Chapter one

1.1 Introduction

Most of the inorganic chemicals in drinking water are naturally occurring. They are acquired by the contact of water with rocks and soil and the effects of the geological setting, including climate [1-4]. However, the chemical composition of drinking water also depends on the contaminating effects of industry, human settlements, agricultural activities and water treatment and distribution [1-4]. Depending on water quality at the source, filtration, coagulation, and addition of chemicals to adjust pH and/or control corrosion treatments are employed [1-5]. In addition, chlorination or iodination may be used for disinfection and fluoridation for the prevention of dental caries [6-8]. Leaching of minerals from metal components used in water treatment plants and plumbing materials occurs when pH and hardness of water are not adjusted. Some of the main sources of dissolved metals include: for Cu- copper or brass plumbing system; Fe- cast iron, steel, and galvanized plumbing system; Zn- zinc galvanized pipes; Ni-chromium-nickel stainless plumbing system; Pb- derived from tin-lead or lead solder; and for Cd- as an impurity in zinc galvanized pipes or cadmium containing solders [1-4,9]. Recently, fortification of drinking water has been used in the prevention of iron deficiency in children [10] and to provide iodine in select populations [11].

In this thesis gives a general introduction to X-Ray fluorescence (XRF) spectrometry and XRF analysis in order to investigate elements that exist in different sources of water. It explains simply how a spectrometer works and how XRF analysis is done. It is intended for people new to the field of XRF analysis. Difficult mathematical equations are avoided and the thesis requires only a basic knowledge of mathematics and physics.

The thesis is not dedicated to one specific type of spectrometer or one Application area, but aims to give a broad overview of the main spectrometer types and applications.

1.2 The problem:

Water in our body has a very important role and an excellent water circulating system is established in our digestive tract. The human body consists of around 65% water and if we lose 3% of it, we will have symptoms of dehydration. If we lose 10~15% of it, we lose sound body conditions. Usually adults drink around 2~2.5 liters of water every day, of water every day, but in our body approximately 6~7 liters of water is circulated in our digestive tract to digest and absorb the nutrients of foods. In the lower parts of the large intestine, there exist intestinal microorganisms which produce short chain fatty acids, of which butyric acid is attributed to be an energy source to absorb water into the large intestine, to give the feces an adequate water content of 70~80%. Water Is the most important constituent of our body and a splendid water recycling system is established.

The availability of clean, fresh water is one of the most important issues facing humanity today – and will be increasingly critical for the future, as growing demand outstrips supplies and pollution continues to contaminate rivers, lakes and streams.

1.3 The aim

To determine the elements those exist in different sources water before and after applying magnetic field.

1.4 The methodology

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 themetal, cement, oil, polymer, plastic and food industries, along with mining, mineralogy and geology, and environmental analysis of water and were tematerials. XRF is also a very useful analysis technique for research and pharmacy.

1.5 Thesis layout

Chapter one is the introduction. Chapter two nondestructive testing. Chapter three briefly explains XRF and its benefits. Chapter four the material T

Chapter two

Basic principles of non-destructive testing (NDT)

2.1 Definition and importance of NDT.

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, labour 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 [21].

2.2 Types of 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 penetrant 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. The basic principles, typical applications, advantages and limitations of the conventional methods will now be briefly described [21].

2.2.1 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 eyesight 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. Figure (2.1) shows a portable light, a mirror on stem, a 2X or 4X hand lens, one illuminated magnifier with magnification 5X or 10X. For internal inspection, light lens systems such as borescopes allow remote surfaces to be examined. More sophisticated devices of this nature using fibre 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 [21].
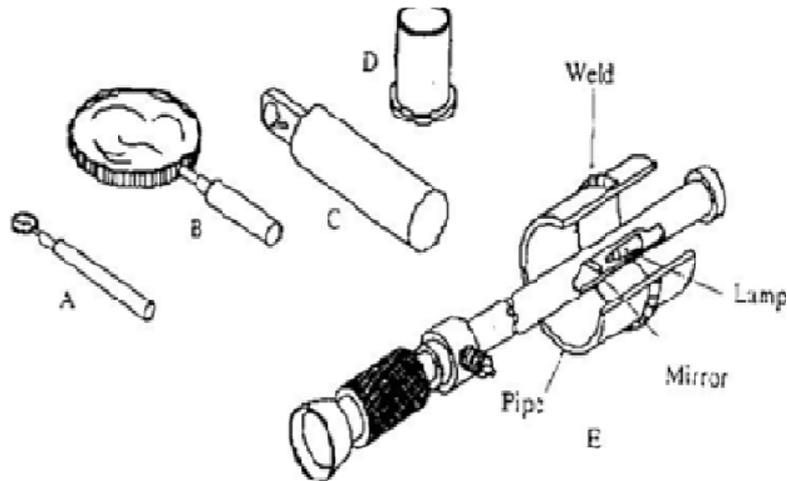
FIG. (2.1) various optical aids used in visual inspection:

(A) Mirror

(B) Hand magnifying glass (magnification usually 2–3X).

(C) Illuminated magnifier; field of view more restricted than D (magnification 5–10X)

(D) Inspection glass (magnification 5–10X)

(E) Bore scope or intrascope with built-in illumination (magnification 2–3X) .

2.2.2.1 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.

2.2.1.2 Some of the advantages of visual testing are as follows

(a) Testing is simple

(b) Testing speed is high

(c) Cost is low

(d) Less training

(e) On-line testing possibility

(f) Permanent record available

2.2.1.3 Some of the limitations of visual testing are as follows

• Can detect only surface defects

• Difficulty in sizing depth of defects [21].

2.2.2 Liquid penetrant testing

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. This method is widely used for testing of non-magnetic materials. In this method, a liquid penetrant is applied to the surface of the product for a certain predetermined time, after which the excess penetrant is removed from the surface. The surface is then dried and a developer is applied to it. The penetrant 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.2.2 Penetrant 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 penetrant is made under ultraviolet (or black) light under darkened conditions. Liquid penetrant processes are further sub-divided according to the method of washing of the component. Penetrants can be:

 (i) water-washable,

 (ii) post-emulsifiable, i.e. an emulsifier is added to the excess penetrant on surface of the component to make it water-+3

washable, and

(iii) Solvent removable, i.e. the excess penetrant needs to be dissolved in a solvent to remove it from the component surface [21].

In order of decreasing sensitivity and decreasing cost, the liquid penetrant processes can be listed as follows:

(a) Post emulsifiable fluorescent dye penetrant.

(b) Solvent removable fluorescent dye penetrant.

(c) Water washable fluorescent dye penetrant.

(d) Post emulsifiable visible dye penetrant.

(e) Solvent removable visible dye penetrant.

(f) Water washable visible dye penetrant.

2.2.2.1 The advantages of liquid penetrant testing

(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 [21].

