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Structural studies of Fe_2O_3 using X-Ray diffraction

استخدام تقنية حيود الاشعة السينية لدراسة الخواص التركيبية لاكسيد الحديد

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الآية

بسم الله الرحمن الرحيم

قَالَ تَعَالَىٰ:

﴿لَقَدۡ أَرۡسَلۡنَا رُسُلَنَا بِٱلۡبَيِّنَٰتِ وَأَنزَلۡنَا مَعَهُمُ ٱلۡكِتَٰبَ
وَٱلۡمِيزَانَ لِيَقُومَ ٱلنَّاسُ بِٱلۡقِسۡطِۖ وَأَنزَلۡنَا ٱلۡحَدِيدَ فِيهِ بَأۡسٞ
شَدِيدٞ وَمَنَٰفِعُ لِلنَّاسِ وَلِيَعۡلَمَ ٱللَّهُ مَن يَنصُرُهُۥ وَرُسُلَهُۥ بِٱلۡغَيۡبِۚ إِنَّ ٱللَّهَ
قَوِيٌّ عَزِيزٞ﴾

سورة الحديد الايه: (25)

Dedication

To dear mother…

To dear father……

To all my brothers, I dedicate this research

To my colleagues comrades the trail to my teachers .

To all those who love me honestly, and I love them equally.

Acknowledgements

Thanks first to Allah…

Thanks to my family…

And thanks a lot to our supervisor:

Dr.nodar osman khalifa

Abstract

Iron oxides are generally studied due to their interesting physical properties. For instance, their electric and magnetic properties have many applications in electrical, magnetic, and gas sensing devises. In particular, we note that over the past few years, the study of system of hematite (α-Fe_2O_3) has attracted considerable attention both from basic and applied viewpointsIron oxide is the inorganic compound, it is one of the three main oxides of iron, the other two being (FeO), which is rare, and (Fe_3O_4), which also occurs naturally as the mineral magnetite.

This research studied the structural properties of Fe_2O_3(99.9%) compound using diffraction phenomena, have been studied. The X-Ray data was refined by two software program (Win-fit and powdercell method) in order to find the lattice parameters of this compound. The crystal structure of $Fe_2O_3$was found to hexagonal with lattice parameters a=5.0305/A, b=5.0305/A^o, and c=13.7380/ A^o, and the angle are α=90, β=90 and γ=120^o .The experimental work has been done at Al Neelain University, physics department X-Ray unit.

الملخص

تدرس اكاسيد الحديد عموماً بسبب خصائصها الفيزيائية الممتعة. علي وجه الخصوص ، خصائصها الكهربية والمغناطيسية. نلاحظ علي مدي السنوات القليلة الماضية ان دراسة نظام الهيماتيت قد جذب اهتماماً كبيراً من الجوانب الاساسية والتطبيقية. اكسيد الحديد هو مركب غير عضوي وهو احد الاكاسيد الثلاثة الرئيسيةفي هذا البحث قد درست الخواص التركيبية لمركب اكسيد الحديد باستخدام ظاهرة الحيود. حللت نتائج حيود الاشعة السينية باستخدام برنامجي (winfit and powdercell) وتم الحصول علي البنية التركيبة لاكسيد الحديد Fe_2O_3وهي سداسية التركيب ووجدت المعاملات البلورية له تساوي $a=5.0305/A$ و $b=5.0305/A$ و$c=13.7380/A$ والزوايا تساوي $\alpha=90$ و$\beta=90$ و $\gamma=120$ وتمت التجربة في جامعة النيلين قسم الفيزياء وحدة الاشعه السينية.

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Chapter one

Introduction

In this chapter, we briefly outline the some review of electromagnetic spectrum and uses of radiation

1.1: Radiation

The word radiation arises from the phenomenon of waves radiating from a source. This aspect leads to a system of measurements and physical units that are applicable to all types of radiation. Because such radiation expands as it passes through space, and as its energy is conserved (in vacuum), the intensity of all types of radiation from a point source follows an inverse-square law in relation to the distance from its source. This law does not apply close to an extended source of radiation or for focused beams.

