDXRF it is even wider, from beryllium to uranium (Be to U). The concentration range goes from (sub) ppm levels to 100%. Generally speaking, the elements with high atomic numbers have better detection limits than the lighter elements.

The precision and reproducibility of XRF analysis is very high. Very accurate results are possible when good standard specimens are available, but also in applications where no specific standards can be found.

The measurement time depends on the number of elements to be determined and the required accuracy, and varies between seconds and 30 minutes. The analysis time after the measurement is only a few seconds.

Figure 1 shows a typical spectrum of a soil sample measured with EDXRF - the peaks are clearly visible. The positions of the peaks determine the elements present in the sample, while the heights of the peaks determine the concentrations.

2.6 X-ray Fluorescence analyze

XRF analysis is a powerful analytical tool for the petrochemical determination of almost all the elements present in a sample. XRF radiation is induced when photons of sufficiently high energy, emitted from an X-

ray source, impinge on a material. These primary X-rays undergo interaction processes with the analyte atoms. High-energy photons induce ionization of inner shell electrons by the photoelectric effect and thus electron vacancies in inner shells (K, L, M,) are created. The prompt transition of outer shell electrons into these vacancies within some 100 fs can cause the emission of characteristic fluorescence radiation. Not all transitions from outer shells or subshells are allowed, only those obeying the selection rules for electric dipole radiation. The creation of a vacancy in a particular shell results in a cascade of electron transitions, all correlated with the emission of photons with a well-defined energy corresponding to the difference in energy between the atomic shells involved. The family of characteristic X-rays from each element including all transitions allows the identification of the element. Next to this radiative form of relaxation, a competing process can take place: the emission of Auger electrons. Both processes have Z-dependent probabilities that are complementary: the Auger yield is high for light elements and the fluorescence yield is high for heavy elements.

The working principle of XRF analysis is the measurement of wavelength or energy and intensity of the characteristic X-ray photons emitted from the sample. This allows the identification of the elements present in the analyte and the determination of their mass or concentration. All the information for the analysis is stored in the measured spectrum, which is a line spectrum with all characteristic lines superimposed above a certain fluctuating background.

Other interaction processes, mainly the elastic and inelastic scattering of the primary radiation on sample and substrate, induce the background.

Measurement of the spectrum of the emitted characteristic fluorescence radiation is performed using wavelength dispersive (WD) and energy dispersive (ED) spectrometers. In wavelength dispersive X-ray fluorescence analysis (WDXRF), the result is an intensity spectrum of the characteristic lines versus wavelength measured with a Bragg single crystal as dispersion medium while counting the photons with a Geiger-Müller, a proportional or scintillation counter. In energy dispersive X-ray fluorescence analysis

(EDXRF), a solid-state detector is used to count the photons, simultaneously sorting them according to energy and storing the result in a multichannel memory. The result is an X-ray energy vs. intensity spectrum. The range of detectable elements ranges from Be ($Z = 4$) for the light elements and goes up to U ($Z = 92$) on the high atomic number Z side. The concentrations that can be determined with standard spectrometers of WD or ED type lie in a wide dynamic range: from the percent to the $\mu\text{g/g}$ level. In terms of mass the nanogram range is reached with spectrometers having the standard excitation geometry.

By introducing special excitation geometries, optimized sources and detectors, the picogram and even femtogram range of absolute analyte detection capacity can be reached; in terms of concentrations, the same improvement factor can be attained, i.e. from the $\mu\text{g/g}$ towards the pg/g level under the best conditions.

In principle, XRF analysis is a multielement analytical technique and in particular, the simultaneous determination of all the detectable elements present in the sample is inherently possible with EDXRF. In WDXRF both the sequential and the simultaneous detection modes are possible.