2.2.2.2 The limitations of liquid penetrant testing:

(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 Zuse on very rough surfaces.
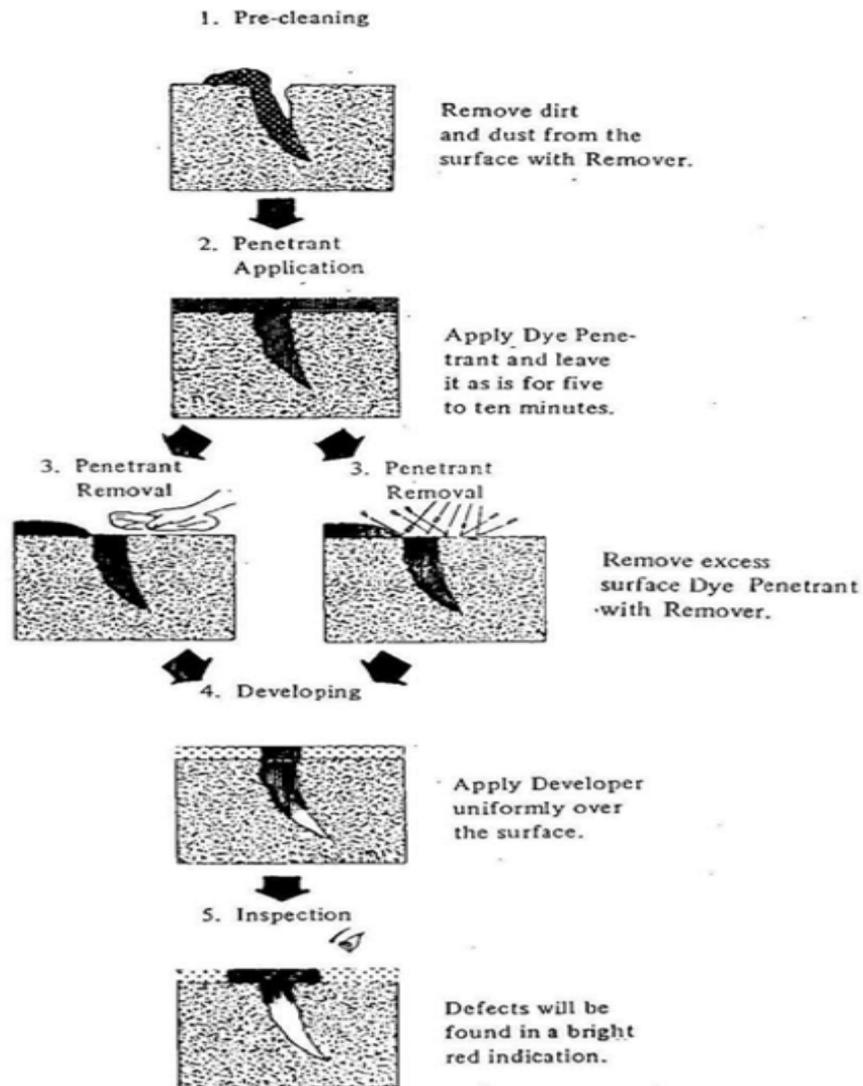
(d) No permanent record [21].

FIG. 2.2 Different stages of liquid penetrant testing

2.2.3. 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. 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 [21].
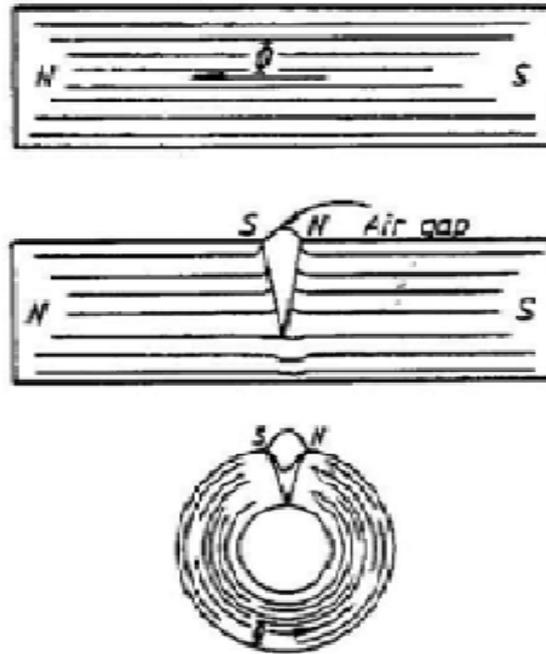

Figure. (2.3) Basic principle of magnetic particle testing

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.

(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 [21].

2.2.3.2 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.

2.2.3.3 The limitations of magnetic particle testing

It was 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.

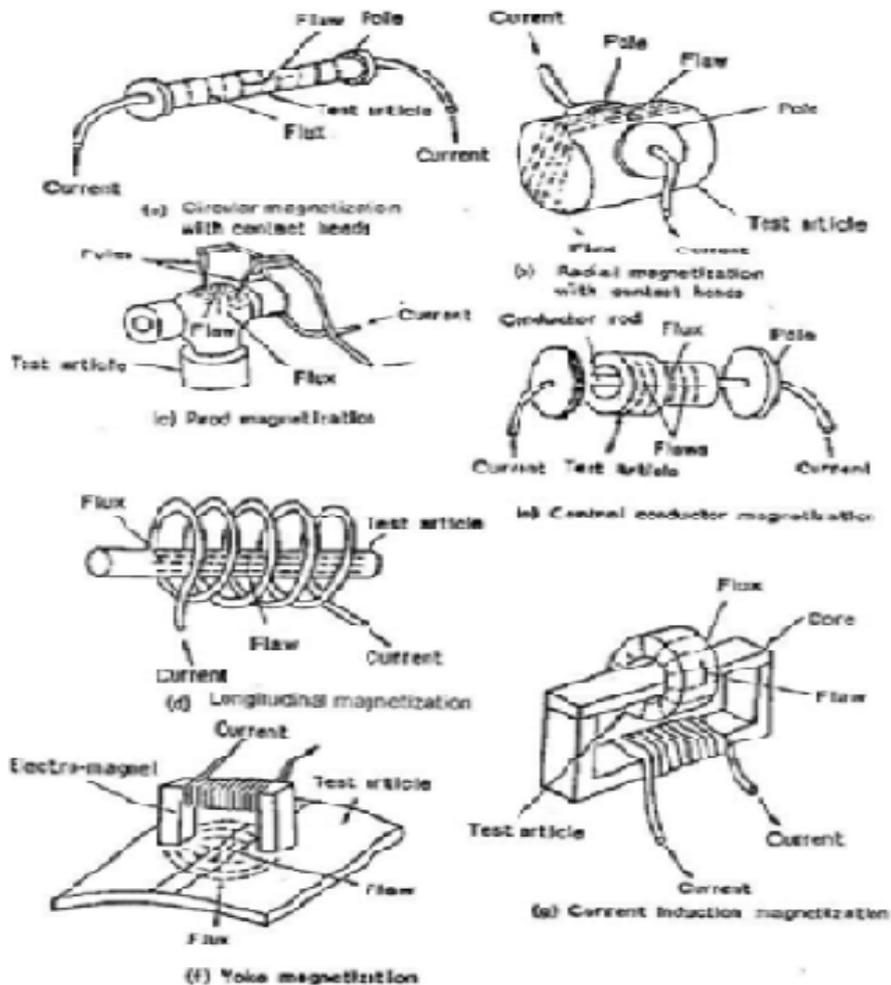(d) After testing the object must be demagnetized, which may be difficult sometimes [21].

Figure. 2.4 Different magnetizations used in magnetic particle testing.

2.2.4 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 case hardening 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.

There are three types of probes 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 [21].

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.
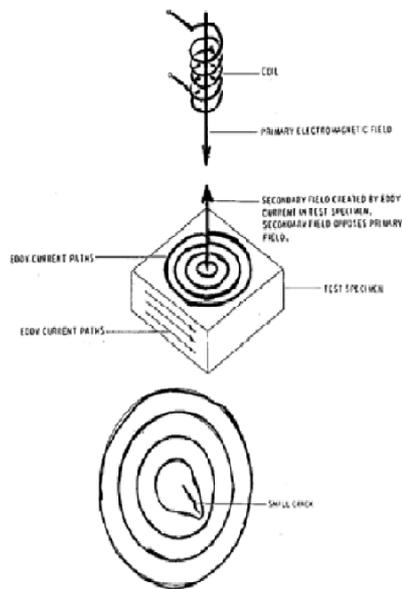
(e) Non contact testing of hot defects.

FIG. 2.5 (a) Generation of eddy currents in the test object.

(b) Distortion of eddy currents due to defect.

2.2.4.1 The advantages of eddy current testing

it was 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.