In physics, radiation is the emission or transmission of energy in the form of waves or particles through space or through a material medium [1,2]. This includes:

- Electromagnetic radiation, such as radio waves, visible light, x-rays, and gamma radiation (γ).

- Particle radiation, such as alpha radiation (α), beta radiation (β), and neutron radiation.
- Acoustic radiation, such as ultrasound, sound, and seismic waves (dependent on a physical transmission medium).
- Gravitational radiation, radiation that takes the form of gravitational waves, or ripples in the curvature of space-time.

Figure (1.1) represents Illustration of the relative abilities of three different types of ionizing radiation to penetrate solid matter. Typical alpha particles (α) are stopped by a sheet of paper, while beta particles (β) are stopped by an aluminum plate. Gamma radiation (γ) is damped when it penetrates lead.

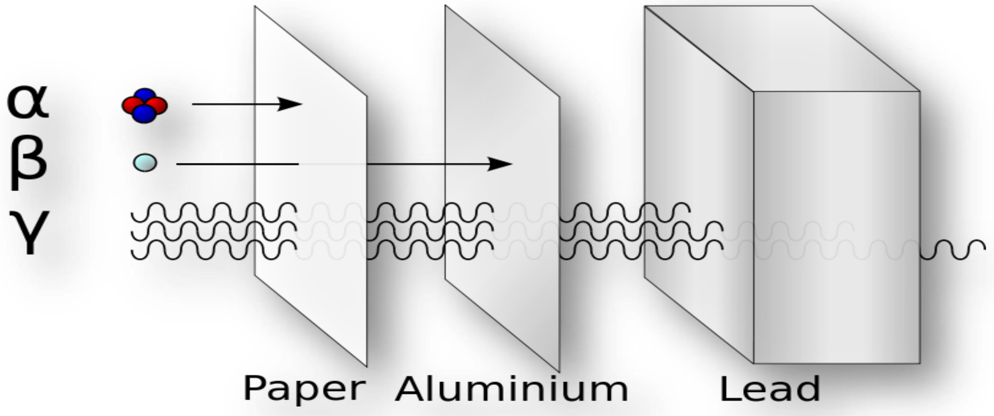


Figure (1.1): represents the abilities of radiation to penetrate the material.

