Sudan University of Science and Technology

College of Graduate Studies

Investigation of the Contamination Elements Founded in Powder Milk

التحقق من العناصر الملوثة الموجود في لبن البودرة

A Thesis Submitted in Partial Fulfillment for requirement of

A Master Degree in Physics

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الآيــة

قال تعالي:

((وَلَقَدْ عَلِمْتُمُ النَّشْأَةَ الأُولَى فَلَوْلا تَذَكَّرُونَ))(62)

صدق الله العظيم

سورة الواقعة.

Dedication

To whom my words are not enough to express my deep indebtedness, thanks and gratefulness.

To my parents the sustainable source of tenderness support and specific.

And to the pure spirit my grand mother

And my sisters

And my fiancée

And my friends

Acknowledgment

Grapple with the characters overcome feelings of words and their tongues to provide thanks are recognition to all helped me to complete this research and humble thanks to the an example of science and perseverance, my supervisor:

Dr. Rawia Abdelghani Elobaid and to Dr. Ali Silliman Mohammed and to my collage mate Dr. Ahmed Mohamed Salh and to the NDT manger Astaz Mohamed Abdullah and to the staff at my university for providing me a place to study, to all of you many thanks and deep gratitude.

Abstract

Powder milk and liquid milk are considered as one of the responsible food contamination with heavy metals. The objectives of the study were to assess the content of selected metals in milk and its associated human health risks in the food chain. A total of 6 powder and 2 liquid milk samples of branded, were collected from different companies in city market randomly. The metals and trace elements founded are Chromium (Cr), Lead (Pb), Manganese (Mn), Copper (Cu), Iron (Fe), Nickel (Ni), Zinc (Zn), Molybdenum (Mo),Vanadium (V), Cobalt (Co), Tungsten (W), Titanium (Ti), Tantalum (Ta), and Niobium (Nb), contents in collected milk samples were determined using X-rays fluorescence (XRF) spectrometer. To ensure quality control, one used computer Microsoft office program to draw diagrams.

المستـــــخلص

يعتبر لبن البودرة و الحليب السائل واحد من الأغذية الملوثة بالمعادن الثقيلة, و الهدف من هذه الدراسة التعرف علي بعد المعادن المختارة و ما يرتبط بها من المخاطر علي صحة الإنسان في السلسلة الغذائية.أخذت 6 عينات من لبن البودرة الجاف و 2 عينة من الحليب السائل من علامات تجارية مختلفة تم جمعها من أسواق المحلية في المدينة. ووجد أن المعادن و العناصر الملوثة وهي الكروم و الرصاص والمغنيزيوم والنحاس والتنجيستن والتنتاليوم والنيكل والزنك والموليبدنيوم والفاناديوم والكوبالت و النوبيون , تم تحديد المكونات الموجودة في العينات لبن البودرة الجاف والسائل بواسطة استخدام مطياف الأشعة السينية المتوهجة. و استخدمت برامج كمبيوتر ميكروسوفت اكسل لرسم النسب المئوية.

Contents

Content	Page
الاية	I
Dedication	Ii
Acknowledgments	Iii
Abstract/English	V
المستخلص	Vi
Contents	Vii
Chapter One **Introduction**	
Basic elements analysis	1
The problem of research	2
The aim	2
Literature review	3
The presentation	3
Chapter Two **Non-destructive testing**	
Introduction	4
Basic principle of Non-destructive Testing (NDT)	5
Definition and methodology of application basic methods	5
NDT methods	5
Visual testing (VT)	6
Liquid penetrating testing (PT)	7
Magnetic particle testing (MT)	8
Eddy current testing (ET)	11
Ultrasonic testing (UT)	12
Comparison of different NDT methods	14
New development in NDT	14
Acoustic emission (AE)	16
Materials and defects physical and properties of materials	16
Metallic materials	16
Elastic and plasticity	17
Strength	18
Hardness	19

Ductility	19
Malleability	19
Brittleness	20
Notch toughness	20
Electrical conductivity	20
Non-metallic materials	21
Ceramics	21
Composite	22
Concrete	22
Structures metals and alleys	23
Chapter Three **XR fluorescence**	
Introduction	24
The XRF fluorescent	24
Basic of XRF	25
The x-rays	26
Interaction of x-rays with matter	26
Production of characteristic fluorescent radiation	27
Absorption and enhancement effects	31
Absorption and analysis depths	32
Raleigh and Compton scatter	32
Polarization	34
XRF analysis	35
Samples preparation	35
Powders	35
Liquid	35
Chapter Four **Experiment**	
Introduction	37
Apparatus of experiment	37
Methodology	37
Result	37
Discussion	46
Conclusion	46
Recommendation	46
References	47

List of figures

Fig 2.1 Various optical aids used in inspection	6
Fig 2.2 Different stages of liquid penetrate	8
Fig 2.3 Basic principle of magnetic particle testing	9
Fig 2.4 Different magnetization used in magnetic particle testing	10
Fig 2.5 (a) Generation of eddy currents in the test object (b) Distortion of eddy currents due to defect	11
Fig 2.6 Arrangement for radiography testing method.	12
Fig 2.7 Basic components of an ultrasonic flaw detection system	13
Fig 2.8 Stress-strain elastic and plastic deformation and fracture.	18
Fig 2.9 Schematic illustration of a metallic bond	23
Fig 3.1 Typical spectrum of a soil sample measured with and EDXF spectrometer.	25
Fig 3.2 X-rays and other electromagnetic radiation	26
Fig 3.3 Three main interactions of x-rays with matter	27
Fig 3.4 Production of characteristic fluorescent radiation.	28
Fig 3.5 Fluorescent yield for K and L electrons.	29
Fig 3.6 Major lines and their transitions.	30
Fig 3.7 Absorption of incoming and fluorescent X-ray	31
Fig 3.8 primary and secondary fluorescent	32
Fig 3.9 Compton scatter.	33
Fig 3.10 Rayleigh scatter.	33
Fig 3.11 Rayleigh scatter for light and heavy element	34
Fig 3.12 X-ray polarize in vertical direction .	35
Fig 4.1 powdered milk from Alwaddi.	38
Fig 4.2 Powdered milk from Delta.	39
Fig 4.3 powdered milk from CAPO.	40
Fig 4.4 powdered milk from Junion.	41
Fig 4.5 Powdered milk from Neel Cow.	42
Fig 4.6 Powdered milk from milko.	43
Fig 4.7 Liquid milk from local market.	44
Fig 4.8 Liquid milk from CAPO.	45

List of Tables

Tab 4.1 Powdered milk from Alwaddi.	38
Tab 4.2 Powdered milk from Delta.	39
Tab 4.3 Powdered milk from CAPO.	40
Tab 4.4 Powdered milk from Junion.	41
Tab 4.5 Powdered milk from Neel Cow.	42
Tab 4.6 Powdered milk from Milko.	43
Tab 4.7 Liquid milk from local market.	44
Tab 4.8 Liquid milk from CAPO.	45

Chapter One

INTRODUCTION

1.1 Basic Elements Analysis:

The analysis of milk is important because milk is an indication of environment contamination, a significant and pathway for toxic metal in take by humans and a source of essential nutrients. Essential elements required by the human body include the four basic elements H, C, N, CL, and the essential trace elements Mn, Fe, Ni, Cu, Zn, Mo, Ni, Cr, Co, V, W, Ti, Ta, Nb. 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 levels in food stuffs especially milk. Could play an important role in understanding a number of deficiency-related diseases. Diet quality is determined by the proportion of individual component, 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 other are heavy metals or toxic elements, which demonstrate their toxic influence on human health and a deficiencies on human health and growth even at small concentrations. Some diseases are clearly correlated with and imbalance on human body. The excess or deficiencies on human health. For, example a deficiency in Fe causes anemia and deficiency in Zn is association with depressed sexual immaturity and skin lesions. In contrast, an excess of Mn may inhabit Fe absorption and an excess of Zn may cause a Cu deficiency. The determination of minor and trace elements in milk powder can also be used as an indicator for environmental pollution [1]. X-rays fluorescent (XRF) analysis was employed for the precise and accurate determination of the elements

because it is a powerful method for the direct analysis of solid, liquid food samples without dissolution, thus eliminating the possibility of contamination. Thus technique also has low detection limits for many inorganic constituents, moreover ,the quality control and quality assurance of this technique is maintained through the use of reference materials .The main goal of this work was to investigate the concentrations of certain elements in powdered milk samples using X-rays fluorescence to analysis the presence for the trace elements and passive technique to determine the natural radioactivity The objective of this study was to measure the concentrations of Cr, Cu, Fe, Ni, Nb, Mo, Mn, Cr, V, W, Ta, Co, Zn, and Ti in the milk samples using prompt XRF analysis. This technique performs a simultaneous, sequential and biological samples with the advantage of a short analysis and low limit of detection [1].

1.2 The Problem of Research:

There are radioactive elements in powder milk. Founded in this research contamination elements in powder milk, which it is damage health of human life.

1.3 The Aim:

There are many dry dairy companies and their consumption increasing in a community as a daily food. I have the idea that we will study the chemical elements found in dry powder milk and liquid milk.

1.5 Literature Review:

(1) Investigations of X-Rays on rate Serum Albumin Marked with Tritium – Labeled Leucine. By: J. Done And P.R. Paye (February 1956).

(2) Quantitative Detection of Melamine Adulteration in Milk Powder Using X-Ray Spectroscopic Technique. By: Chickkappa Udagani & Thimmasandra Narayan Ramesh .Issue In 2 Fe 2014.

(3) Assessment of Natural Radio nuclides in Powdered Milk Consumed in Iraq. By: Sahar A, Mohammed S.M. Al-kafaje.

(i) Environment Research Center, University of Technology Baghdad. Iraq.

(ii) Department of Laser Engineering and Electronic Optics. Issue 2016.

(4) Methodology for Determination of Radio strontium in Milk. By: Stephanie Brun, Yaun Kergdallan, Bernadette Baursier, Jean Marc Fremy, Francoise Janin. Jan,2003.
(5) Investigation of the Potassium Does Founded in Powdered Milk. By: ImanSedik Ahmed Al. issue 2015.
(6) Potassium Concentration by Natural K40, Ar40, γ-Rays Detection in Four Basic Diet Products (Milk, Eggs, Wheat and Corn). By: Juan Maneul Navarrete, Trinided Martinz, Luis Cabrera, PilarLizarraga, Miguel Angel. August 30 ,2011.

1.4 Presentation:

This research includes on four chapters. In chapter one we review introduction of the heavy metals and trace elements. In chapter tow we review the non-constructive testing (NDT), and materials and defects –physical properties of materials. In chapter three we review the X-Rays Fluorescence Analysis .In chapter four we talked about experiment analysis samples and results of powder liquid milk with tables and figures, discuss the results, conclusions and references.

Chapter Two

Non-Destructive Testing

2.1 Introduction:

The field of Nondestructive Testing (NDT) is a very broad, interdisciplinary field that plays a critical role in assuring that structural components and systems perform their function in a reliable and cost effective fashion. (NDT) technicians and engineers define and implement tests that locate and characterize material conditions and flaws that might otherwise cause planes to crash, reactors to fail, trains to derail, pipelines to burst, and a variety of less visible, but equally troubling events. These tests are performed in a manner that does not affect the future usefulness of the object or material. In other words, (NDT) allows parts and materials to be inspected and measured without damaging them. Because it allows inspection without interfering with a product's final use, (NDT) provides an excellent balance between quality control and cost-effectiveness. Generally speaking, (NDT) applies to industrial inspections. While technologies are used in (NDT) that are similar to those used in the medical industry, typically nonliving objects are the subjects of the inspections. This section will provide a basic introduction to materials and material fabrication processing. It is important that (NDT) personnel have some background in material science for a couple of reasons. First, nondestructive testing almost always involves the interaction of energy of some type (mechanics, sound, electricity, magnetism or radiation) with a material. To understand how energy interacts with a material, it is necessary to know a little about the material. Secondly, (NDT) often involves detecting manufacturing defects and service induced damage and, therefore, it is necessary to understand how defects and damage occur**.**

2.2 Basic Principles of Non-Destructive Testing (NDT):

2.2 .1 Definition And Methodology of Applications of Basic NDT Methods:

NDT plays an important role in the quality control of a product. It is used during all the stages of manufacturing of a product. It is used to monitor the quality of the: (a) Raw materials which are used in the construction of the product (b) Fabrication processes which are used to manufacture the product. (c) Finished product before it is put into service. Use of (NDT) during all stages of manufacturing results in the following benefits: (a) It increases the safety and reliability of the product during operation. (b) It decreases the cost of the product by reducing scrap and conserving materials, labor and energy. (c) It enhances the reputation of the manufacturer as producer of quality goods. (d) It enables design of new products. All of the above factors bring profitability to the manufacturer. (NDT) is also used widely for routine or periodic assessment of quality of the plants and structures during service life. This increases the safety of operation and eliminates any forced shut down of the plants.