2.2.4.2 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.
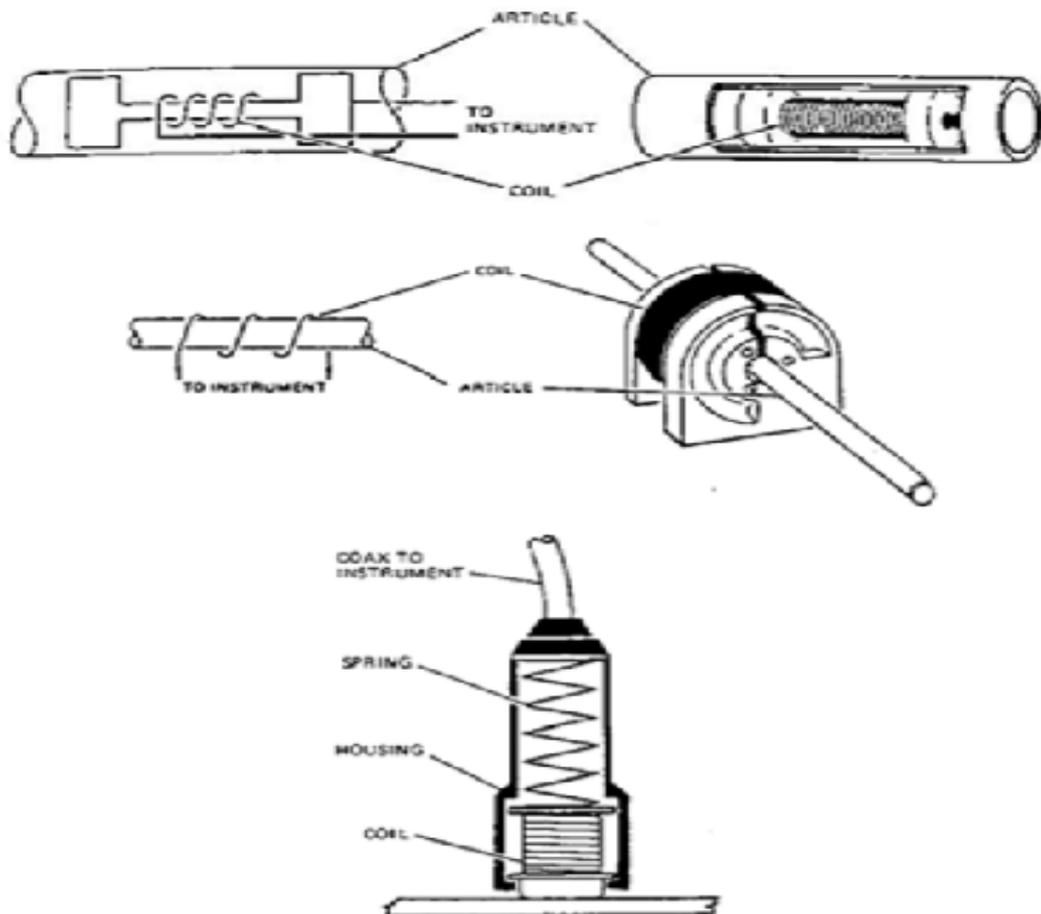
(d) Its depth of penetration is limited to 8 mm.

FIG. 2.6 Types of probes used in eddy current testing

(a) Internal Coil   (b) Encircling Coil   (c) Surface Probe.

2.2.5. Radiographic testing method (RT)

The radiographic testing method is used for the detection of internal flaws in many different materials and configurations. An appropriate radiographic film is placed behind the test object (Fig. 2.2.5.1) and is exposed by passing X- rays or gamma rays (Co-60 & Ir-192 radioisotopes) through it. The intensity of the rays while passing through the object is modified according to its internal structure and thus the exposed film, after processing, reveals a shadow picture known as a radiograph. It is then interpreted to obtain data about the present defects. This method is used on a wide variety of objects such as forgings, castings and weldments [21].

2.2.5.1 The advantages of radiographic testing include that:

(a) It is useful on wide variety of materials.

(b) It can be used for checking internal malstructure, misassembly or misalignment.

(c) It provides permanent record.

(d) Devices for checking the quality of radiograph are available. Some of the limitations are that:

(a) Access to both sides of the object is required.

(b) It cannot detect planar defects readily.

(c) The thickness range that can be inspected is limited.

(d) Sensitivity of inspection decreases with thickness of the test object.

(e) Considerable skill is required for interpretation of the radiographs.

(f) The depth of defect is not indicated readily.

(g) X rays and gamma rays are hazardous to human health. The iaea's radiation safety series are referred for personal safety and radiation protection.
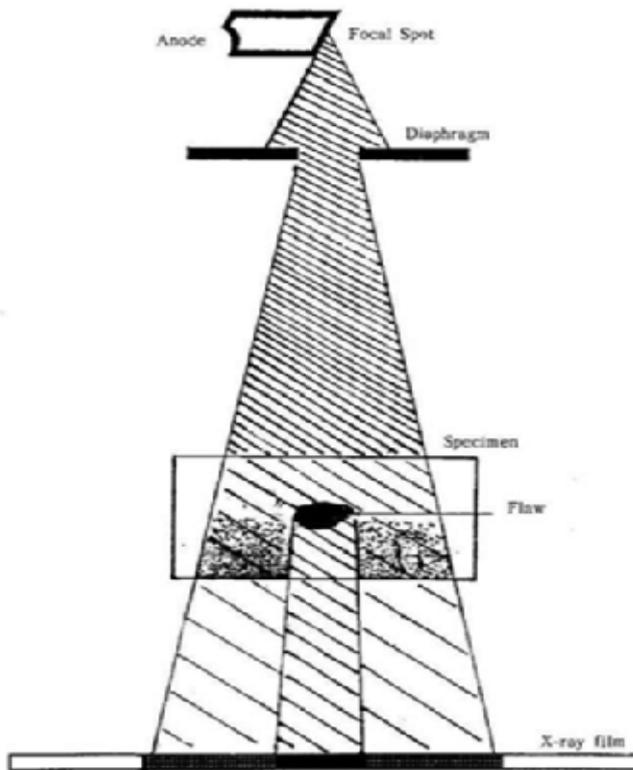
Fig. 2.7 Arrangement for radiographic testing method.

2.2.6 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 metalliquid or metal-solid interfaces. 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 [21].

2.2.6.1 The application of ultrasonic testing method:

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.

2.2.6.2 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.

2.2.6.3 The limitations of the 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
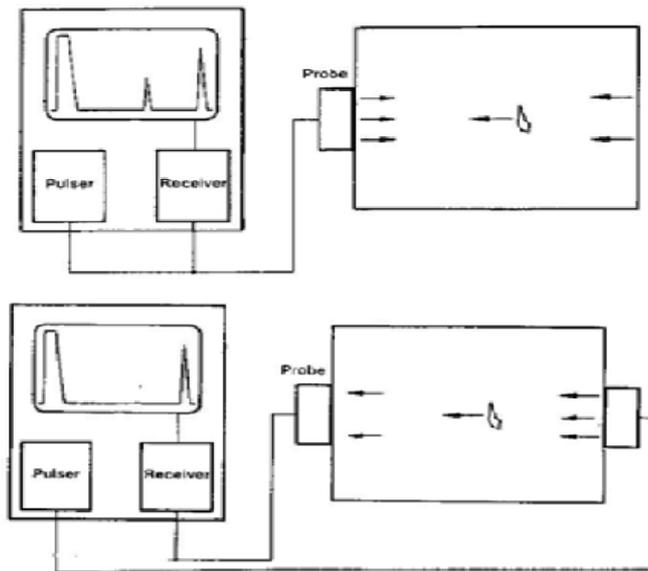
FIG. 2.8 Basic components of an ultrasonic flaw detection system.

(a) Pulse echo method

(b) Through transmission method.

2.2.7. Leak testing (LT)

The leak testing method of NDT includes several techniques to detect, locate and measure leaks which have occurred in the pressure boundary or envelope of a component, vessel, pipeline or piping component.