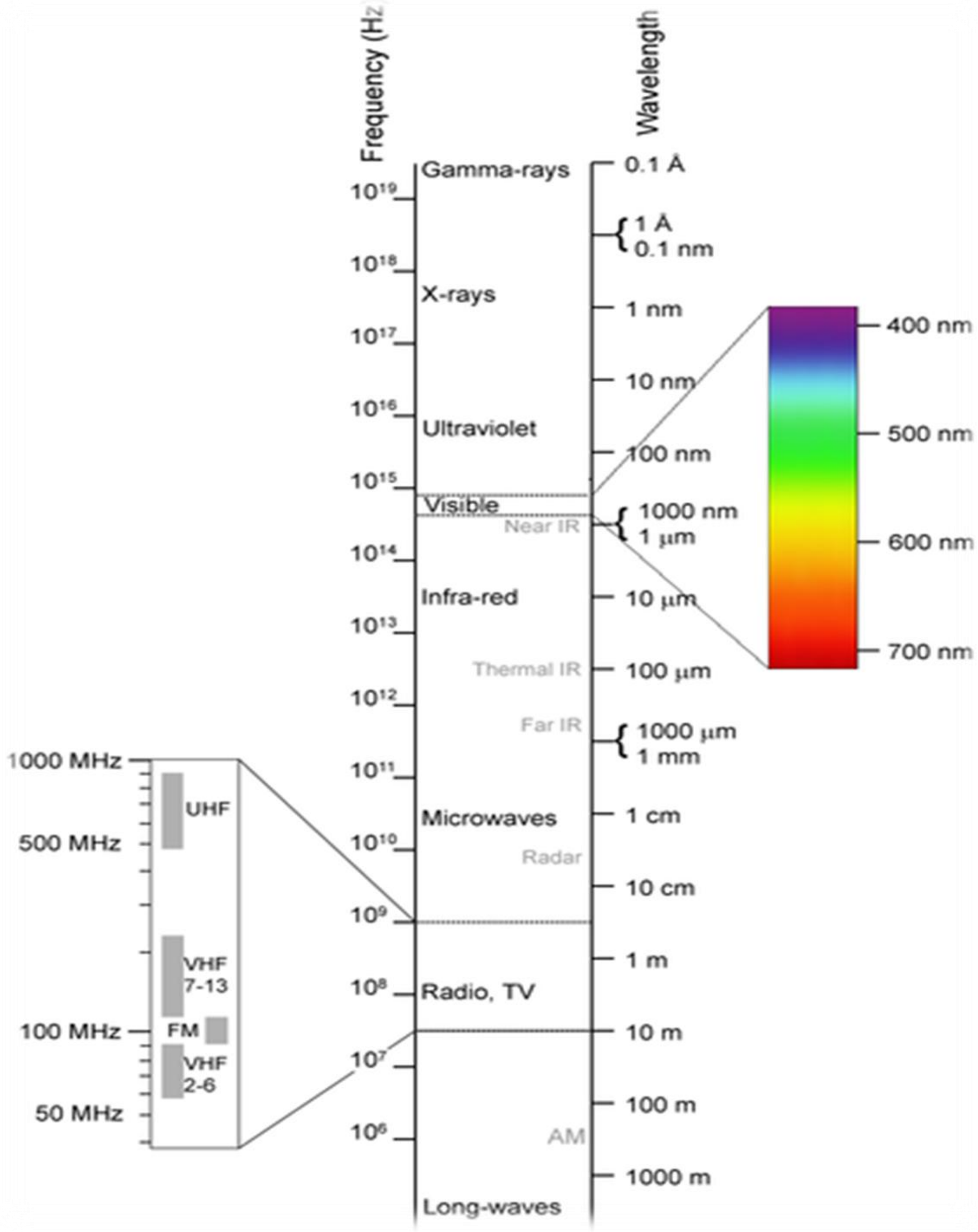
Radiation is often categorized as either ionizing or non-ionizing depending on the energy of the radiated particles. Ionizing radiation carries more than 10 eV [electron volt], which is enough to ionize atoms and molecules, and break chemical bonds. A common source of ionizing radiation is radioactive materials that emit α, β, or γ radiation, consisting of helium nuclei, electrons or positrons, and photons, respectively. Other sources include X-rays from medical radiography examinations and muons, mesons, positrons, neutrons and other particles that constitute the secondary cosmic rays that are produced after primary cosmic rays interact with Earth's atmosphere [5].

Gamma rays, X-rays and the higher energy range of ultraviolet light constitute the ionizing part of the electromagnetic spectrum. The lower-energy, longer-wavelength part of the spectrum including visible light, infrared light, microwaves, and radio waves is non-ionizing its main effect when interacting with tissue is heating. This type of radiation only damages cells if the intensity is high enough to cause excessive heating. Ultraviolet radiation has some features of both ionizing and non-ionizing radiation. While the part of the ultraviolet spectrum that penetrates the Earth's atmosphere is non-ionizing, this radiation does far more damage to many molecules in biological systems than can be accounted for by heating effects, sunburn being a well-known example. These properties derive from ultra-violet’s power to alter chemical bonds, even without having quite enough energy to ionize atoms.

1.1.1: **The electromagnetic spectrum**

The electromagnetic spectrum is the range of all possible electromagnetic radiation frequencies.The electromagnetic spectrum of an object is the characteristic distribution of electromagnetic radiation emitted by, or absorbed by, that particular object.The non-ionizing portion of electromagnetic radiation consists of electromagnetic waves that (as individual quanta or particles) are not energetic enough to detach electrons from atoms or molecules and hence cause their ionization. These include radio waves, microwaves, infrared, and (sometimes) visible light. The lower frequencies of ultraviolet light may cause chemical changes and molecular damage similar to ionization, but is technically not ionizing. The highest frequencies of ultraviolet light, as well as all X-rays and gamma-rays are ionizing [3].