2.2.2 NDT Methods:

For the purposes of these notes, (NDT) methods may be divided into conventional and nonconventional. To the first group belong commonly used methods like visual or optical inspection, liquid penetrate testing, magnetic particle testing, eddy current testing, radiographic testing and ultrasonic testing. The second group includes those (NDT) methods used only for specialized applications like neutron radiography, acoustic emission, infrared testing, microwave techniques, leak testing, holography etc. It must also be remembered that none of these methods provide solutions to all possible problems, i.e. they are not optional alternatives but rather complementary to each other [12]. The

basic principles, typical applications, advantages and limitations of the conventional methods will now be briefly described.

2.2.3 Visual Testing (VT):

Often overlooked in listings of NDT methods, visual inspection is one of the most common and powerful means of non-destructive testing. Visual testing requires adequate illumination of the test surface and proper eye-sight of the tester. To be most effective visual testing requires training (knowledge of product and process, anticipated service conditions, acceptance criteria, record keeping, for example). It is also a fact that all defects found by other (NDT) methods ultimately must be substantiated by visual testing. Visual testing can be classified as direct visual testing, remote visual testing and translucent visual testing. Often the equipment needed is simple. Fig.2.1 shows a portable light, a mirror on stem, a 2X or 4X hand lens, one illuminated magnifier with magnification 5X or10X. For internal inspection, light lens systems such as bores copes allow remote surfaces to be examined. More sophisticated devices of this nature using fiber optics permit the introduction of the device into very small access holes and channels. Most of these systems provide for the attachment of a camera to permit permanent recording [13].

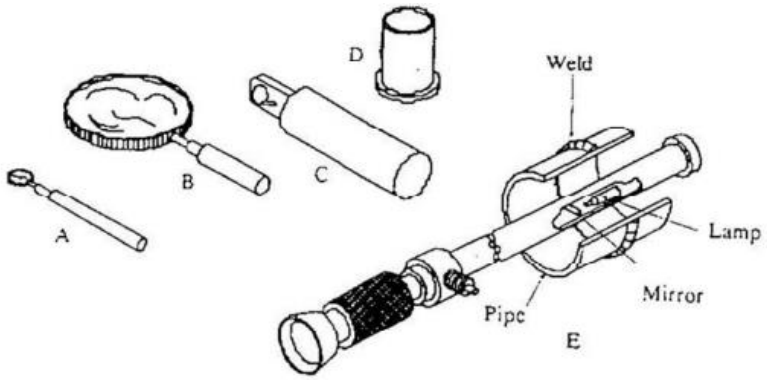

Fig: (2.1) Various optical aids used in visual inspection. (a) Mirror on stem (b) Hand magnifying glass (c) Illuminated magnifier (d) Inspection glass (e) Bore scope [13].

The applications of visual testing include:
a) Checking of the surface condition of the component. (b) Checking of alignment of mating surfaces. (c) Checking of shape of the component. (d) Checking for evidence of leaking. (e) Checking for internal side defects.
Some of the advantages of visual testing are as follows:
(a) Testing is simple (b) Testing speed is high (e) Testing is possible while test object is being used (f) Permanent records are available when latest equipment are used. Some of the limitations of visual testing are as follows:
a) Can detect only surface flaws (b) Eye resolution is weak (c) Eye fatigue

2.2.4 Liquid Penetrate Testing (PT):

This is a method that can be employed for the detection of surface-breaking defects in any industrial product made from a non-porous material [13]. This method is widely used for testing of non-magnetic materials. In this method, a liquid penetrated is applied to the surface of the product for a certain predetermined time, after which the excess penetrate is removed from the surface. The surface is then dried and a developer is applied to it. The penetrate which remains in the defect is absorbed by the developer to indicate the presence as well as the location, size and nature of the defect. The process is illustrated in (Fig. 1.2). Penetrates used are either visible dye or fluorescent dye. The inspection for the presence of visible dye indications is made under white light while inspection of presence of indications by fluorescent dye penetrates is made under ultraviolet (or black) light under darkened conditions. Liquid penetrates processes are further sub-divided according to the method of washing of the component. Penetrates can be: (i) water-washable, (ii) post-emulsifiable, i.e. an emulsifier is added to the excess penetrate on surface of the component to make it water-washable, and (iii) solvent removable, i.e. the excess penetrate needs to be dissolved in a solvent to remove it from the component surface. In order of decreasing sensitivity and decreasing cost, the liquid penetrate processes can be listed as follows: (a) Post emulsifiable fluorescent dye penetrated. (b) Solvent removable fluorescent dye penetrate. (c) Water washable fluorescent dye penetrate. (d) Post emulsifiable visible dye penetrate. (e) Solvent removable visible dye penetrate. (f) Water washable visible dye penetrate. The advantages of liquid penetrate testing are: (a) Relatively low cost. (b) High portability. (c) Highly sensitive to fine, tight cracks. (d) Fairly simple method. (e) Can be used on a variety of materials. (f) All surface defects are detected in one operation, regardless of orientation. The limitations of liquid penetrate testing are : (a) Test surface must be free of all contaminants (dirt, oil, grease, paint, rust, etc.). (b) Detects surface defects only. (c) Cannot be used on porous surfaces and is difficult to use on very rough surfaces. (d) No permanent record [13].

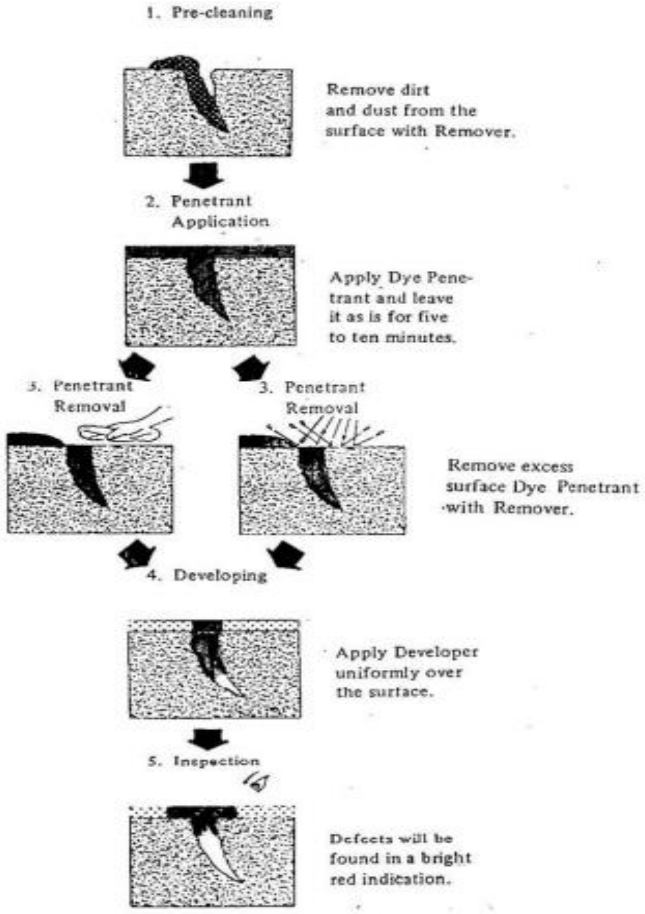

Fig: (2.2) Different stages of liquid penetrate testing [13].

2.2.5 Magnetic Particle Testing (MT):

Magnetic particle testing is used for testing materials that can be easily magnetized. This method is capable of detecting open to surface and just below the surface defects. In this method the test object is first magnetized by using either a permanent or an electromagnet, or by passing electric current through or around the object. The magnetic field thus introduced into the object is composed of magnetic lines of force. Whenever there is a defect which interrupts the flow of magnetic lines of force, some of these lines must exit and re-enter the object. These points of exit and re-entry form opposite magnetic poles [13]. Whenever minute magnetic particles are sprinkled onto the surface of such an object, these particles are attracted by these magnetic poles to create

a visual indication approximating the size and shape of the defect. Fig (2.3) illustrates the basic principles of this method [13].

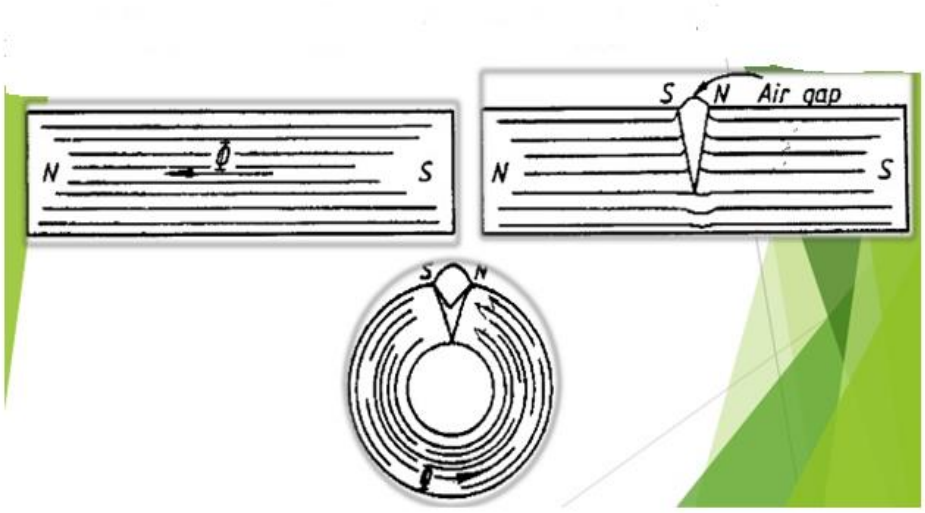

Fig: (2.3) Basic principle of magnetic particle testing [13].

Depending on the application, there are different magnetization techniques used in magnetic particle testing. These techniques can be grouped into two categories:

(a) Direct current techniques: These are the techniques in which the current flows through the test object and the magnetic field produced by this flow of current is used for the detection of defects. These techniques are shown in Fig. 2.4 (a, b & c).(b) Magnetic flux flow techniques: In these techniques magnetic flux is induced into the object either by the use of a permanent magnet or by flowing current through a coil or a conductor. These techniques are shown in (Fig. 2.4) (d–g). Advantages of magnetic particle testing:

(a) It does not need very stringent pre-cleaning operation.

(b) Best method for the detection of fine, shallow surface cracks in ferromagnetic material.

(c) Will work through thin coating.

(d) Inspection of complex geometries.

(e) Portable (NDT) method. The limitations of magnetic particle testing include the following:

(a) Applicable only to ferromagnetic materials.

(b) Orientation and strength of magnetic field is critical. There is a need to magnetize twice: longitudinally and circumferentially.

(c) Large currents sometimes required and "burning" of test parts is a possibility.

(d) After testing the object must be demagnetized, which may be difficult sometimes [13].

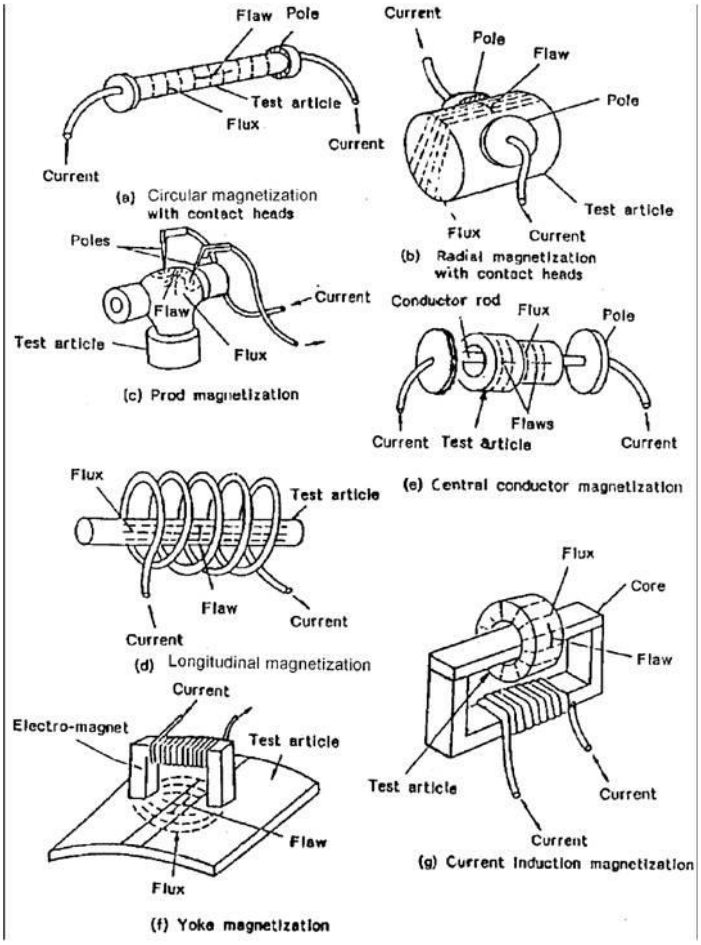

Fig: (2.4) Different magnetizations used in magnetic particle testing.