The most striking feature of XRF analysis is that this technique allows the qualitative and quantitative analysis of almost all the elements (Be–U) in an unknown sample. The analysis is in principle nondestructive, has high precision and accuracy, has simultaneous multielement capacity, requires only a short irradiation time so that a high sample throughput is possible; on-line analysis is also possible and the running costs are low. The technique is extremely versatile for applications in many fields of science, research and quality control, has low detection limits, and a large dynamic range of concentrations covering up to 9 orders of magnitude. The physical size of an XRF spectrometer ranges from handheld, battery-operated field units to high-power laboratory units with compact tabletop units and larger ones requiring several cubic meters of space including a 10–20 kW electrical power supply and efficient cooling units with high pressure water and a heat sink.

In contrast to all these attractive properties there are some disadvantages. The absorption effects of the primary radiation and the fluorescence radiation created in the analyte result in a shallow layer a few tenths of a millimeter deep that provides information on its composition. This requires a perfectly homogeneous sample which often occurs naturally but must sometimes be produced by acid dissolution into liquids or by grinding and the preparation of pressed pellets. In both examples the feature of non-destructiveness is lost. Most ideally thin films or small amounts of microcrystalline structure on any substrate are the ideal analyte where also the quantification process is simple because there is linearity between fluorescence intensity and concentration. In thick samples corrections for absorption and enhancement effects are necessary.

While the roots of the method go back to the early part of this century, where electron excitation systems were employed, it is only during the last

30 years or so that the technique has gained major significance as a routine means of elemental analysis.

2.7 Basic Principles

2.7.1 X-ray wavelength and energy scales

The X-ray or Röntgen region of the electromagnetic spectrum start at ca. 10 nm and extends towards the shorter wavelengths. The energies of X-ray photons are of the same order of magnitude as the binding levels of inner-shell electrons (K, L, M, levels) and therefore can be used to excite and/or probe these atomic levels. The wavelength λ of an X-ray photon is inversely related to its energy E according to:

$$\lambda \text{ (nm)} = 1.24/E \text{ (keV)}$$

Where 1 eV is the kinetic energy of an electron that has been accelerated over a voltage difference of 1 V ($1\text{eV} = 1.602 \cdot 10^{-19}$ J). Accordingly, the X-ray energy range starts at 100 eV and continues towards higher energies. X-ray analysis methods most commonly employ radiation in the 1-50 keV (1 - 0.02 nm) range.

2.7.2 Interaction of X-rays with matter

When X-ray beam passes through matter, some photons will be absorbed inside the material or scattered away from the original path, as illustrated in Fig. 3.1. The intensity I_0 of an X-ray beam passing through a layer of thickness d and density ρ is reduced to an intensity I according to the well-known law of Lambert-Beer:

$$I = I_0 e^{-\mu\rho d}$$

The number of photons (the intensity) is reduced but their energy is generally unchanged. The term μ is called the mass attenuation coefficient and has the dimension cm^2/g . The product $\mu L = \mu\rho$ is called the linear

absorption coefficient and is expressed in cm^{-1} . $\mu(E)$ is sometimes also called the total cross-section for X-ray absorption at energy E .

Fig. 3.2 shows a log-log plot of the energy dependence of the mass attenuation coefficient of several chemical elements in the X-ray energy range between 1 and 100 keV. The absorption edge discontinuities (due to photoelectric absorption – see below) are clearly visible. Low Z materials attenuate X-rays of a given energy less than high Z materials. A given material will attenuate high energy (i.e. hard) X-rays less than low energy (soft) X-rays.

The mass absorption coefficient $\mu(M)$ of a complex matrix M consisting of a mixture of several chemical elements (e.g., an alloy such as brass), can be calculated from the mass attenuation coefficient of the n constituting elements:

$$\mu(M) = \sum_{i=1}^n w_i \mu_i \quad (2)$$

Where μ_i is the mass attenuation coefficient of the i th pure element and w_i its mass fraction in the sample considered. This is called the mixture rule.

The mass absorption coefficient μ plays a very important role in quantitative

XRF analysis. Both the exciting primary radiation and the fluorescence radiation are attenuated in the sample. To relate the observed fluorescence intensity to the concentration, this attenuation must be taken into account.