Figure (1.2) represents the electromagnetic spectrum and illustrates the frequency and wavelength to radiation.



Figurer (1.2): represents the electromagnetic spectrum

The occurrence of ionization depends on the energy of the individual particles or waves, and not on their number. An intense flood of particles or waves will not cause ionization if these particles or waves do not carry enough energy to be ionizing, unless they raise the temperature of a body to a point high enough to ionize small fractions of atoms or molecules by the process of thermal-ionization.

1.2: Non-ionizing radiation

The kinetic energy of particles of non-ionizing radiation is too small to produce charged ions when passing through matter. For non-ionizing electromagnetic radiation, the associated particles (photons) have only sufficient energy to change the rotational, vibrational or electronic valence configurations of molecules and atoms. The effect of non-ionizing forms of radiation on living tissue has only recently been studied. Nevertheless, different biological effects are observed for different types of non-ionizing radiation.

1.2: Ultraviolet light

The lower part of the spectrum of ultraviolet, called soft UV, from 3 eV to about 10 eV, is non-ionizing. However, the effects of non-ionizing ultraviolet on chemistry and the damage to biological systems exposed to it (including oxidation, mutation, and cancer) are such that even this part of ultraviolet is often compared with ionizing radiation.

1.2.1: Visible light

Light, or visible light, is a very narrow range of electromagnetic radiation of a wavelength that is visible to the human eye, or 380–750 nm which equates to a frequency range of 790 to 400 THz respectively. More broadly, physicists use the term "light" to mean electromagnetic radiation of all wavelengths, whether visible or not.

1.2.2: Infrared

Infrared (IR) light is electromagnetic radiation with a wavelength between 0.7 and 300 micrometers, which corresponds to a frequency range between 430 and 1 THz respectively. IR wavelengths are longer than that of visible light, but shorter than that of microwaves. Bright sunlight provides an irradiance of just over 1 kilowatt per square meter at sea level. Of this energy, 53% is infrared radiation, 44% is visible light, and 3% is ultraviolet radiation.

1.2.3: Microwave

Microwaves are electromagnetic waves with wavelengths ranging from as short as one millimeter to as long as one meter, which equates to a frequency range of 300 MHz to 300 GHz.

1.2.4:Radio waves

Radio waves are a type of electromagnetic radiation with wavelengths in the electromagnetic spectrum longer than infrared light. Like all other electromagnetic waves, they travel at the speed of light. Naturally occurring radio waves are made by lightning, or by certain astronomical objects.

1.3: Ionizing radiation

Radiation with sufficiently high energy can ionize atoms; that is to say it can knock electrons off atoms and create ions. Ionization occurs when an electron is stripped (or "knocked out") from an electron shell of the atom, which leaves the atom with a net positive charge. Because living cells and, more importantly, the DNA in those cells can be damaged by this ionization, exposure to ionizing radiation is considered to increase the risk of cancer. Particle radiation from radioactive material or cosmic rays almost invariably carries enough energy to be ionizing.

If the source of the ionizing radiation is a radioactive material or a nuclear process such as fission or fusion, there is particle radiation to consider. Particle radiation is subatomic particles accelerated to relativistic speeds by nuclear reactions. Because of their momenta they are quite capable of knocking out electrons and ionizing materials, but since most have an electrical charge, they don't have the penetrating power of ionizing radiation. The exception is neutron particles. There are several different kinds of these particles, but the majority are alpha particles, beta particles, neutrons, and protons. Roughly speaking, photons and particles with energies above about 10 electron volts (eV) are ionizing [4].

1.3.1: Ultraviolet radiation

Ultraviolet, of wavelengths from 10 nm to 125 nm, ionizes air molecules, causing it to be strongly absorbed by air and by ozone (O3) in particular. Ionizing UV therefore does not penetrate Earth's atmosphere to a significant degree, and is sometimes referred to as vacuum ultraviolet. Although present in space, this part of the UV spectrum is not of biological importance, because it does not reach living organisms on Earth.