2.2.6 Eddy Current Testing (ET):

This method is widely used to detect surface defects, to sort materials, to measure thin walls from one surface only, to measure thin coatings and in some applications to measure casehardening depth. This method is applicable to electrically conductive materials only. In the method, eddy currents are induced in the object by bringing it close to an alternating current carrying coil. The alternating magnetic field of the coil is modified by the magnetic fields of the eddy currents. This modification, which depends on the condition of the object near to the coil, is then shown as a meter reading or cathode ray oscilloscope presentation. (Fig.2.5) shows generation and distortion of eddy current. There are three types of probes (Fig. 2.6) used in eddy current testing. Internal probes are usually used for the in-service testing of heat exchanger tubes. Encircling probes are commonly used for the testing of rods and tubes during manufacturing. The uses of surface probes include the location of cracks in plates, sorting of materials, measurement of wall and coating thickness, and case depth measurement. The ET method may be used for: (a) Detection of defects in tubes. (b) Sorting materials. (c) Measurement of thin wall thickness from one surface access. (d) Measurement of thickness of thin coatings and case depth. (e) Noncontact testing of hot defects.

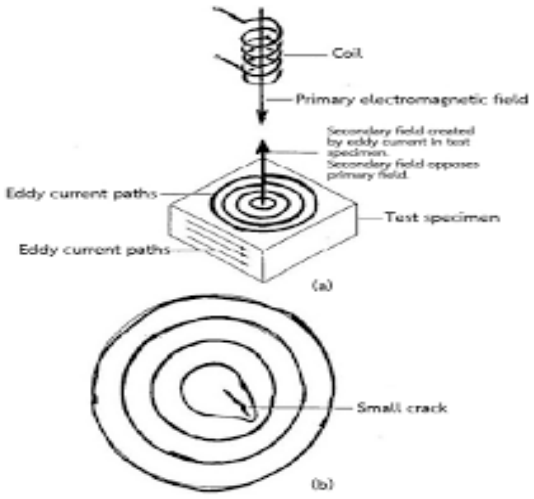

Fig: (2.5) (a) Generation of eddy currents in the test object. (b) Distortion of eddy currents due to defect [13].

The advantages of eddy current testing include that it: (a) does not require couplant. (b) Gives instantaneous response.(c) Is extremely sensitive to surface cracks.(d) Allows use of high scanning speeds (as high as 10 m/s).(e) Accurate for sizing defects and coating thickness measurement[12].The limitations of eddy current testing include the following: (a) Extremely sensitive to surface variations and therefore requires a good surface. (b) It is applicable to electrically conducting materials only. (c) Not reliable on carbon steel for the detection of subsurface flaws. (d) Its depth of penetration is limited to 8 mm. (e) The thickness range that can be inspected is limited. (f) Sensitivity of inspection decreases with thickness of the test object. (g) Considerable skill is required for interpretation of the radiographs. (h) The depth of defect is not indicated readily. X rays and gamma rays are hazardous to human health. The radiation safety series are referred for personal safety and radiation protection.

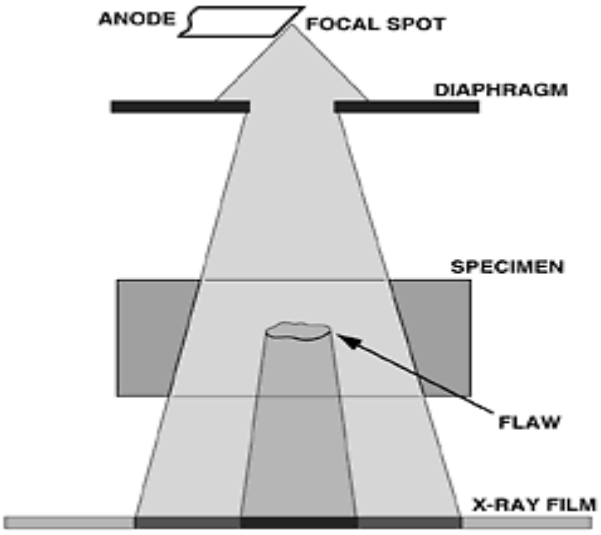

Fig: (2.6) Arrangement for radiographic testing method [13].

2.2.7 Ultrasonic Testing (UT):

Ultrasonic inspection is a non-destructive method by which high frequency sound waves are introduced into the object being inspected. Most ultrasonic inspection is done at frequencies between 0.5 and 20 MHZ. The sound waves travel through the material with some loss of energy (attenuation) due to material characteristics. The intensity of sound waves is either measured, after reflection (pulse echo) at interfaces (or flaw) or is measured at the opposite surface of the specimen (pulse transmission). The reflected beam is detected and analyzed to define the presence and location of flaws. The degree of reflection depends largely on the physical state of matter on the opposite side of the Interface. Partial reflection occurs at metal liquid or metal-solid interfaces [13].

Ultrasonic testing has a higher penetrating power than radiography and can detect flaws deep in the test object (up to about 7 meters of steel). It is quite sensitive to small flaws and allows the precise determination of the location and size of the flaws. The ultrasonic testing method is:
(a) Used for detection of flaws in materials and for thickness measurement.
(b) Used for the determination of mechanical properties and grain structure of materials. Some of the advantages of ultrasonic testing are that:
(a) It has high sensitivity which permits detection of minute defects.
(b) It has high penetrating power which allows examination of extremely thick sections. (c) It has a high accuracy of measurement of flaw position and size.
(d) It has fast response which permits rapid and automatic inspection.
(e) It needs access to only one surface of the specimen. Some of the limitations of this method are:(a) Unfavorable geometry of the test object causes problems during inspection.(b) Inspection of materials having coarse grain microstructure is difficult.(c) It requires the use of a couplant. (d) Defect orientation affects defect delectability. (e) Reference standards and calibration are required. (f) Rough surfaces can be a problem and surface preparation is necessary [13].

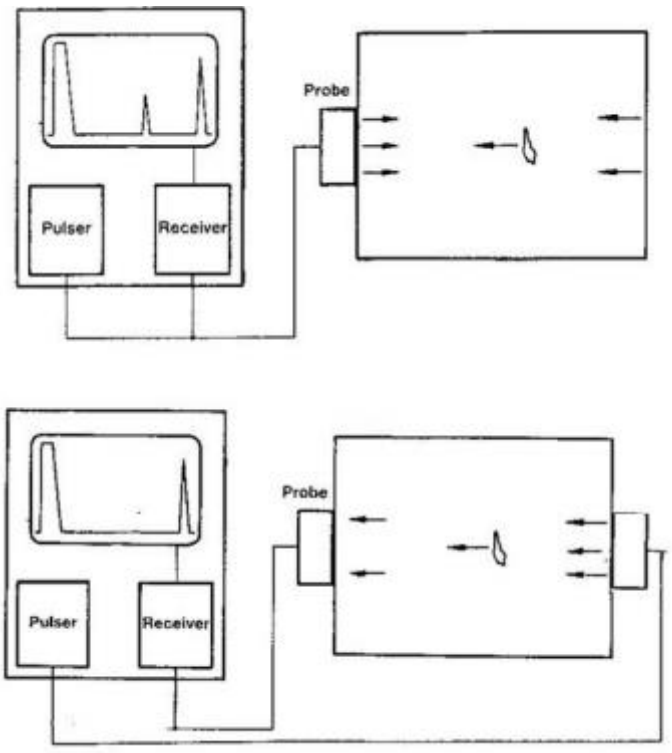

Fig: (2.7) Basic components of an ultrasonic flaw detection system [13].

2.3.1 Comparison of Different (NDT) Methods:

It is frequently necessary to use one method of NDT to confirm the findings of another. Therefore, various methods must be considered to be complementary rather than in competition with each other. Each method has its particular advantages and limitations and these must be taken into account when any testing program is planned.

2.3.2 New Developments in (NDT):

Many modern developments in all areas of technology require use of stronger, lighter materials, with other exotic properties and made of as little raw materials as possible. This is aimed at increasing the efficiency, durability and reliability of components made of such materials. There are stringent requirements for detecting and characterizing the extremely small sizes of flaws [13]. Consequently there is a great challenge to improve upon the existing (NDT) techniques and develop new ones where required. (NDT) has continued to play a vital role for quality control of industrial products in the highly aggressive and competitive world markets. It may not be totally out of place to say that the relative share of a particular country in the world market depends more and more on it investment in quality control and quality assurance, which in real terms and in most cases means an investment in (NDT). (NDT) equipment has been made more reliable and sensitive to make it as independent of operator errors as possible. This has led to a greater use of computers and automation. We see most of the modern (NDT) with microprocessors and computers with enhanced capabilities for data acquisition, analysis and image processing. There is a growing trend towards using multiple transducers and multi-channel systems both for ultrasonic and eddy current testing. Similarly the concept of using multiple methods of inspection simultaneously is increasing, for example for the inspection of reactor pressure vessels. On-line and continuous monitoring of plant and equipment inspection is now commonly applied. To cope with the increased use of composite materials, high sensitivity test methods such as micro-focus radiography and high frequency ultrasonic testing are now well established. (NDT) has been increasingly applied in process control as a means to fulfilling the requirement of the good quality assurance concept of making the products with zero defects. Such a shift towards use of (NDT) for process control has simultaneously demanded development in several related fields such newer and faster (NDT) techniques, computers and

data handling which bake it easier to analyze (NDT) data rapidly and use it in a feedback loop to modify, control and optimize the process. Thus, we see development of large installations employing automated radiography, tomography, ultrasonic testing, eddy current testing, optical-visual testing and infrared techniques. But increasing the degree of automation also increases the consequences of error. Therefore, a high degree of automation requires a high degree of (automated) monitoring and control. Consequently, a steady need for automated (NDT) is observed in industry. Process integrated (NDT) has to fulfill the requirements of today' s industrial production concerning integrate ability, automation, speed, reliability and profitability [13]. A confluence of developments in the fields of electronics, computer technology, simulation tools and signal processing is contributing to the excitement and fuelling some of the most compelling advances. With the availability of X ray tubes with focal spots as small as a few micros, high definition micro focal radiography with magnification is possible. Radiographic testing has seen rapid development of digital methods. Starting with laser-based digitization of conventional film radiographs, the technology has moved fast to the development of Imaging plates and digital detector rays also called flat panel detectors using semiconductors such as Europium-doped Ba F Br and CMSO or amorphous Si and a-Se or Cd Te. These have the capability of directly converting the X or gamma rays into electrical signals which can be fed to the computer for further processing and formation of images. The images can also be erased and the detectors be reused, for example, close to 1000 times. The conventional fluoroscopic methods have moved into the digital domain through the use of high quality image intensifiers: images can be directly photographed using the charge couple device (CCD) cameras which convert visual signals to electrical signals that can be processed by a computer. This offers the possibility of producing low cost real-time digital radiographic systems as against more expensive systems. Computed tomography, having the capability of producing three-dimensional images of internal structures of specimens, is now in use. High resolution digital micro-radiography is in the market; it uses X-ray machines with micron level focal spots. Characteristics corresponding to those of conventional radiographic images have been defined for digital images. The equivalent of optical film density is signal to noise ratio (SNR). The equivalent of film contrast is contrast to noise ratio (CNR), that of film resolution and granularity is Basic Spatial Resolution and that of dynamic range is Specific Material. Thickness range. Standards such as ASTM E2597 have been

developed to study the characteristics of digital radiographic systems. Digital radiography offers numerous benefits over conventional radiography including: (a) Radiation dosage and exposures are reduced resulting in less risk to the operator.(b) Reduces radiographic inspection time, cost and improves productivity. (c) Eliminates chemicals, chemicals disposal and chemical storage costs. (d) Allows radiographic data to be archived and analyzed using image processing algorithms. (e) Storage costs are minimized and images can also be accessed and interpreted remotely [12,13].

2.3.3 Acoustic Emission (AE):

Acoustic Emission is another technique with growing applications. It is commonly defined as generation of transient elastic waves within a material caused by the release of localized stress energy which can be caused by sources not involving material failure including friction, active corrosion, leakage, cavitation's and impact. Additionally, events can also come quite rapidly when materials begin to fail, in which case (AE) activity rates are studied as opposed to individual events. Commonly studied AE events in material failure processes include the extension of a fatigue crack or fiber breakage in composite materials. The monitoring of the level of AE activity during multiple load cycles forms the basis for many (AE) safety inspection methods that allow the parts undergoing inspection to remain in service. In materials under active stress, such as some components of an airplane during flight, transducers mounted in an area can detect the formation of a crack and its growth. A group of transducers can be used to record signals and then locate the precise area of crack by measuring the time for the sound to reach different transducers. The technique is also valuable for detecting cracks forming in pipelines transporting liquids under high pressures.