Many modern designs are based on the "leak before break" principle, in which critical thicknesses and other dimensions are selected so that a vessel will crack and leak before the crack grows to catastrophic proportions. In these applications, the ability to detect the leak at a very early stage is important. In other applications, checking for a through leak is the only way to fully assure the integrity of a component or weld.

The pressure envelope separates two areas which are or could be at different pressures. The contained fluid will try to travel from the region of higher pressure to that of lower pressure.

The relative pressures may be natural or imposed (where the envelope is pressurized or the potential exit point is subjected to a vacuum as part of the inspection procedure [21].

2.2.9. 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. 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 its 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 make 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, and 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 fulfil the requirements of today's industrial production concerning integrateability, automation, speed, reliability and profitability.

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.

A few recent developments in some areas of NDT are briefly reviewed below [21].

Chapter three

The XRF principles

3.1 definition of XRF

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 [14].

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 [15].

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 [22].

3.2 Basics of XRF

In XRF, X-rays produced by 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 discrete energies (equivalent to colors in optical light) that are characteristic for these elements. A different energy is equivalent to a

different color. By measuring the energies (determining the colors) of the radiation emitted by the sample it is possible to determine which elements are present. This step is called qualitative analysis. By measuring the intensities of the emitted energies (colors) it is possible to determine how much of each element is present in the sample. This step is called quantitative analysis [22].

3.3 basic principles X-rays

X-rays can be seen as electromagnetic waves with their associated wavelengths, or as beams of photons with associated energies. Both views are correct, but one or the other is easier to understand depending on the phenomena to be explained. Other electromagnetic waves include light, radio waves and γ-rays. Figure 3.2 shows that X-rays have wavelengths and energies between γ-rays and ultra violet 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 proportional to its energy, according to $E*\lambda$=hc. E is the energy in keV and λ the wavelength in nm. The term hc is the product of Planck's constant and the velocity of light and has, using keV and nm as units, a constant value of 1.23985 [22].

3.3.1 Interaction of X-rays with matter

There are three main interactions when X-rays contact matter: Fluorescence, Compton scatter and Rayleigh scatter (see Figure 3.2). If a beam of X-ray photons is directed towards a slab of material a fraction will be transmitted through, a fraction is absorbed (producing fluorescent radiation) and a fraction is scattered back. Scattering can occur with a loss of energy and without a loss of energy. The first is known as Compton scatter and the second Rayleigh 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 [22].
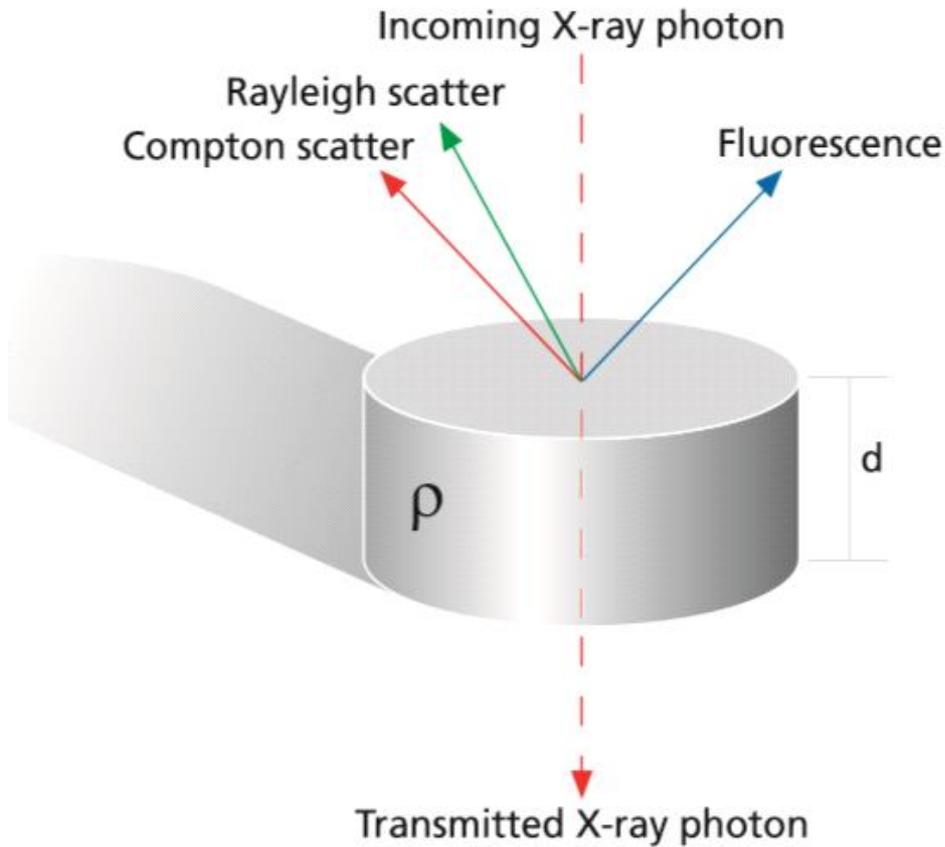
Figure 3.1. Three main interactions of X-rays with matter

3.4 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.
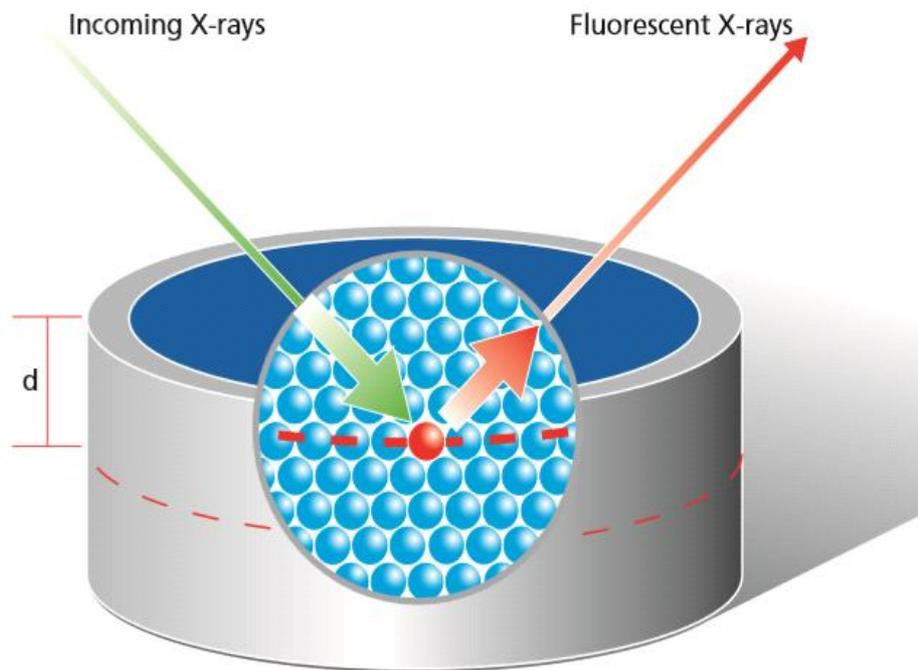
Figure 3.2. Absorption of incoming and fluorescent X-rays