1.3.2:X-ray

X-rays are electromagnetic waves with a wavelength less than about 10^{-9} m (greater than $3x10^{17}$ Hz and 1,240 eV). When an X-ray photon collides with an atom, the atom may absorb the energy of the photon and boost an electron to a higher orbital level or if the photon is very energetic, it may knock an electron from the atom altogether, causing the atom to ionize. Generally, larger atoms are more likely to absorb an X-ray photon since they have greater energy differences between orbital electrons.

1.3.3: Gamma radiation

Gamma (γ) radiation consists of photons with a wavelength less than $3x10^{-11}$ meters (greater than 10^{19} Hz and 41.4 keV). Gamma radiation emission is a nuclear process that occurs to rid an unstable nucleus of excess energy after most nuclear reactions. Both alpha and beta particles have an electric charge and mass, and thus are quite likely to interact with other atoms in their path. Gamma radiation, however, is composed of photons, which have neither mass nor electric charge and, as a result, penetrates much further through matter than either alpha or beta radiation.

1.3.4: Alpha radiation

Alpha particles are helium-4nuclei (two protons and two neutrons). They interact with matter strongly due to their charges and combined mass, and at their usual velocities only penetrate a few centimeters of air, or a few millimeters of low density material (such as the thin mica material which is specially placed in some Geiger counter tubes to allow alpha particles in). This means that alpha particles from ordinary alpha decay do not penetrate the outer layers of dead skin cells and cause no damage to the live tissues below. Some very high energy alpha particles compose about 10% of cosmic rays, and these are capable of penetrating the body and even thin metal plates. However, they are of danger only to astronauts, since they are deflected by the Earth's magnetic field and then stopped by its atmosphere.

1.3.5:Beta radiation

Beta-minus (β^-) radiation consists of an energetic electron. It is more penetrating than alpha radiation, but less than gamma. Beta radiation from radioactive decay can be stopped with a few centimeters of plastic or a few millimeters of metal. It occurs when a neutron decays into a proton in a nucleus, releasing the beta particle and an antineutrino. Beta radiation from linac accelerators is far more energetic and penetrating than natural beta radiation. It is sometimes used therapeutically in radiotherapy to treat superficial tumors.

Beta-plus (β^+) radiation is the emission of positrons, which are the antimatter form of electrons. When a positron slows to speeds similar to those of electrons in the material, the positron will annihilate an electron, releasing two gamma photons of 511 keV in the process. Those two gamma photons will be traveling in (approximately) opposite direction. The gamma radiation from positron annihilation consists of high energy photons, and is also ionizing.

1.3.6: Neutron radiation

Neutrons are categorized according to their speed/energy. Neutron radiation consists of free neutrons. These neutrons may be emitted during either spontaneous or induced nuclear fission. Neutrons are rare radiation particles; they are produced in large numbers only where chain reactionfission or fusion reactions are active; this happens for about 10 microseconds in a thermonuclear explosion, or continuously inside an operating nuclear reactor; production of the neutrons stop almost immediately in the reactor when it goes non-critical.Neutrons are the only type of ionizing radiation that can make other objects, or material, radioactive. This process, called neutron activation, is the primary method used to produce radioactive sources for use in medical, academic, and industrial applications. Even comparatively low speed thermal neutrons cause neutron activation (in fact, they cause it more efficiently). Neutrons do not ionize atoms in the same way that charged particles such as protons and electrons do (by the excitation of an electron), because neutrons have no charge.It is through their absorption by nuclei which then become unstable that they cause ionization.

1.3.7: Cosmic radiation

There are two sources of high energy particles entering the Earth's atmosphere from outer space: the sun and deep space. The sun continuously emits particles, primarily free protons, in the solar wind, and occasionally augments the flow hugely with coronal mass ejections (CME).

1.4: Uses of the radiation

1.4.1 Medicine

Radiation and radioactive substances are used for diagnosis, treatment, and research. X-rays, for example, pass through muscles and other soft tissue but are stopped by dense materials. This property of X-rays enables doctors to find broken bones and to locate cancers that might be growing in the body. Radiation used for cancer treatment is called ionizing radiation because it forms ions in the cells of the tissues it passes through as it dislodges electrons from atoms. This can kill cells or change genes so the cells cannot grow.