2.4 Materials And Defects - Physical And Mechanical Properties of Materials:

2.4.1 Metallic Materials:

Mechanical properties are defined as the properties of a material that reveal its elastic and inelastic (plastic) behavior when force is applied, thereby indicating its suitability for mechanical applications, for example, modulus of elasticity, tensile strength, elongation, hardness, and fatigue limit. Other mechanical properties, not mentioned specifically above, are yield strength, yield point,

impact strength, and reduction of area. In general, any property relating to the strength characteristics of metals is considered to be a mechanical property. Physical properties relate to the physics of a metal such as density, electrical properties, thermal properties, magnetic properties and the like. These and other properties will be described here in slightly more detail [13].

2.4.3 Elasticity And Plasticity:

When stress or force is applied to a metal, it changes shape. For example a metal under a compressive stress will shorten and metal in tension will lengthen. This change in shape is called strain. The ability of metal to strain under load and then return to its original size and shape when unloaded is called elasticity. The elastic limit (proportional limit) is the greatest load a material can withstand and still spring back into its original shape when the load is removed. Within the elastic range stress is proportional to strain and this is known as Hooke's law. The relationship between applied stress or load and the consequent strain or change in length is shown in Fig (2.11). The end of the straight line portion is known as the elastic limit. A point on the curve slightly higher than the elastic limit is known as the yield point or yield strength. The allowable or safe load for a metal in service should be well below the elastic limit. If higher loads are applied, however, the range of elasticity or elastic deformation is exceeded and the metal is now permanently deformed. Now it will not return to its original dimensions even when the load is removed. For this reason, the area of the stress strain curve beyond the elastic limit is called the plastic range. It is this property that makes metals so useful. When enough force is applied by rolling, pressing or hammer blows, metals can be formed, when hot or cold, into useful shapes. If the application of load is increased in the plastic region a stage comes when the material fractures [13].

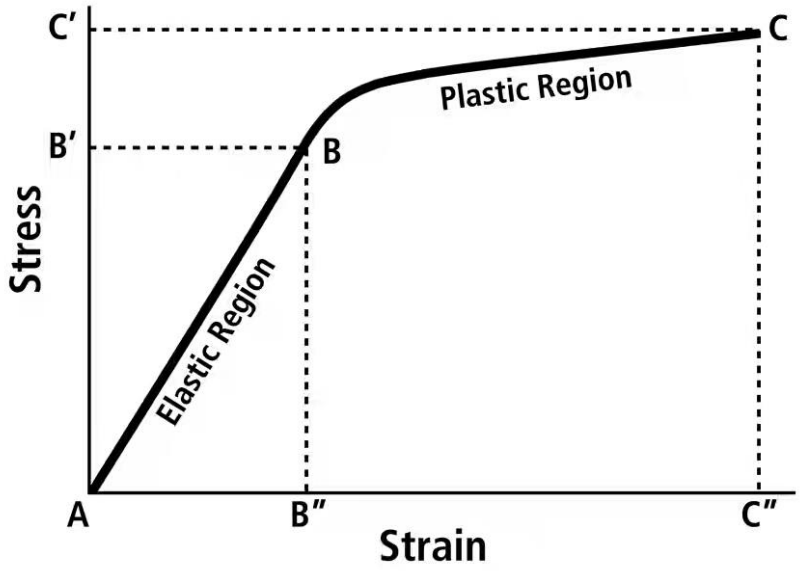

Fig: (2.8) Stress-strain curve showing elastic and plastic deformation and fracture [13].

A very important feature of the stress-strain curve must be pointed out. The straight-line or elastic part of the stress-strain curve of a given metal has a constant slope, which cannot be changed by changing the microstructure or heat treatment. This slope, called the modulus of elasticity, measures the stiffness of the metal in the elastic range. Changing the hardness or strength does not change the stiffness of the metal. The stiffness of any metal varies inversely with its temperature; that is, as temperature increases, stiffness decreases, and vice versa [13].

2.4.4 Strength:

The strength of a metal is its ability to resist change in shape or size when external forces are applied. There are three basic types of stresses namely tensile, compressive, and shear. When we consider strength, the type of stress to which the material will be subjected must be known. Steel has equal compressive and tensile strength, but cast iron has low tensile strength and high compressive strength. Shear strength is less than tensile strength in virtually all metals. The tensile strength of a material can be determined as: Tensile strength = (Maximum load) / (Original cross - sectional area) (1.1) A specimen of known dimensions is placed in the tensile testing machine and loaded slowly until it breaks. Instruments are sometimes used to make a continuous record of the load and the amount of strain (proportional change in length). This information is put

on a graph called a stress-strain diagram (see Fig. 2.8). A stress-strain diagram can be made for any metal.

2.4.5 Hardness:

The hardness of a metal is its ability to resist being permanently deformed. There are three ways that hardness is measured; resistance to penetration, elastic hardness, and resistance to abrasion. Hardness varies considerably from material to material. This variation can be illustrated by making an indentation in a soft metal such as aluminum and then in a hard metal such as alloy tool steel. Rockwell, Vicker and Brinell hardness testers are the most commonly used types of hardness testers for industrial and metallurgical purposes. Heat treaters, Inspectors, and many others in industry often use these machines. The Rockwell hardness test is made by applying two loads to a specimen and measuring the difference in depth of penetration in the specimen between the minor load and the major load. The Brinell hardness test is made by forcing a steel ball, usually 10 millimeters (mm) in diameter, into the test specimen by using a known load weight and measuring the diameter of the resulting impression. A small microscope is used to measure the diameter of the impressions [12,13]. Various loads are used for testing different materials, for example, 500kilograms (kg) for soft materials such as copper and aluminum and 3000 kg for steels and cast irons. Generally the harder the material is, the greater its tensile strength will be, that is, its ability to resist deformation and rupture, when a load is applied.

2.4.6 Ductility:

The property that allows a metal to deform permanently when loaded in tension is called ductility. Any metal that can be drawn into a wire is ductile. Steel, aluminum, gold, silver, and nickel are examples of ductile metals. The tensile test is used to measure ductility. Tensile specimens are measured for area and length between gauge marks before and after they are pulled. The per cent of elongation (increase in length) and the per cent of reduction in area (decrease of area at the narrowest point) are measures of ductility. A high per cent elongation (about 40%) and reduction in area (about 70 per cent) indicates a high ductility. A metal showing less than 20 per cent elongation is said to have low ductility.

2.4.7 Malleability:

The ability of a metal to deform permanently when loaded in compression is called malleability. Metals that can be hammered or rolled into sheets are

malleable. Most ductile metals are also malleable, but some very malleable metals such as lead are not very ductile and cannot be drawn into wire easily. Metals with low ductility, such as lead, can be extruded or pushed out of a die to form wire and other shapes.

2.4.8 Brittleness:

A material that will not deform plastically under load is said to be brittle. Excessive cold working causes brittleness and loss of ductility. Cast iron does not deform plastically under a breaking load and is therefore brittle. A very sharp "notch" that concentrates the load in a small area can also reduce plasticity. Notches are common causes of premature failure in parts. Weld undercut, sharp shoulders on machined shafts, and sharp angles on forgings and castings are examples of unwanted notches (stress raisers) [13].

2.4.9 Notch Toughness:

Notch toughness (impact strength) is the ability of a metal to resist rupture from impact loading when there is a notch or stress raiser present. A metal may show high ductility or strength when tensile tested or be hard or soft when hardness tested, but often the behavior of metals under shock loads is not seemingly related to those properties. Of course, as a rule, a brittle metal such as grey cast iron will fail under low shock loads; that is, its shock resistance is low, and soft wrought iron or mild steel has a high shock resistance. But soft, coarse grained metals will have lower shock resistance than fine-grained metals. A notch or groove in a part will lower the shock resistance of a metal, so a specific notch shape and dimension is machined on the test specimen in order to give uniform results. In general the tensile strength of a metal changes in proportion to hardness. However, this relationship does not always hold true at high hardness levels or with brittle materials because these materials are more sensitive to stress concentrations, or notches, and may fracture prematurely when stressed in tension.

2.4.10 Electrical Conductivity:

Electrical conductivity is a measure of the ability of a material to conduct electric current. It is the reciprocal of resistivity. Conductivity is commonly expressed as mhos per meter, since the unit of resistivity is the ohm. The conductivity of metallic elements varies inversely with absolute temperature over the normal range of temperatures but at temperatures approaching absolute

zero the imperfections and impurities in the lattice structure of a material make the relationship more complicated. Metals and materials exhibit a wide range of conductivity. Between the most conductive substances (silver and copper) and the most resistive (polystyrene for example) the difference amounts to 23 orders of magnitude [12].

2.5 Non-Metallic Materials:

2.5.1 Ceramic:

Ceramics offer unique properties as engineering materials, notably exceptionally high hardness and resistance to abrasion and corrosion as well as high temperature properties considerably superior to those of any metals. However, they are less ductile, intrinsically brittle and susceptible to thermal shock which can limit their maximum service temperature on applications involving thermal cycling. Resistance to thermal shock is directly dependent on a low coefficient of thermal expansion and high thermal conductivity, which properties differ appreciably between different ceramic materials. The fabrication of ceramics does not set particular problems since they can be formed by traditional techniques such as slip casting wet pressing and extrusion; and by such modern methods as injection moulding, iso-static pressing, tape casting and dry pressing. Ceramics which can be classified (or are usable or potentially usable) as engineering materials currently embrace: (i) alumina, (ii) beryllia (beryllium oxide) and boron nitride, (iii) porcelain(aluminum silicates), (iv) steatite and forsterite (magnesium silicates), (v) silicon nitride and silicon carbide, (vi) titanium diboride and (vii) vitreous carbon. Ceramics are finding an increasing use in the fabrication of electronic components, engineering components, medicine and dentistry and jewellery. The use of ceramic-coated metals and ceramic-metal combinations has now assumed significant proportions, particularly in the fields of practical nuclear physics (e.g. parts for nuclear reactors) and jet engine manufacture. Metal ceramic combinations are of two types: a ceramic coating on the metal, or a chemical and mechanical combination of metals and ceramics in a cermets material. Both are essentially attempts to produce satisfactory high temperature materials, either with reduced costs and better availability or with an overall performance superior to existing metal or ceramic materials on their own. Broadly speaking the mechanical properties of these two types of materials represent extremes. Metals have high tensile strength and shock resistance, but lose these properties rapidly with

increasing temperature[13]. Ceramics of the refractory kind have extremely high melting points and excellent general stability, but are low in tensile strength and both mechanical and thermal shock resistance. Normally cermets are formed by techniques similar to those employed in powder metallurgy. The ceramic content usually comprises refractory oxides, carbides or nitrides whilst the metal powder component is usually chromium, nickel, molybdenum or titanium. The resulting properties are different from those of either of the separate constituents. A number of cermets have particularly high melting points, best realized in an open flame.

2.5.2 Composites:

A composite is a material in which a stronger, sometimes fibrous material is usually combined with another to reinforce or strengthen the resultant mass. The needs of the aerospace industry led to the development and acceptance of composite materials. Low weight, high strength and great rigidity were of paramount interest of military aviation. The sesame qualities are also in demand in many non-military applications. The most common forms of composites are based on a plastic matrix. The fibrous reinforcing material may be in sheet form, as in thermo set plastic laminates; filament form, woven or random, as in glass reinforced plastics; or short fiber form as in filled or reinforced thermoplastics. These materials are well established and widely available. In the case of thermo set laminate composites, phenolic, melamine and epoxide are the main resin systems used with paper, cotton fabric, glass fabric and asbestos as the main alternative reinforcing materials. Ceramic and metal composites find several applications in engineering industry.

2.5.3 Concrete:

Concrete is a mixture of stone and sand held together by a hardened paste of hydraulic cement and water. When the ingredients are thoroughly mixed, they make a plastic mass which can be cast or moulded into a predetermined size and shape. When the cement paste hardens, the concrete becomes very hard like a rock. It has great durability and has the ability to carry high loads especially in compression. The required strength and properties of concrete can be obtained by careful selection of its ingredients, correct grading of ingredients, reinforcement, accurate water additions and adopting a good workmanship in mixing, transportation, placing, compaction, finishing, and curing of concrete in the construction work. The main ingredients of concrete are cement, coarse

aggregate (i.e. screenings, gravel, etc.), fine aggregate (i.e. sand), chemical admixtures (if necessary) and fibrous materials (as necessary). Aggregates in concrete constitute by far the bulk of the mass [13]].

2.5.4 Structures of Metals And Alloys:

The properties of metals can be explained in terms of the manner in which the atoms of metal are bonded together. In this bond, called the "metallic bond", which is formed among similar metal atoms, when some electrons in the valence shell separate from their atom and exist in a cloud surrounding all the positively charged atoms. These positively charged atoms arrange themselves in a very orderly pattern. The atoms are held together because of their mutual attraction for the negative electron cloud. Because the electrons are free to move in an electric field, metals conduct electricity. Because free electrons absorb and then radiate back most of the light energy that falls on them, metals are opaque and lustrous. Because free electrons can transfer thermal energy, metals conduct heat effectively. The metallic bond is non-specific, which explains why different metals can be alloyed or joined one to another. It is also non-directional, pulling equally hard in all directions. It therefore binds the metal atoms tightly, so that their cores (nuclei and inner shell electrons) fit closely among one another. The close packing favored by the metallic bond is best realized in certain regular crystalline structures. These structures, although resistant to tension, offer less resistance to shearing forces, and thus they explain the ductility of metals. They are by definition dense, and thus they explain the comparative heaviness of metals [13].