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 electronsfrom 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 [22].
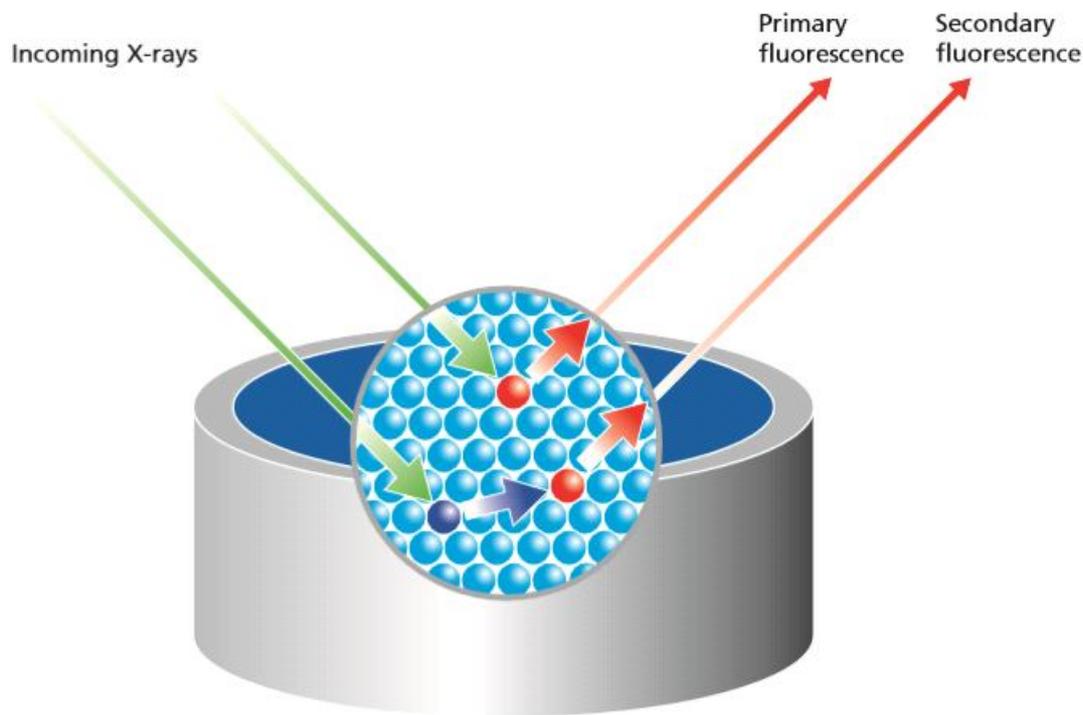
Figure 3.3Primary and secondary fluorescence

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%.

3.5 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 Figure3.5. 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.
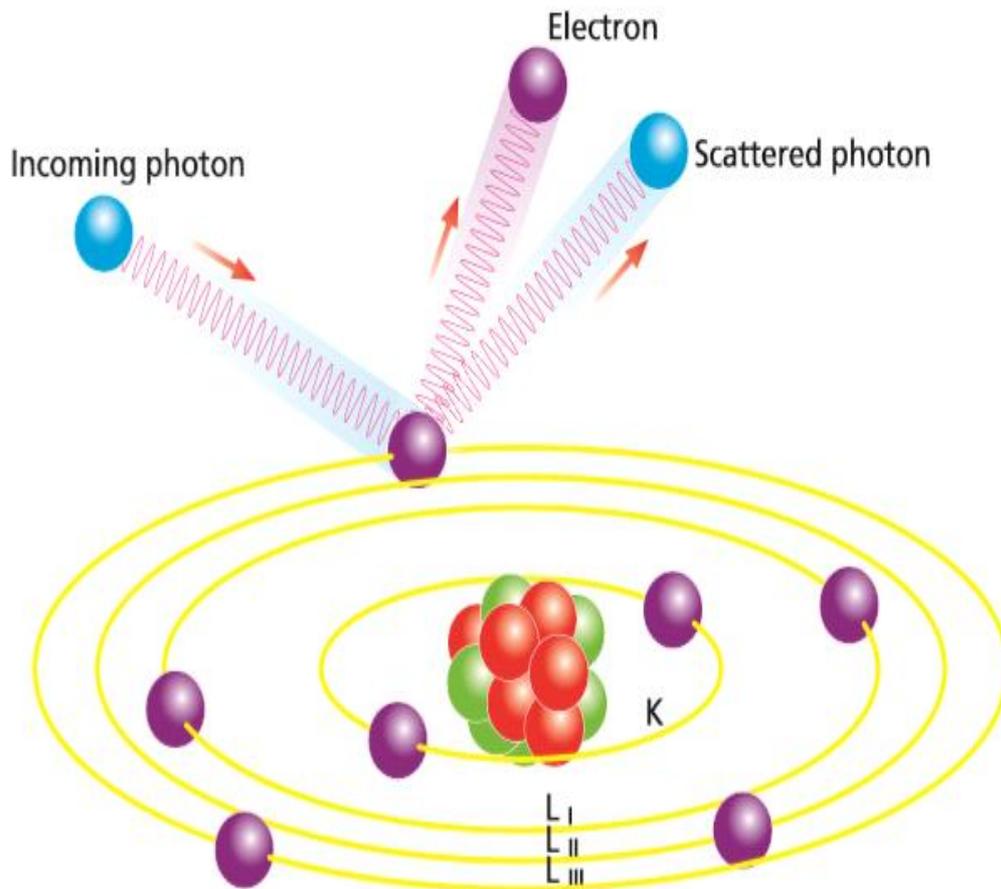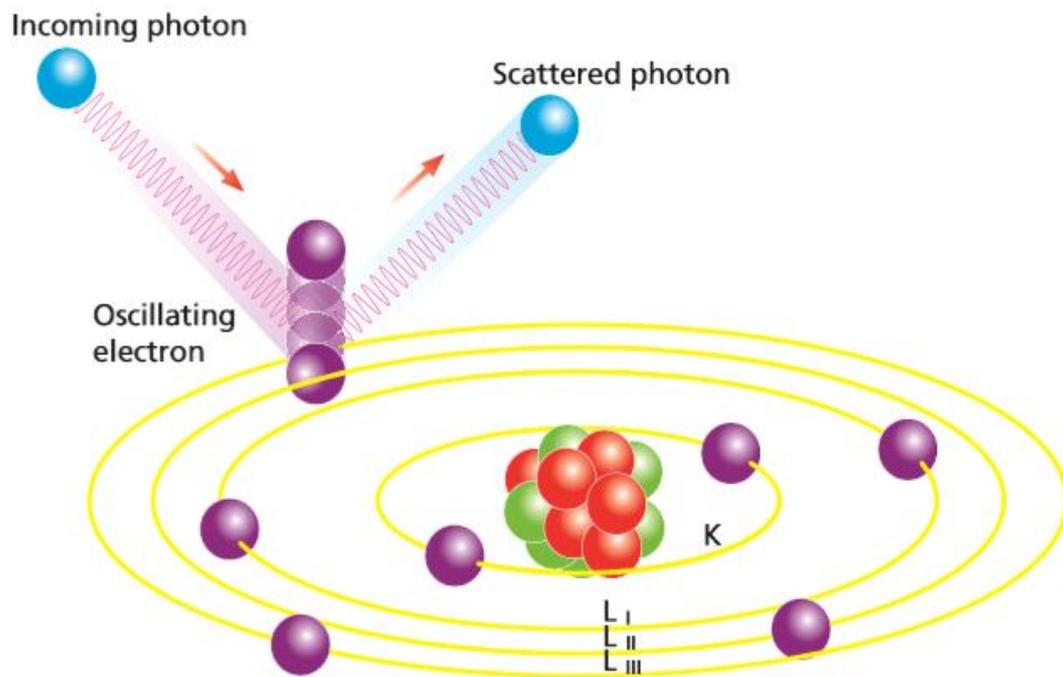
Fig. 3.4 Compton scatter

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 [22].