1.4.2 Communication

All modern communication systems use forms of electromagnetic radiation. Variations in the intensity of the radiation represent changes in the sound, pictures, or other information being transmitted. For example, a human voice can be sent as a radio wave or microwave by making the wave vary to correspond variations in the voice. Musicians have also experimented with gamma sonification, or using nuclear radiation, to produce sound and music.

1.4.3 Science

Researchers use radioactive atoms to determine the age of materials that were once part of a living organism. The age of such materials can be estimated by measuring the amount of radioactive carbon they contain in a process called radiocarbon dating. Similarly, using other radioactive elements, the age of rocks and other geological features (even some man-made objects) can be determined; this is called Radiometric dating. Environmental scientists use radioactive atoms, known as tracer atoms, to identify the pathways taken by pollutants through the environment. Radiation is used to determine the composition of materials in a process called neutron activation analysis. In this process, scientists bombard a sample of a substance with particles called neutrons. Some of the atoms in the sample absorb neutrons and become radioactive. The scientists can identify the elements in the sample by studying the emitted radiation [5].

Chapter two

X-ray physics

In this chapter, we briefly outline the production and physics of X-Ray then we will give some information about the detectors that can be used to detect this radiations.

X-ray were discovered by Roentgen in 1895.He found that the operation of cathode-ray tube produced fluorescence in a platinum-barium-cyanide screen placed at some distance from the tube. The source of the rays causing this fluorescence was traced to the walls of the cathode-ray tube. In further experiments he found that the interposition of various thicknesses of different substance between the screen and the tube reduced the intensity of the fluorescence but did not obliterate it completely. This showed that this x-rays, had very great penetrating power. It was also found that these rays could blacken a photographic plate and could ionize a gas [6].X-rays are electromagnetic radiation with wavelengths between about 0.02 Å and 100 Å (1Å = 10^{-10} meters). The wavelength of X-rays is on an atomic level and is much smaller than that of visible light (3000 to 8000 Å).

Haga and wind, in 1899, sent abeam of x-rays through a narrow aperture they actually succeeded in getting a diffraction pattern, but the effect was so small that their results were not generally accepted as conclusive . It was not until 1912 that the wave nature of x-ray was definitely established by Laue’s experiments on the diffraction of x-rays by crystal . Barkla’s experiments 1906 on the polarization of x-rays established the fact these rays were transverse wave similar to light waves.

2.1: Nature of X-rays

X-rays can be thought of as waves with wavelengths on the order of 0.1 Å to ~10 Å. The shorter the wavelength,the more energetic the wave Because of the relatively short wavelengths of electromagnetic radiation in the X-ray region, X-rays are high energy waves and are much more penetrating compared to UV, visible, IR, or radio waves.[7] The conversion between energy, frequency, and wavelength is the well-known de Broglie relationship:

$$\boldsymbol{E = hv = {}^{hc}/_{\lambda}} \qquad (2.1)$$

Where v is the frequency, h is Planck’s, c is the speed of light and $\lambda$is the wavelength of the radiation.

2.2 Production of X-ray

An X-ray tube is a vacuum tube designed to produce X-ray photons. The first X-ray tube was invented by Sir William Crookes. The Crookes tube is also called a discharge tube or cold cathode tube. A schematic x-ray tube is shown below.