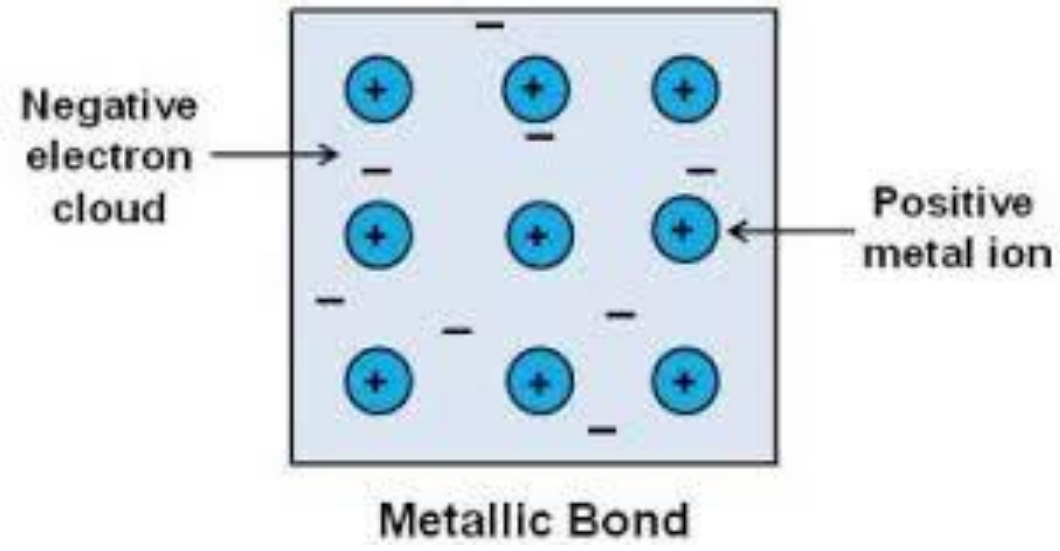

Fig: (2.9) Schematic illustration of a metallic bond [13].

Chapter Three

X-Rays Fluorescence

3.1 Introduction:

X-ray fluorescence (XRF) spectrometry is an elemental analysis technique with broad application in science and industry. (XRF) is based on the principle that individual atoms, when excited by an external energy source, emit X-ray photons of a characteristic energy or wavelength. By counting the number of photons of each energy emitted from a sample, the elements present may be identified and quantitated. Henry Moseley was perhaps the father of this technique, since he, building on W.C. Röentgen's discovery of the high-energy radiation dubbed X-rays, built an X-ray tube which he used to bombard samples with high-energy electrons. Moseley in 1912 discovered a mathematical relationship between the element's emitted X-ray frequency and its atomic number. In 1925 Coster and Nishina were the first to use primary X-rays instead of electrons to excite a sample. After Glocker and Schreiber were the first to perform quantitative analysis of materials using XRF in 1928, detector technology had to catch up in order to make the technique practical, which didn't begin to happen until the 1940's. The 1950's saw the first commercially produced X-ray spectrometers. In 1970, the lithium drifted silicon detector was developed, and this technology is still in use today. Modern XRF instruments are capable of analyzing solid, liquid, and thin-film samples for both major and trace (ppm-level) components. The analysis is rapid and usually sample preparation is minimal or not required at all. These instruments are used primarily for the provenance research on obsidian artifacts from around the world, but they are also used in special circumstances for the non-destructive analysis of other materials such as metals, ceramic paints, and soils.

3.2 The X-Ray Fluorescence:

Acronym by 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 [7].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. Fig (3.1) 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 [3].

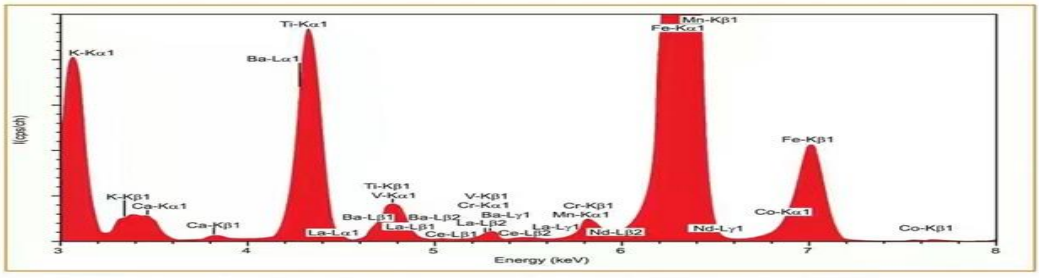

Fig: (3.1) Typical spectrum of a soil sample measured with an EDXRF spectrometer [7].

3.3 Basic of XRF:

In XRF, X-rays produced by a source irradiate the sample .In most cases, the a source irradiate the sample. In most cases, the source is an X-ray tube but alternatively it could be a synchrotron or a radioactive material. The elements present in the sample will emit fluorescent X-ray radiation with direct energies (equivalent to colors in optical light) that are characteristic for those elements. A

different energy is equivalent to different color. By measuring the energies (differencing the colors) of the radiation emitted by the sample is it possible to determine which element are present. This step it is called qualitative analysis. By measuring the intensities if emitted energies (colors) it is called quantitative analysis.

3.4 The X-Rays

X-ray can be seen as electromagnetic waves with their associated wavelengths or as beams of photons with associated energies .Both views are correct, but on or the other is easier to understand depending on the phenomena to explained other electromagnetic waves include light, radio waves and ɣ-rays .Figure 2 shows that X-rays have wavelengths and energy between ɣ-ray, and ultraviolet light. The wavelengths of X-rays are in the range from 0.01 to 10 nm, which corresponds to energies in the range from (0.125 to 125 Kev) .The wavelength of X-rays is inversely proportion to its energy, according to (E*λ=h c) .E is the energy in Kev and λ the wavelength in nm. The term hc is product of Planck's constant and the velocity of light and has, using Kev and nm as units. A constant values of 1.23985.

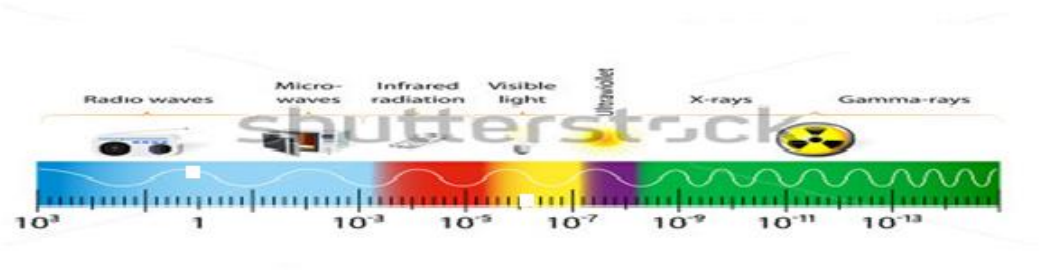

Fig: (3.2) X-rays and other electromagnetic radiation [7].

3.5 Interaction of X-Rays with Matter:

There are three main interactions when x-rays contact fluorescent Compton scatter and Rayleigh scatter (see figure 3.3). If a beam of X-rays photons is directed towards a slab of material a fraction will transmitted through, a fraction is absorbed (producing fluorescent radiation) and fraction is scattered back. Scattering can occur is known as Compton scatter and the second Rayleigh scatter. The fluorescence and the scatter depend on the thickness (d), density (p) and composition of the material, and on the energy of the X-rays. The next

sections will describe the production of fluorescence radiation and scatter. The fluorescence and the scatter depend on the thickness (d), density (ρ) and composition of the material, and on the energy of the X-rays. The next sections will describe the production of fluorescent radiation and scatter [13].

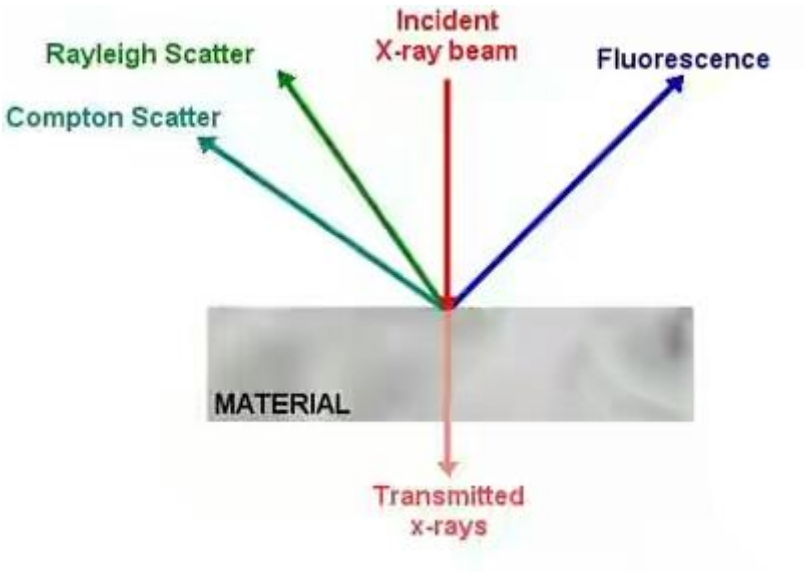

Fig: (3.3) Three main interactions of X-rays with matter [7].

3.6 Production of Characteristic Fluorescent Radiation:

The classical model of an atom is a nucleus with positively charged protons and non-charged neutrons, surrounded by electrons grouped in shells or orbital. The innermost shell is called the K-shell, followed by L-shells, M-shells etc. as one moves outwards. The L-shell has 3 sub-shells called. The K-shell can contain 2 electrons, the L-shell 8 and the M-shell 18. The energy of an electron depends on the shell it occupies, and on the element to which it belongs. When irradiating an atom, particles such as X-ray photons and electrons with sufficient energy can expel an electron from the atom (Fig 3.4) [9].

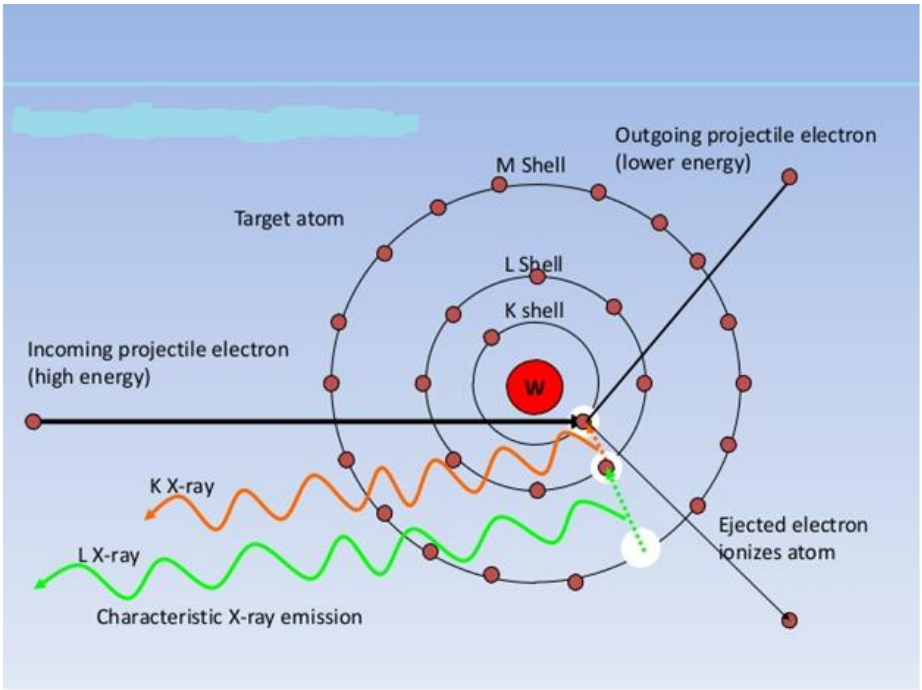

Fig: (3.4) Production of characteristic radiation [7].