'Figure(3.5)Compton and Rayleigh scatters

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. Figure 12 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 [12].
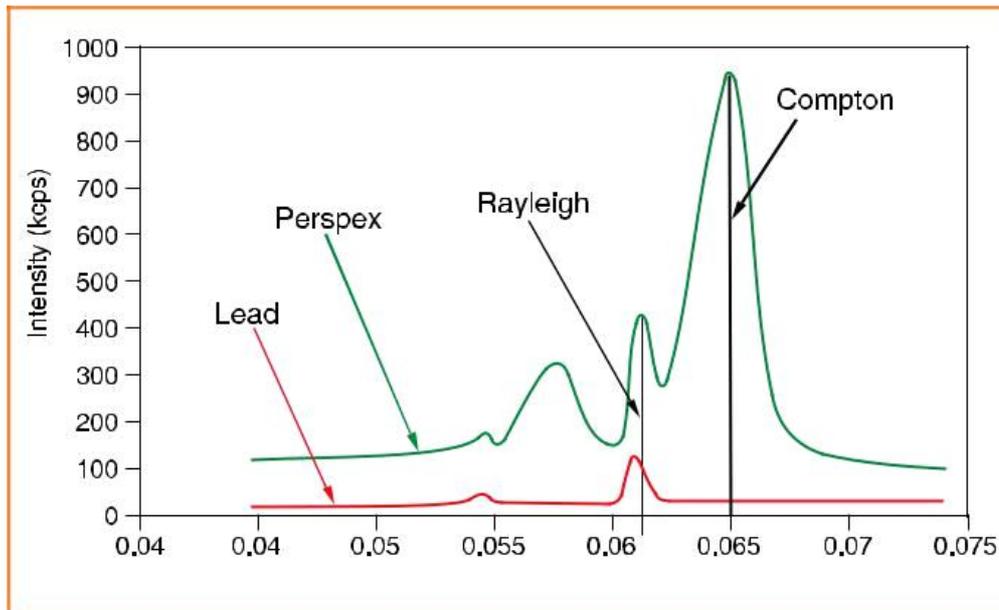
Wavelength (nm)

Figure 3.6.Compton and Rayleigh scatter for light and heavy elements

3.6 The XRF spectrometer

The basic concept for all spectrometers is a source, a sample and a detection system. The source irradiates a sample, and a detector measures the radiation coming from the sample.

In most cases the source is an X-ray tube, and this booklet will only discuss such spectrometers (alternative types use a radioactive source or synchrotron). Spectrometer systems are generally divided into two main groups: energy dispersive systems (EDXRF) and wavelength dispersive systems (WDXRF).

The difference between the two systems is found in the detection system.

EDXRF spectrometers have a detector that is able to measure the different energies of the characteristic radiation coming directly from the sample. The detector can separate the radiation from the sample into the radiation from the elements in the sample. This separation is called dispersion.

WDXRF spectrometers use an analyzing crystal to disperse the different energies. All radiation coming from the sample falls on the crystal. The crystal diffracts the different energies in different directions, similar to a prism that disperses different colors in different directions. The next sections explain the differences between the spectrometer types in more detail, followed by a description of all the individual spectrometer components [22].

3.7 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 done 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 [22].

3.8 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 [22].

3.9 XRF measurements

XRF is a very sensitive technique and samples must be clean. Even fingerprints on a sample can affect the result of the analysis. For accurate results, the spectrometer (for example, the kV settings of the tube or the detector settings) is tuned to the elements to be analyzed. Bad settings can lead to poor results. In EDXRF a whole spectrum is measured simultaneously and the area of a peak profile determines the concentration of an element. Measuring the height of the peak profile is an alternative, but a lot of information would be lost because the area of a peak profile is less sensitive to noise than the height of the same peak.

In WDXRF it is common practice to measure only at the top of the peak profile. The positionsof the peaks are known and measuring only at the top position gives the best accuracy and the lowest measuring time [22].

3.12.1 Optimum measurement conditions

'Optimum' can be defined in many different ways, and the definition depends on the criteria used. The criteria can be highest intensity, lowest background, minimum line overlap and many others. A high intensity and low background have the advantage that lines can be detected and measured accurately and quickly. Minimum line overlap has the advantage that the intensity of the lines can be determined directly, without sophisticated mathematical techniques. Weak lines can also be difficult to detect on the tails of strong lines.

The maximum intensity of a line is achieved if the energy of the incoming radiation is just above the absorption edge of that line. In WDXRF systems and EDXRF systems with direct excitation, this can be done by applying a voltage to the tube so that the largest part of the tube spectrum (continuum or tube line) has an energy just above the absorption edge of the analytical line.

In EDXRF systems using secondary targets, it is done by using a target that has a fluorescent line with an energy just above the absorption edge of the analytical line. The voltage applied to the tube is such that the spectrum of the tube excites the element(s) in the target optimally, according to the same principles described for WDXRF systems. If no such target is available, a Barkla target is used to scatter the total tube spectrum. The tube voltage is selected so that the largest part of the tube spectrum has an energy just above the absorption edge of the analytical line.

Line overlap occurs when the line of one element overlaps the line of another element. The interfering line can come from an element in the sample, but also from an element in the tube, crystal, secondary target, or any other component in the optical path.

High resolution and/or dispersion achieve minimum line overlap. In WDXRF spectrometers, the crystal and collimator have a large effect on the line overlap, which can be minimized by selecting the proper crystal and collimator. In EDXRF spectrometers, the detector and MCA settings have a large effect on the resolution and must be selected carefully. In some cases it is also worth measuring a weaker line of an element if a strong line is overlapped and the weaker line is not.

For spectrometers using secondary targets, the selection of the proper target is essential. Scattered target lines can interfere with the lines of the sample so

Selecting the target that gives the lowest interference is advisable. Using a target that only excites the elements of interest and not elements that give interfering lines helps to reduce the line overlap [22].

Chapter four

Material and methods

4.1 Introduction

In this thesis experiment was carried out in petroleum technical center (PTC) Khartoum AirPort Street using XRF liquid techniques methods.

4.2 materials and method

Four different types of water Tab, Miner,Zamzam and River water were collected from its different regions and sources in order to investigate the elements that exist in those samples before and after magnetization using XRF techniques.

4.2.1 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.

4.3.2 Liquids preparation

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.

4.5 Results

The results obtained for different samples after and before subjected to magnetization were tabulated in the tables below and graphs of the datawas explored in figures.

No	Kind of Water	Magnetized		None magnetized	
		element	%	element	%
1		Cr	0.04	Cr	0.02
2		Ni	0.01	Ni	0.01
3	From tab	Mn	0.00	Mn	0.01
4		Fe	0.02	Fe	0.02
5		Pb	0.00	Pb	0.00
1		Cr	0.02	Cr	0.03
2		Ni	0.01	Ni	0.01
3	Miner water	Fe	0.02	Fe	0.02
4		Pb	0.00	Pb	0.00
5		Mn	0.00	Zn	0.00
6		Zn	0.00		
1		Cr	0.04	Cr	0.04
2		Ni	0.01	Mn	0.01
3	Zamzam water	Fe	0.02	Ni	0.01
4		Pb	0.00	Fe	0.03
5				Zn	0.01
1		Cr	0.03	Cr	0.05
2	River water	Ni	0.01	Ni	0.01
3		Fe	0.02	Fe	0.05
4		Pb	0.00	Pb	0.00