As illustrated in Fig. 3.1, the absorption of radiation in matter is the

Cumulative effect of several types of photon-matter interaction processes that take place in parallel. Accordingly, in the X-ray range the mass attenuation coefficient μ_i of element i can be expressed as:

$$\mu_i = \tau_i + \sigma_i \quad (3)$$

Where τ_i is the cross-section for photo-electric ionization and σ_i the cross-section for scattering interactions. All above-mentioned cross-sections are energy (or wavelength) dependent. Except at absorption edges (see below), μ is more or less proportional to $Z^4 \lambda^{-3}$.

This method can be used to detect and quantify elements within radioactive materials. The polycapillary focusing optics act as spatial filters to eliminate background radiation from the sample and increase detection sensitivity for sample elements of interest. Additionally, confocal XRF can be used for elemental depth profiling. Confocal XRF acts as a material probe by exciting and detecting emitted characteristic X-ray photons from within the confocal analysis volume as this volume is through the sample. In this way elemental concentrations can be measured on the object's surface and throughout the object's interior. Confocal X-ray fluorescence with X-ray optics has been used for many unique applications including thin film thickness measurement, forensics, art restoration, and small feature analysis.

2.8 PH Meter

A pH meter is a scientific instrument that measures the hydrogen-ion activity in water-based solutions, indicating its acidity or alkalinity expressed as pH.

The pH meter is the difference in electrical potential between a pH electrode and a reference electrode, and so the pH meter is sometimes referred to as a "potentiometric pH meter".

The difference in electrical potential relates to the acidity or pH of the solution.[3] The pH meter is used in many applications ranging from laboratory experimentation to quality control.

2.8.1 Applications

The rate and outcome of chemical reactions taking place in water often depends on the acidity of the water, and it is therefore useful to know the acidity of the water, typically measured by means of a pH meter.[5] Knowledge of pH is useful or critical in many situations, including chemical laboratory analyses. pH meters are used for soil measurements in agriculture, water quality for municipal water supplies, swimming pools, environmental remediation; brewing of wine or beer; manufacturing, healthcare and clinical applications such as blood chemistry; and many other applications.[4]

Advances in the instrumentation and detection have expanded the number of applications in which pH measurements can be conducted. The devices have been miniaturized, enabling direct measurement of pH inside of living cells.[6] In addition to measuring the pH of liquids, specially designed electrodes are available to measure the pH of semi-solid substances, such as foods. These have tips suitable for piercing semi-solids, have electrode materials compatible with ingredients in food, and are resistant to clogging.

Chapter Three

Methodology

Chapter Three

Methodology

3.1 Introduction

The aim of this chapter is to present the materials and apparatus and method used in this work (sample preparation and setup).

3.2 Materials

Two samples of milk powder (Kapo milk powder and Nedo milk powder) were collected by XRF and their PH were known by PH Meter.



Figure 3.1: sample of Kapo powder milk



Figure 3.2: Sample of Nedo powder milk

3.3 Apparatus



Figure 3.3 XRF device

XRF device used to analyze sample that by stimulate the atoms inside and determine the type of elements within the sample.



Figure 3.3: 3510 PH of Nedo powder milk.