This produces a 'hole' in a shell, in the example (Fig.3. 4) a hole in the K-shell, putting the atom in an unstable excited state with a higher energy. The 'hole' in the shell is also called the initial vacancy. The atom wants to restore the original configuration, and this is done by transferring an electron from an outer shell such as the L-shell to the hole in the K-shell. An L-shell electron has a higher energy than a K-shell electron, and when an L-shell electron is transferred to the K-shell, the energy surplus can be emitted as an X-ray photon. In a spectrum, this is seen as a line. The energy of the emitted X-rays depends on the difference in energy between the shell with the initial hole and the energy of the electron that fills the hole (in the example, the difference between the energy of the K and the L shell). Each atom has its specific energy levels, so the emitted radiation is characteristic of that atom. An atom emits more than a single energy (or line) because different holes can be produced and different electrons can fill these. The collection of emitted lines is characteristic of the element and can be considered a finger print of the element. To expel an electron from an atom, the X-rays must have a higher energy level than the binding energy of the electron. If an electron is expelled, the incoming radiation is absorbed, and the higher the absorption the higher the fluorescence. If, on the other hand, the energy is too high, many photons will 'pass' the atom and only a few electrons will be removed. Figure 5 shows that high energies are hardly absorbed and produce low fluorescence. If the energy of the incident photons is lower and comes closer to the binding energy of the K-shell electrons, more and more of the

radiation is absorbed. The highest yield is reached when the energy of the photon is just above the binding energy of the electron to be expelled. If the energy becomes lower than the binding energy, a jump or edge can be seen: the energy is too low to expel electrons from the corresponding shell, but is too high to expel electrons from the lower energetic shells. The figures show the K-edge corresponding to the K-shell, and three L-edges corresponding with the L Not all initial vacancies created by the incoming radiation produce fluorescent photons. Emission of an Auger electron is another process that can take place [5]. The fluorescent yield is the ratio of the emitted fluorescent photons and the number of initial vacancies. Figure 6 shows the fluorescence yield for K- and L-lines as function of the atomic number Z. The figure clearly shows that the yield is low for the very light elements, explaining why it is so difficult to measure these elements [7].

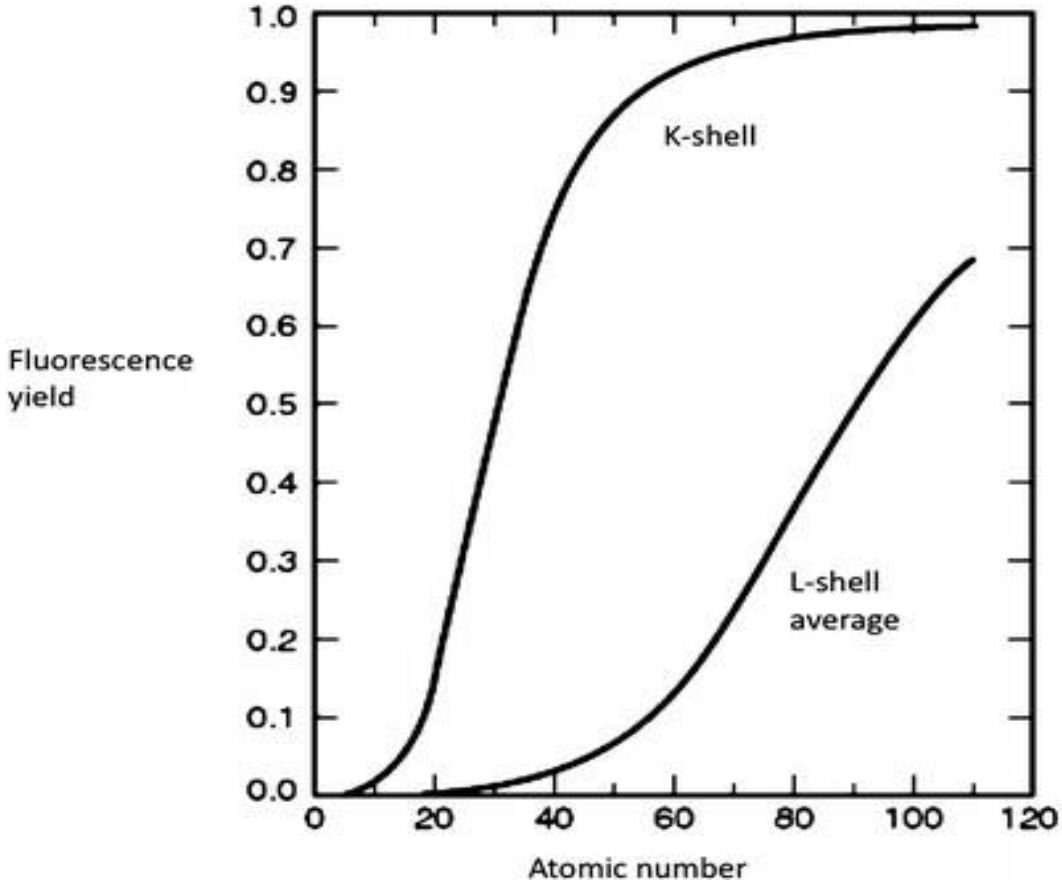

Fig: (3.5) Fluorescence yield for K and L electrons [7].

There are several ways to indicate different lines. The Siegbahn and IUPAC notations are the two most often found in the literature. The Siegbahn notation indicates a line by the symbol of an element followed by the name of the shell where the initial hole is plus a Greek letter (α,β,γ etc.) indicating the relative intensity of the line. For example, Fe K α is the strongest iron line due to an expelled K electron [5]. The Siegbahn notation however does indicate which shell the electron comes from that fills the hole. In the IUPAC notation, a line is indicated by the element and the shell where the initial hole was, followed by the shell where the electron comes from that fills this hole. For example, Cr KLIII is chromium radiation due to a hole produced the K –shell filled by an electron in the L –shell. Generally, K-lines are more intense than L-lines, which are more intense than M-lines, and so on. Quantum mechanics teaches that not all transitions are possible, for instance a transition from the L- to the K-shell. Figure 7 gives an overview of the most important lines with their transitions in Siegbahn notation. K-lines L-lines.

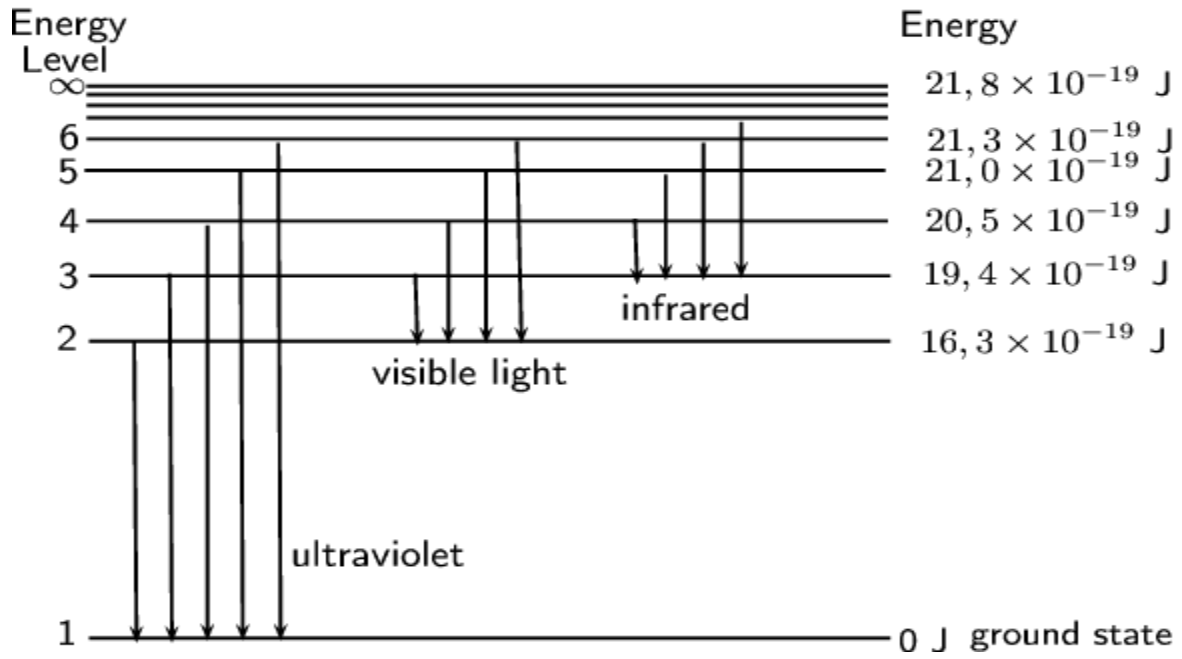

Fig: (3.6) Major lines and their transitions [10].

3.7 Absorption And Enhancement Effects:

To reach the atoms inside the sample, the X-rays have to pass through the layer above it, and this layer will absorb a part of the incoming radiation. The characteristic radiation produced also has to pass through this layer to leave the sample, and again part of the radiation will be absorbed.

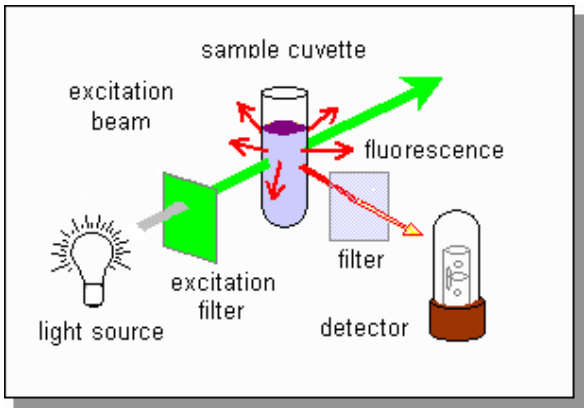

Fig: (3.7) Absorption of incoming and fluorescent X-rays [7].

The magnitude of the absorption depends on the energy of the radiation, the path length d of the atoms that have to be passed, and the density of the sample. The absorption increases as the path length, density and atomic number of the elements in the layer increase, and as the energy of the radiation decreases. The absorption can be so high that elements deep in the sample are not reached by the incoming radiation or the characteristic radiation can no longer leave the sample. This means that only elements close to the surface will be measured.
The incoming radiation is made up of X-rays, and the characteristic radiation emitted by the atoms in the sample itself is also X-rays. These fluorescent X-rays are sometimes able to expel electrons from other elements in the sample. This, as with the X-rays coming from the source, results in fluorescent radiation. The characteristic radiation produced directly by the X-rays coming from the source is called primary fluorescence, while that produced in the sample by primary fluorescence of other atoms is called secondary fluorescence [3].

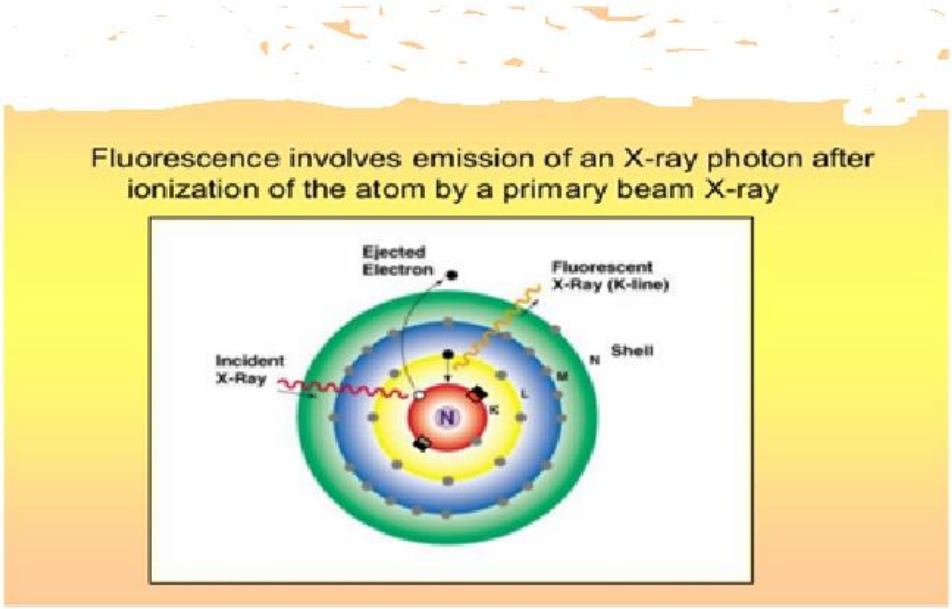

Fig: (3.8) Primary and secondary fluorescence [7].

A spectrometer will measure the sum of the primary and secondary fluorescence, and it is impossible to distinguish between the two contributions. The contribution of secondary fluorescence to the characteristic radiation can be significant (of the order of 20%). Similarly, tertiary and even higher order radiation can occur. In almost all practical situations these are negligible, but in very specific cases can reach values of 3%.

3.8 Absorption And Analysis Depths:

As the sample gets thicker and thicker, more and more radiation is absorbed. Eventually radiation produced in the deeper layers of the sample is no longer able to leave the sample. When this limit is reached depends on the material and on the energy of the radiation. When a sample is measured, only the atoms within the analysis depth are analyzed. If samples and standards with various thicknesses are analyzed, the thickness has to be taken in to account [5].

3.9 Rayleigh And Compton Scatter:

A part of the incoming X-rays is scattered (reflected) by the sample instead of producing characteristic radiation. Scatter happens when a photon hits an electron and bounces away. The photon loses a fraction of its energy, which is taken in by the electron as shown in Fig (3.9). It can be compared with one billiard ball colliding with another. After the collision, the first ball loses a part

of its energy to the ball that was hit. The fraction that is lost depends on the angle at which the electron (ball) was hit. This type of scatter is called Compton or incoherent scatter.