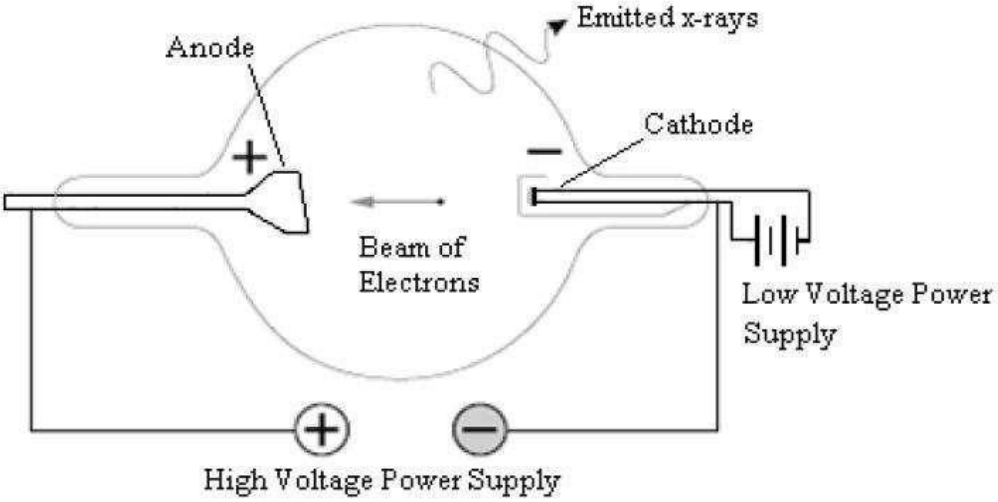


Fig (2.1): A Schematic Diagram of an X-Ray Tube

The glass tube is evacuated to a pressure of air, of about 100 Pascal's, recall that atmospheric pressure is 10^6 Pascal's. The anode is a thick metallic target; it is so made in order to quickly dissipate thermal energy that results from bombardment with the cathode rays. A high voltage, between 30 to 150 kV, is applied between the electrodes; this induces an ionization of the residual air, and thus a beam of electrons from the cathode to the anode ensues. When these electrons hit the target, they are slowed down, producing the X-rays. The X-ray photon-generating effect is generally called the Bremsstrahlung effect,a contraction of the German “brems” for braking, and “strahlung” for radiation. The radiation energy from an X-ray tube consists of discrete energies constituting a line spectrum and a continuous spectrum providing the background to the line spectrum.

2.3 Properties of X-rays

- X-rays travel in straight lines.
- X-rays cannot be deflected by electric field or magnetic field.
- X-rays have a high penetrating power.
- Photographic film is blackened by X-rays.
- Fluorescent materials glow when X-rays are directed at them.
- Photoelectric emission can be produced by X-rays.
- Ionization of a gas results when an X-ray beam is passed through it.

2.4:Continuous Spectrum

When the accelerated electrons (cathode rays) strike the metal target, they collide with electrons in the target. In such a collision part of the momentum of the incident electron is transferred to the atom of the target material, thereby losing some of its kinetic energy, K. This interaction gives rise to heating of the target. The projectile electron may avoid the orbital electrons of the target element but may come sufficiently close to the nucleus of the atom and come under its influence. The projectile electron we are tracking is now beyond the K-shell and is well within the influence of the nucleus. The electron is now under the influence of two forces, namely theattractive Coulomb force and a much stronger nuclear force. The effect of both forces on the electron is to slow it down or decelerate it. The electron leaves the region of sphere of influence of the nucleus with a reduced kinetic energy and flies off in a different direction, because the vector velocity has changed.The loss in kinetic energy reappears as an x-ray photon, as illustrated in Fig (2.2) during deceleration; the electron radiates an X-ray photon of energy $h\nu = \Delta K = K_i - K_f$. The energy lost by incident electrons is not the same for all electrons and so the x-ray photons emitted are not of the same wavelength. This process of X-ray photon emission through deceleration is called Bremsstrahlung and the resulting spectrum is continuous but with a sharp cut-off wavelength. The minimum wavelength corresponds to an incident electron losing all of its energy in a single collision and radiating it away as a single photon.

If K is the kinetic energy of the incident electron, then

$$k = hv = \frac{hc}{\lambda_{min}} \tag{2.2}$$

The cut off wavelength depends solely on the accelerating voltage.