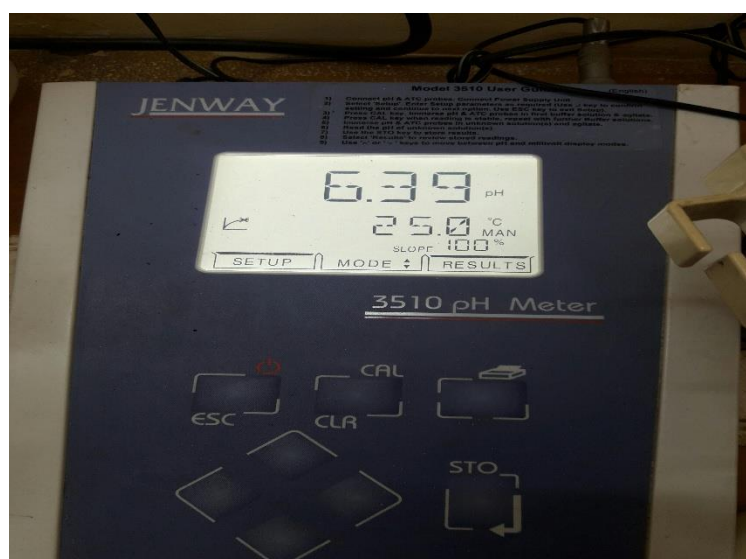


Figure 3.4: PH of Kapo powder milk

3.4 Method Kapo milk powder and Nedo milk powder samples were prepared and subjected to XRF device to detect some of the elements that make up the samples, also subjected to PH meter to know the PH of two samples.

Table 3.1: The result of Nedo milk powder

The Element	Concentration%
Cr	0.06
Mn	0.00
Fe	0.11
Ni	<0.01
Cu	0.00
Zn	0.02
Pb	0.00

Table 3.2The: The result of kapo milk powder

The Element	Concentration%
Cr	0.02
Mn	0.02
Fe	0.04
Ni	<0.00
Cu	0.00
Zn	0.00
Pb	0.01

Chapter Four

Results and Conclusion

Chapter four

Discussion, conclusion, recommendations

4.1 Introduction

This Chapter obtains the results of this research and conclusion and recommendations.

4.2 X-Ray Fluorescence of Samples

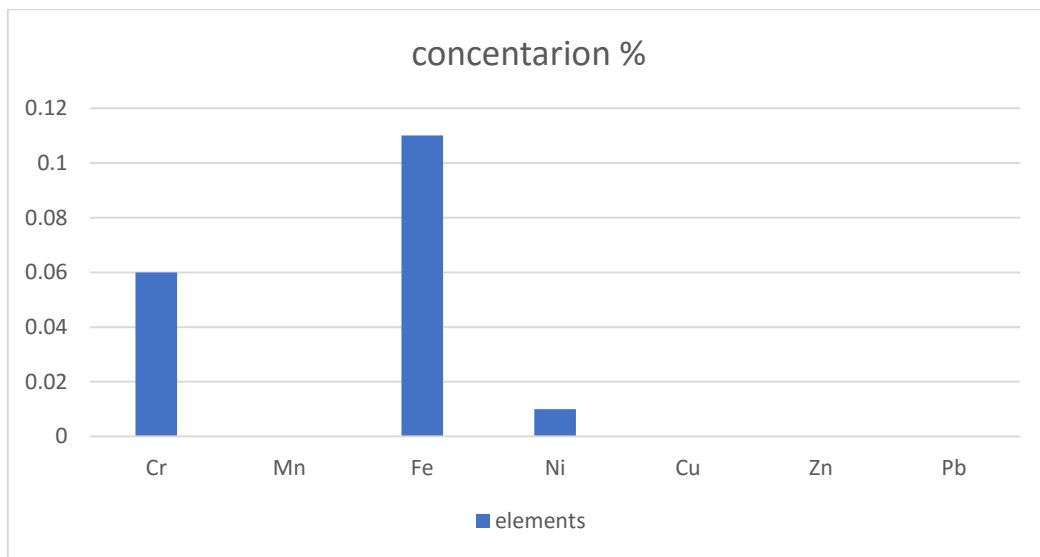


Figure 4.1 shows the analyze sample of Nedo Powder milk by XRF.