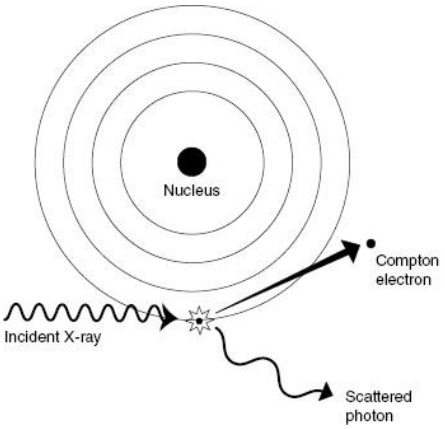

Fig: (3.9) Compton Scatter [10].

Another phenomenon is Rayleigh scatter. This happens when photons collide with strongly bound electrons. The electrons stay in their shell but start oscillating at the frequency of the incoming radiation. Due to this oscillation, the electrons emit radiation at the same frequency (energy) as the incoming radiation. This gives the impression that the incoming radiation is reflected (scattered) by the atom. This type of scatter is called Rayleigh or coherent scatter [7].

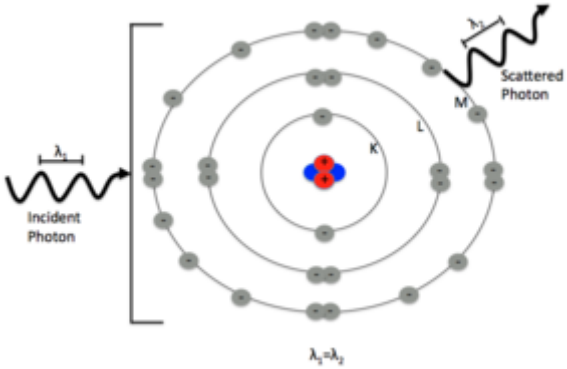

Fig: (3.10) Rayleigh scatter [7].

Samples with light elements give rise to high Compton scatter and low Rayleigh scatter because they have many loosely bound electrons. When the elements get heavier the scatter reduces. For the heavy elements, the Compton

scatter disappears completely, and only Rayleigh scatter remains. Fig (3.11) shows the Compton and Rayleigh scatters for lead (a heavy element) and for Perspex (light elements). The spread of energy in the Compton scatter is larger than for Rayleigh scatter; in a spectrum this can be observed by the Compton peak being wider than the Rayleigh peak [7].

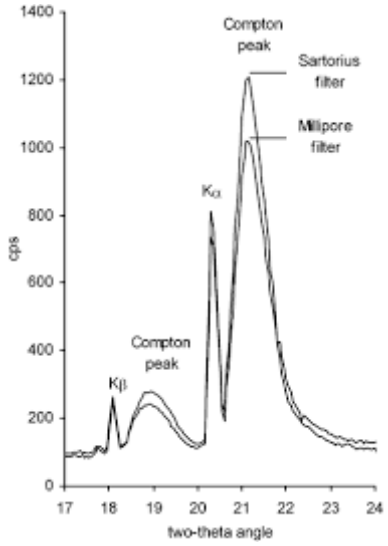

Fig: (3.11) Rayleigh scatter for light and heavy element [7].

3.10 Polarization:

X-rays are electromagnetic waves with electric and magnetic components E and B. This discussion is limited to the electrical component E but also holds for the magnetic component B. The amplitude of the electromagnetic waves corresponds to the intensity of the X-rays. Electromagnetic waves are transversal waves, which means that the electrical component is perpendicular to the propagation direction. This is similar to waves in water. If a stone is thrown into water, the waves are vertical but the propagation direction is horizontal. X-rays are said to be linear polarized if the electrical components are all in one plane as shown in Fig (3.12). If the electrical component has no preferred direction then the waves are called non-polarized.

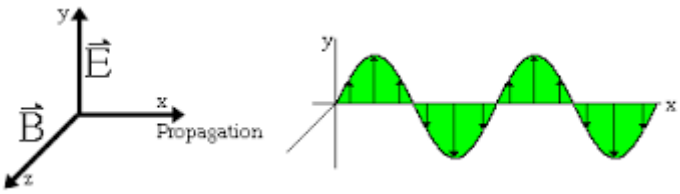

Fig: (3.12) X-rays polarized in vertical direction [7].

An electrical component E pointing in any direction can always be resolved into two perpendicular directions. Fig (3.12) shows how a component is resolved into a vertical and a horizontal direction. If non-polarized X-rays are reflected (scattered) by a specimen through 90°, the reflected X-rays will be polarized in one direction. That the vertical electrical component is not reflected because this would point in the new propagation direction. What remains after one reflection is the horizontal component alone, and the scattered X-rays are polarized horizontally. Perpendicular to the previous direction. In the second reflection the horizontal component is not reflected because it would point in the new propagation direction. Nothing is left from the incoming radiation after two perpendicular reflections. This feature is used in EDXRF spectrometers to eliminate the background profile from a spectrum [7].

3.11 XRF Analysis:

A good analysis starts with a well-prepared sample and a good measurement. This section describes how different sample types are prepared, and how they are measured accurately. After a sample is measured, it is analyzed. This is one in two steps: Qualitative analysis followed by quantitative analysis. Qualitative analysis determines which elements are present and their net intensities from the measured spectra. In many routine situations, the elements in the sample are known and only the net intensities need to be determined. The net intensities are used in the quantitative analysis to calculate the concentrations of the elements present. (EDXRF) and (WDXRF) often use slightly different methods for qualitative analysis. In (EDXRF the area of a peak gives the intensity while in (WDXRF) the height of the peak gives the intensity. Both methods would work for (EDXRF) and (WDXRF, but both have their specific advantages and disadvantages.

3.12 Sample Preparation:

Often, only a small sample of material is analyzed, for instance in a steel plant a small disk represents the full furnace contents. The sample must be representative of the entire material, and so must be taken very carefully. Once

taken, it must also be handled carefully. The sensitivity of modern spectrometers is so high that they even detect fingerprints, which can disturb the analysis. Another basic requirement is that a sample must be homogeneous. Spectrometers only analyze the sample's surface layer, so it must be representative of the whole sample. Most spectrometers are designed to measure samples that are circular disks with a radius between 5 and 50 mm. The sample is placed in a cup, and the cup is placed in the spectrometer. Special supporting films allow the measurement of loose powders and liquids. Different sample types are discussed below.

3.13 Powders:

Powders can be placed on a supporting film and measured directly. Technique is to press them under very high pressures (20,000 kg) into a tablet [7,3]. A binding material is sometimes added to improve the quality of the tablet. The tablet is then measured and analyzed. If a binding material is used, this has to be taken into account in the analysis because it does not belong to the initial sample. Care should be taken that the sample is homogeneous.

3.14 Liquids:

Liquids are poured into special cups with supporting films. Diluents are sometimes added to obtain sufficient liquid. Liquids cannot be measured in vacuum because they would evaporate; measuring in air is possible, but the air absorbs much of the radiation and makes it impossible to measure light elements. The spectrometer chamber is therefore filled with He gas - liquids will not evaporate and hardly any radiation is absorbed [3].

Chapter Four

Experiment

4.1 Introduction:

In this study experiment carried out using different types of milk (powder + liquid)from different companies which expert milk and local companies using X-ray Fluorescence Techniques in order to detect the elements that added in these milk the results reveals the following data as prescribed in the tables below.

4.2 Apparatus of experiment:

Used X5000 XRF mobile device for analysis contamination elements in powder and liquid milk weight 75 g for any samples.

4.3 Methodology:

The quality research is practical. We conducted a laboratory analysis of 6 samples of powdered dry milk and 2 sample of liquid milk using the XRF device and we obtained different chemical elements and the theoretical side we dealt with non-destructive testing and metal materials, XRF and Compton and Rayleigh scatter. I put samples one by one on pot and press the device from top and get readings on screen.

4.4 Results:

From the results; it was found that, the orders of heavy metal and trace elements content in the brand dry powder and liquid milk were , W>Mo>Fe>V>Nb>Ni>Cr>Ti>Mn>Co>Cu, respected among the 14 elements, W only showed to be exceed the highest percentage, and Nb is radioactive element in the CAPO liquid milk.

Table: (4.1) Powdered milk from Alwaddi:

Element	%	STD
Cr	0.05	0.000
Ni	0.01	0.001
Pb	0.00	0.001
Fe	0.02	0.008
Zn	0.00	0.001

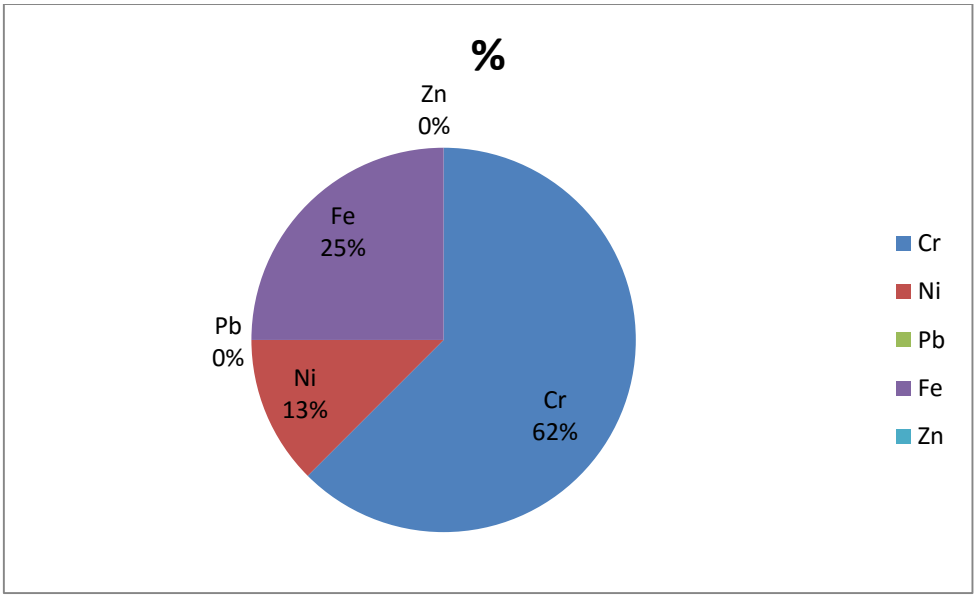

Figure: (4.1) powdered milk from Alwaddi:

Table: (4.2) Powdered milk from Delta:

Element	%	STD
Fe	0.04	0.007
Ni	0.01	0.002
Cr	0.03	0.006
Zn	0.00	0.002

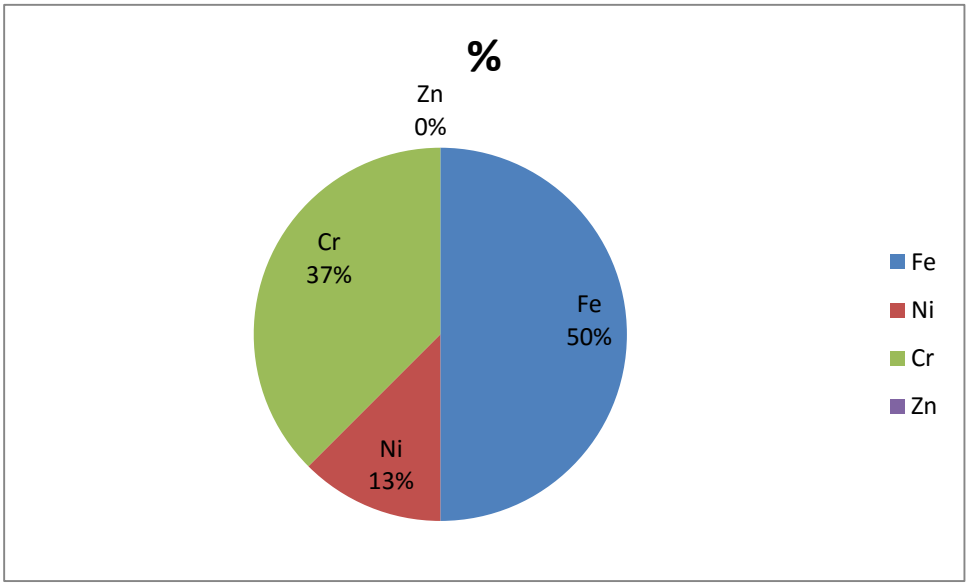

Figure: (4.2) powdered milk from Delta:

Table: (4.3) Powdered milk from CAPO:

Element	%	STD
Mo	27.57	2.304
Fe	19.86	4.389
Ni	2.23	3.151
Mn	0.98	0.035
W	23.95	1.108
Cr	4.54	0.249
V	17.77	0.153
Co	0.14	0.103