Table (4.1) Samples from different sources of water before and after magnetization
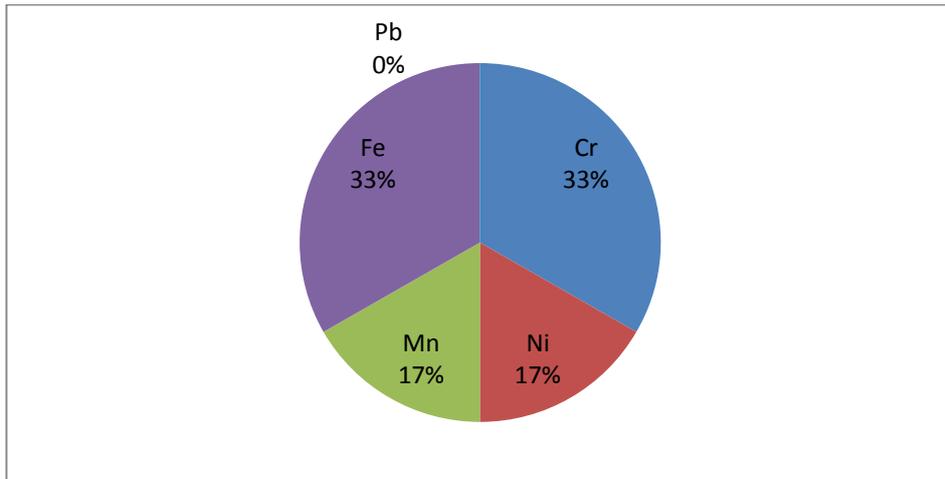
Fig . (4.1) the elements which exist in tab water before subject to magnetization
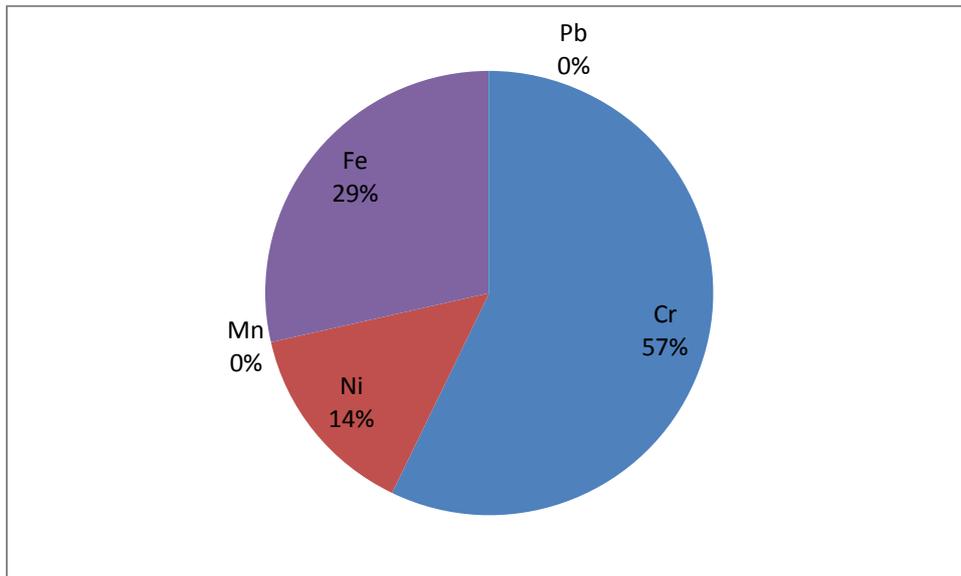

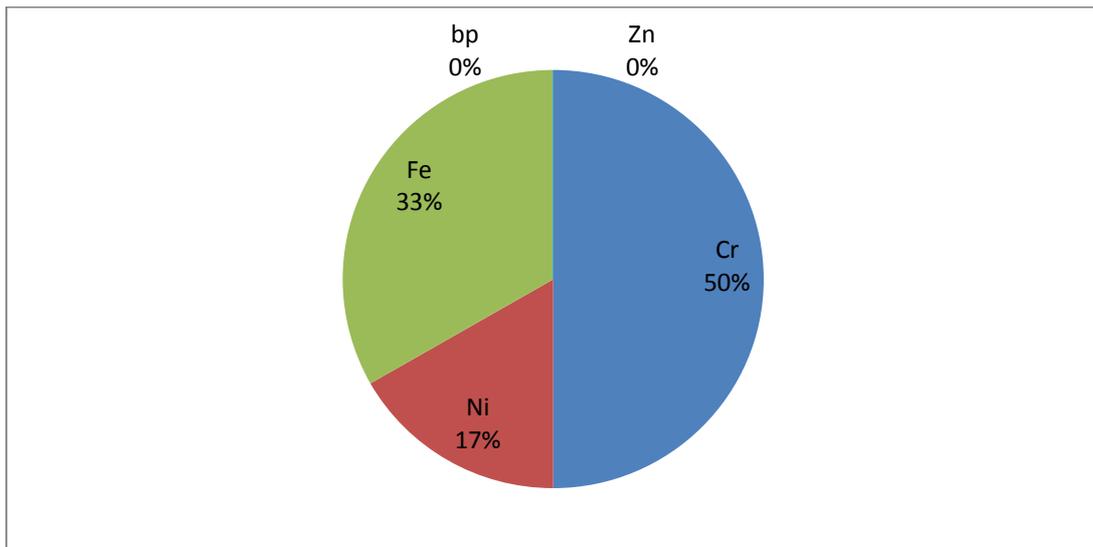Fig .4.2the elements which exist in tab water after subject to magnetization

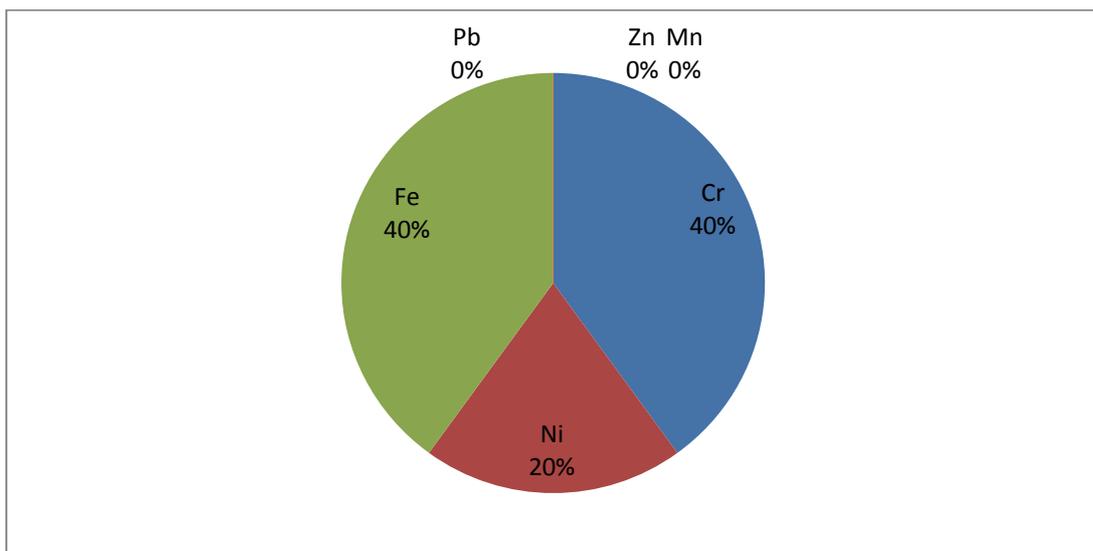Fig .4.3the elements which exist in miner water before subject to magnetization