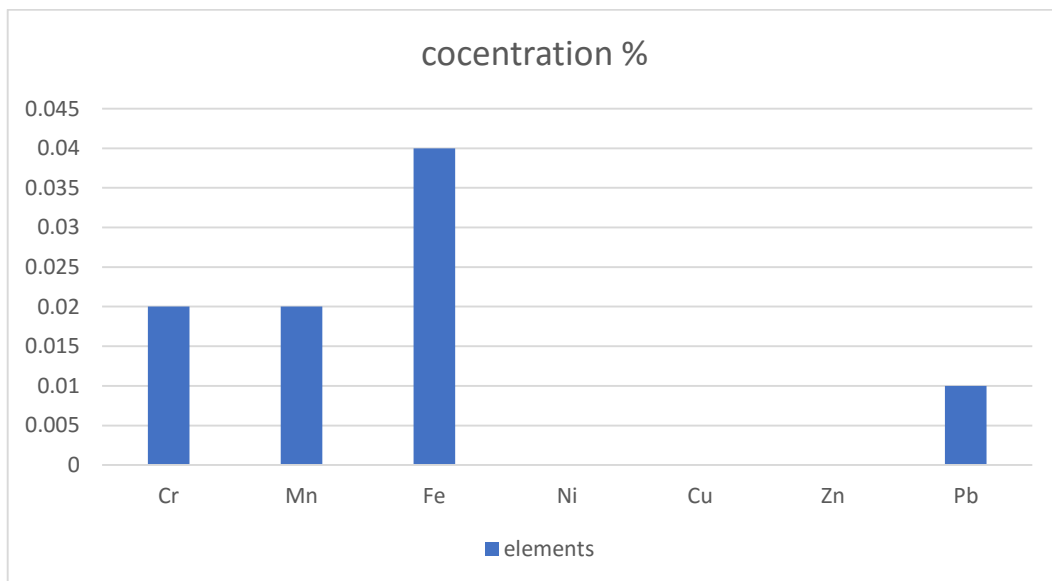


Figure 4.2 shows the analyze sample of kapo Powder milk by XRF.

4.3 The Results:

The elements in two samples were found in different concentrations; wick in Nedo powder milk found chromium element, its concentration was 0.06%, manganese element, concentration 0.00%, Iron element and its concentration was 0.11%, nickel element, its concentration <0.001%, copper element, its concentration 0.00%, zinc element, its concentration 0.02 %and lead element and its concentration was 0.00%, and the PH of Nedo powder milk was 6.25.

The elements in Kapo powder milk sample were found in different concentrations also; wick found chromium element, its concentration was 0.02%, manganese element, concentration 0.02%, Iron element and its concentration was 0.02%, nickel element, its concentration <0.00%, copper element, its concentration 0.00%, zinc element, its concentration 0.00%and lead element and its concentration was 0.01%, and the PH of Kapo powder milk was 6.39.

4.4 Conclusion:

Nedo powder milk and Kapo powder milk contain some important elments that which the human body need them, but Nedo powder milk contains some elements that their concentrations more than some elements in Kapo powder milk, like chromium element, iron element, zinc element and Kapo powder milk contains lead element that Nedo powder milk doesn't contain it, so Nedo powder milk is better than kapo powder milk.

4.5 Recommendation

The further studies in this research problem could be done using another devices; like FTIR, UV-Vis spectrometer ,Raman spectroscopy, X-Ray Diffraction (XRD), also could be done using XRF also but to another samples and people have to use Nedo powder milk rather than Kapo Pomer milk.

References

- [1] Mart Kuresoo, Head of Analysing Laboratory Department November 12th, 2013.
- [2] PANalytical B.V., Almelo, the Netherlands, 2010.
- [3] Skoog, Douglas A., Holler, F. James, Crouch, Stanley R, (principle of instrumental analysis (6th ed). Belmont, CA: Thomson Brooks/Cole. pp. 169-173. ISBN 9780495012016.
- [4] Garcia-Compean, D., Jaquez-Quintana, J.O., Gonzalez-Gonzalez, J.A. and Gigahertz Optics." Reflection, Transmission, and Absorption". [Online] available from: <http://light-measurement.com/reflectioabsorption/>. [Accessed on 2015-08].
- [5] <http://www.diabetes.org/heart-disease-stroke.jsp>