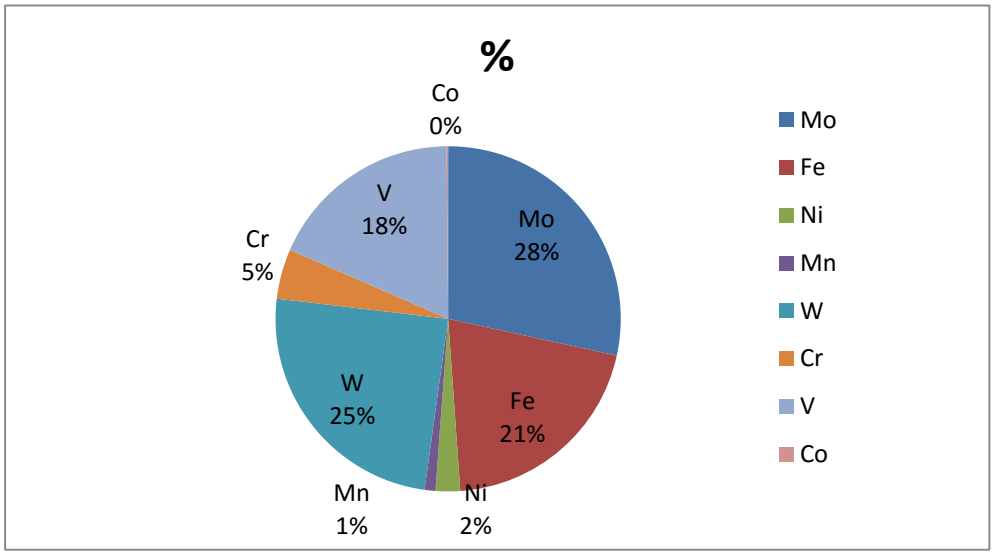

Figure: (4.3) powdered milk from CAPO:

Table: (4.4) Powdered milk from Junion:

Element	%	STD
Mo	26.80	1.883
Fe	21.44	2.127
Ni	4.01	2.421
Co	0.87	1.237
W	24.35	0.517
Cr	4.79	0.104
Mn	1.26	1.052
V	0.79	0.177

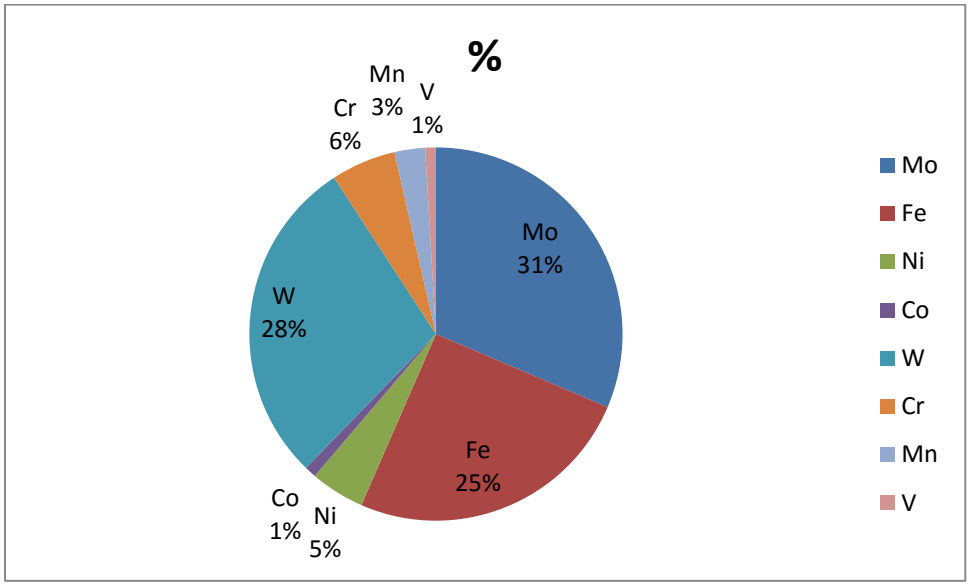

Figure: (4.4) powdered milk from Junion:

Table: (4.5) Powdered milk from Neel Cow:

Element	%	STD
Mo	24.46	0.313
Fe	21.58	2.698
Ni	3.29	0.664
Mn	1.34	0.055
W	24.23	0.413
Cr	4.28	0.660
V	2.10	0.481
Co	1.32	0.039

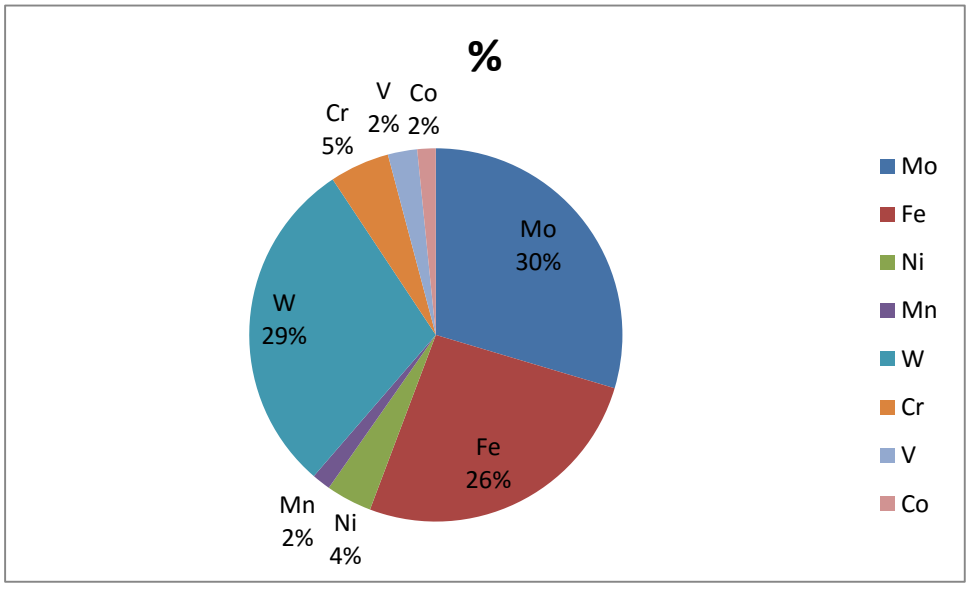

Figure: (4.5) powdered milk from Neel Cow:

Table: (4.6) powdered milk from Milko:

Element	%	STD
Fe	0.05	0.018
Ni	0.01	0.001
Mn	0.00	0.000
Cr	0.03	0.005
Zn	0.00	0.001
Pb	0.00	0.001

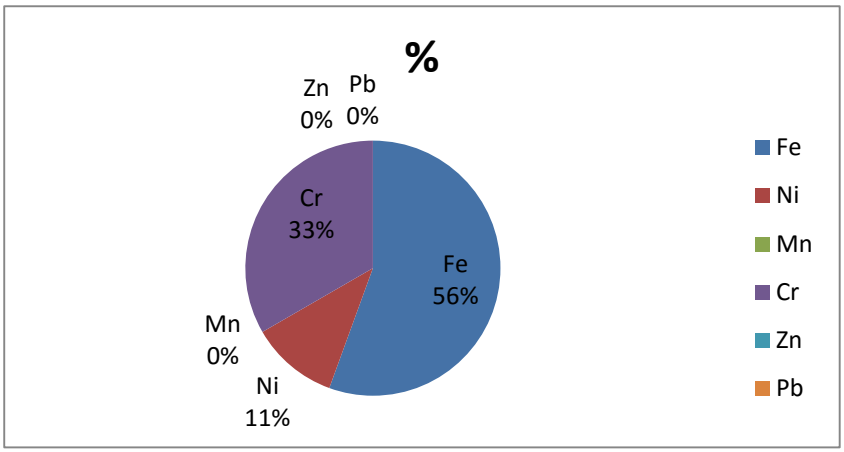

Figure: (4.6) powdered milk from Milko:

Table: (4.7) liquid milk from local market:

Element	%	STD
Fe	0.02	0.006
Ni	0.01	0.000
Cr	0.02	0.000

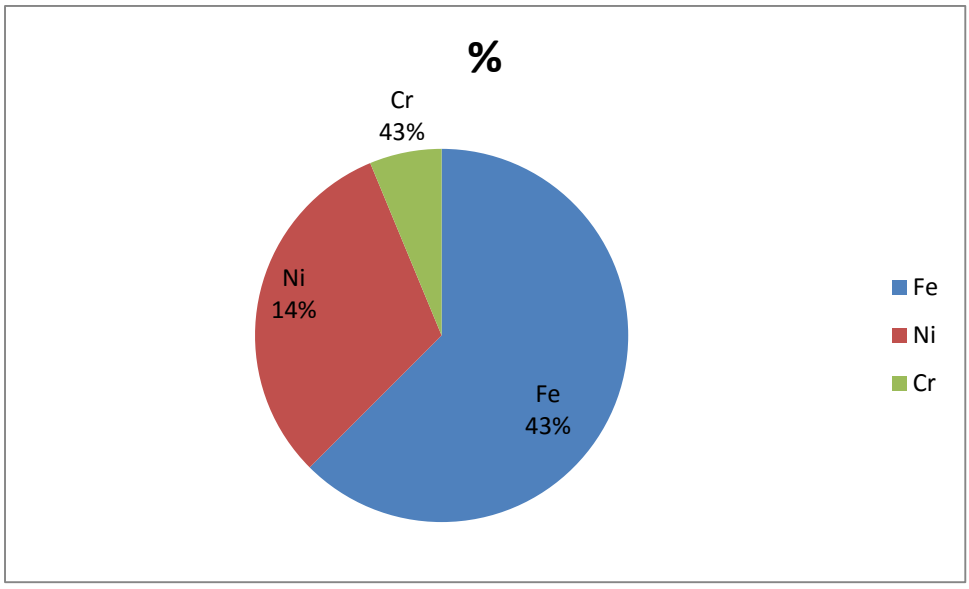

Figure: (4.7) Liquid milk from local market:

Table: (4.8) Liquid milk from CAPO:

Element	%	STD
W	75.69	1.971
Ni	13.80	0.184
Ti	2.08	0.580
Ta	0.93	1.311
Mn	0.62	0.278
Cu	0.12	0.116
Mo	17.44	0.285
Nb	13.72	0.0.169
Fe	2.02	0.167
Cr	0.76	0.890
Co	0.40	0.145

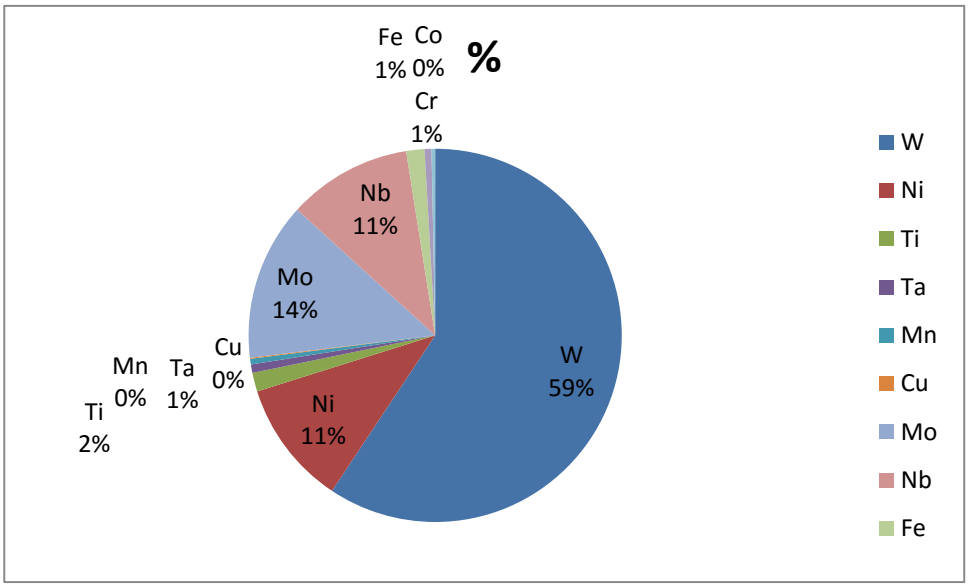

Figure: (4.8) Liquid milk from CAPO:

4.5 Discussion:

In this research six different companies which were produces powder milk and one pure milk as liquid was examined under XRF testing in order to determine the elements dissolved in this to types. In liquid milk found that there were three different elements Cr, Ni, and Fe. While in powder milk found more elements than that exist in liquid one concerning to the company. In CAPO powder milk there was 8 elements, while in liquid case there was eleven elements .In Delta there are four elements .In Alwaddi there was five elements. In Junion there was eight elements. In Neel cow there are eight element. In milko there are six elements. It is was concluded that the main common elements exist in the different types of powder milk for different companies was Cr, Ni, Fe and Mo. In all products for different companies noticed that there is absence of two main elements in the powder milk Ca and K. Nb which exist in CAPO liquid milk was founded to 13.72 which is exceeding the normal amount and W was very higher rate.

4.6 Conclusion:

Although, the metal content in sampled powdered and cow milks were within the safe limit, the potential human health risks cannot be neglected for the regular/long time consumption of contamination elements contained powdered and cow milk.

4.7 Recommendation:

I recommend for more studies to compare between domestic milk from different areas and powder milk. It is also preferable to collect domestic milk from farms directly.

4.8 References

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