Fig 4.4the elements which exist in miner water after subject to magnetization
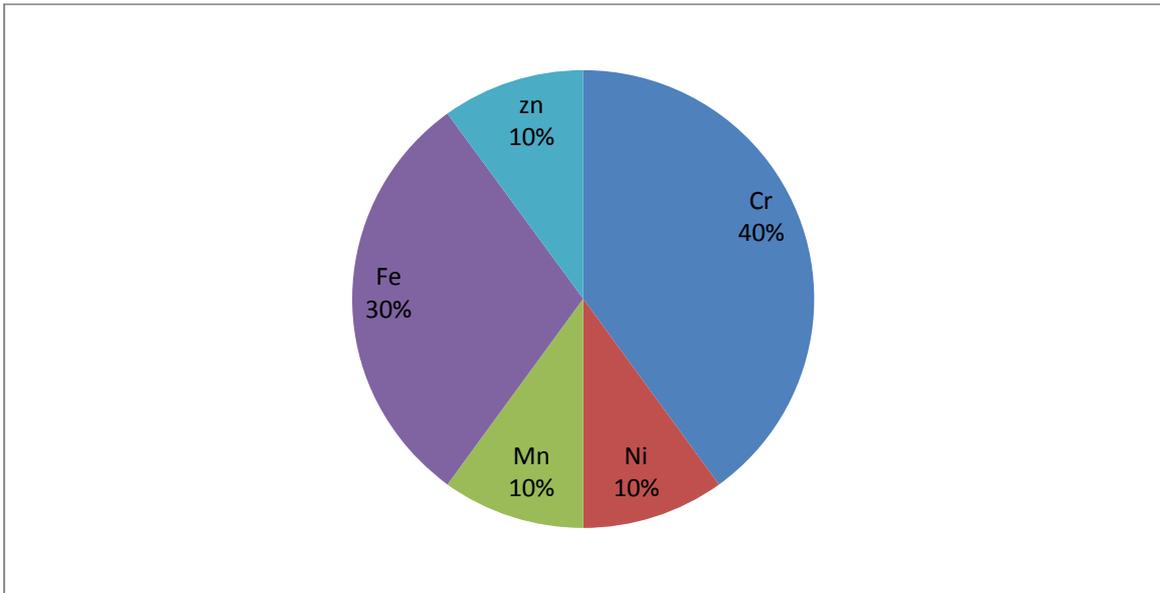
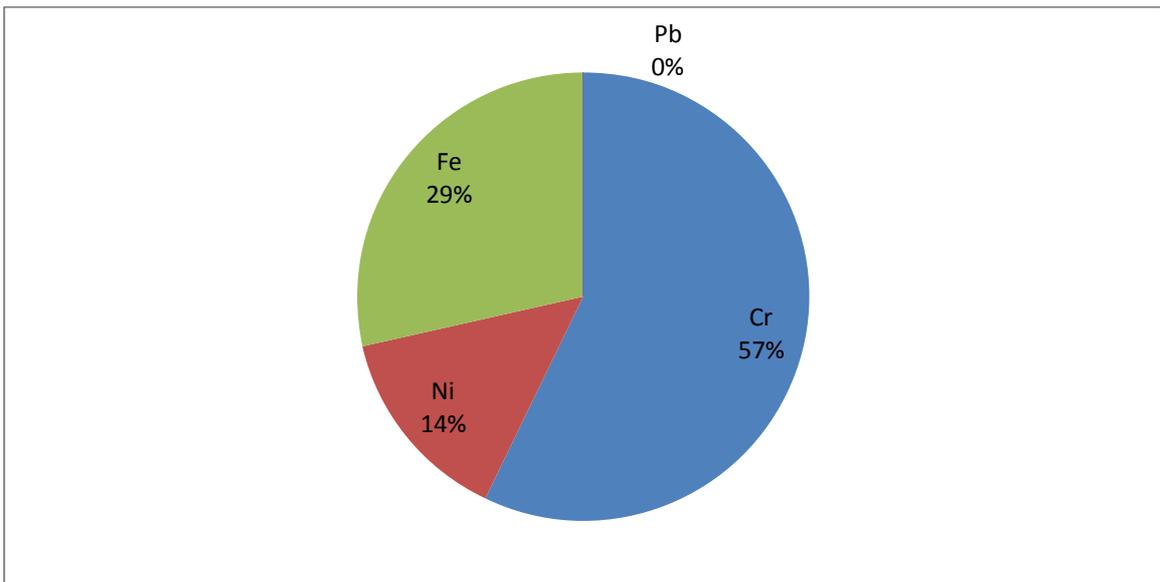Fig .4.5the elements which exist in Zamzam water before subject to magnetization

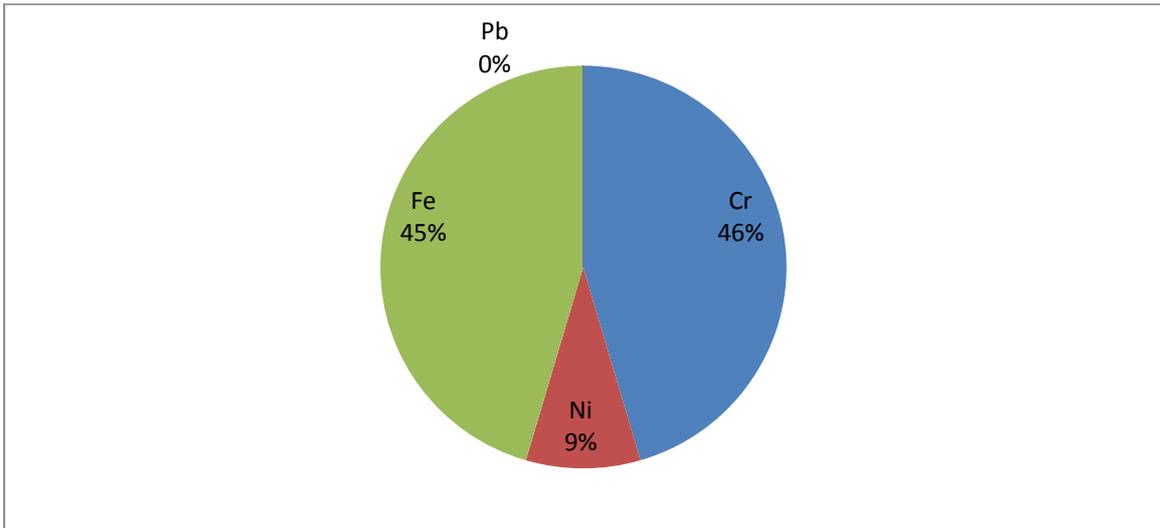Fig .4.6the elements which exist in Zamzam water after subject to magnetization

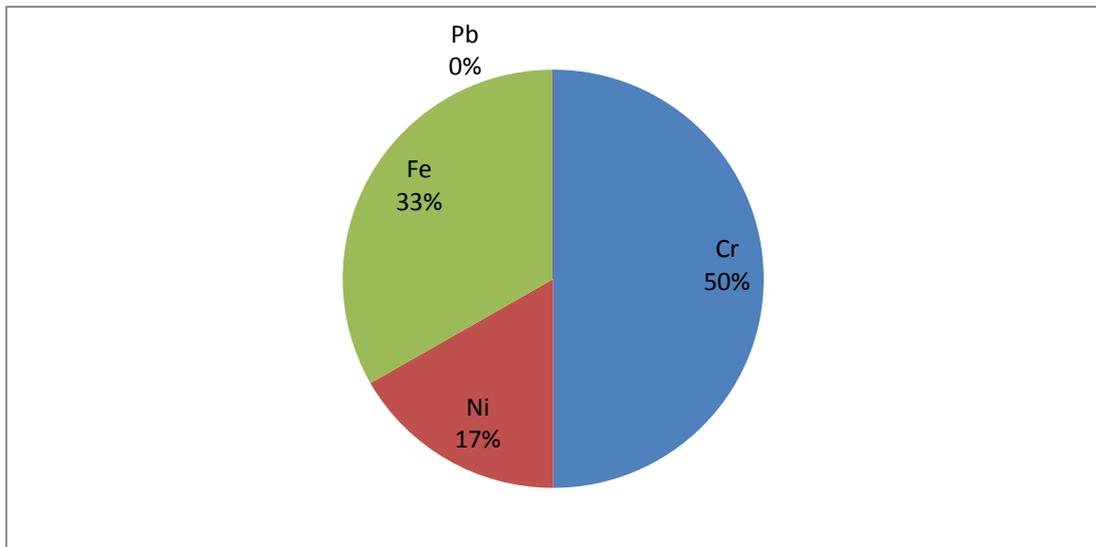Fig .4.7 the elements which exist in river water before subject to magnetization

Fig .4.8 the elements which exist in river water after subject to magnetization

4.6 Discussions

From figures (4.1, 2, 3……8) it was clear that there are some element disappeared after magnetization by bendingitself and not detected by XRF such as Fe and Ni in river, Tab and Zamzam water(its amount was decreased after magnetization) in other solution such as miner water the ferrous was stretched so that the scattering rate will increases because that it was detected (its amount has been increased).

We expect that the Fe and Ni were correlated together they appeared and disappeared to together (they increased and decreased by the same manner).

4.7 Conclusions

In this thesis the main element exist in all sources are Cr and Fe and Ni.

The percentage of ferrous and nickel decreases or increases simultaneously after magnetization while Cr decreases after magnetization.

Some elements such as Pb exist in few percentages as passive elements.

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