$$hv_{max} = \frac{hc}{\lambda_{min}} = ev \qquad (2.3)$$

Where V is the accelerating voltage

$$v_{max} = \frac{ev}{h} \qquad (2.4)$$

$$, \lambda_{min} = \frac{hc}{ev} \qquad (2.5)$$

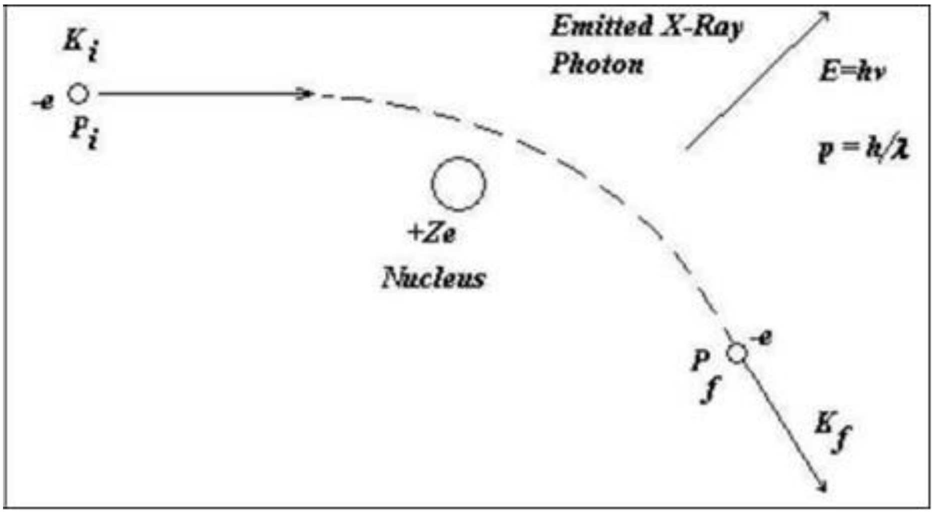


Fig (2.2): Deceleration of an Electron by a Positively Charged Nucleus

2.5: Characteristic X-Ray Spectrum

Because of the large accelerating voltage, the incident electrons can

I. Excite electrons in the atoms of the target.

II. Eject tightly bound electrons from the cores of the atoms.

Excitation of electrons will give rise to emission of photons in the optical region of the electromagnetic spectrum. However when core electrons are ejected, the subsequent filling of vacant states gives rise to emitted radiation in the x-ray region of the electromagnetic spectrum. The core electrons could be from the K-, L- or M- shell [8].

If K-shell (n=1) electrons are removed, electrons from higher energy states falling into the vacant K-shell states, produce a series of lines denoted as K_α, K_β ,... as shown Fig (2.3).

Transitions to the L shell result in the L series and those to the M shell give rise to the M series, and so on.

Since orbital electrons have definite energy levels, the emitted X-ray photons also have well defined energies. The emission spectrum has sharp lines characteristic of the target element.

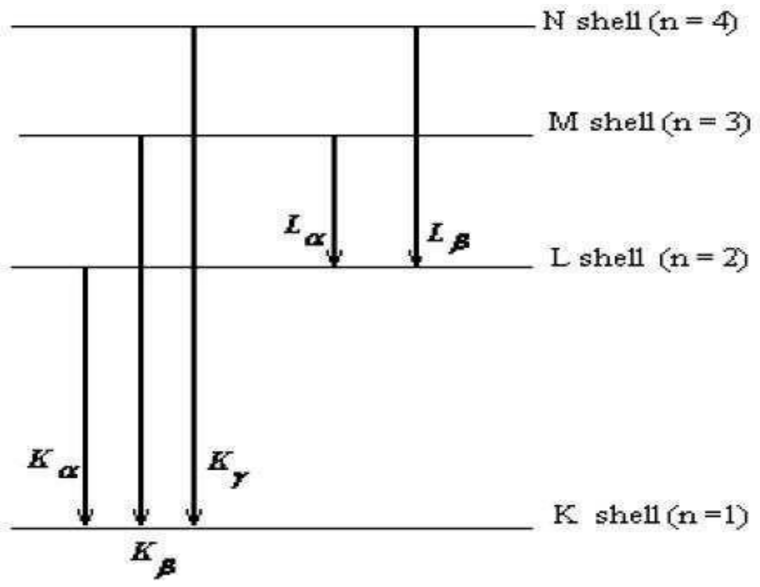


Fig (2.3):The transitions that produce the characteristics X-rays radiations

Not all transitions are allowed. Only those transitions which fulfill the following selection rule are allowed$\Delta\lambda = \pm 1$.

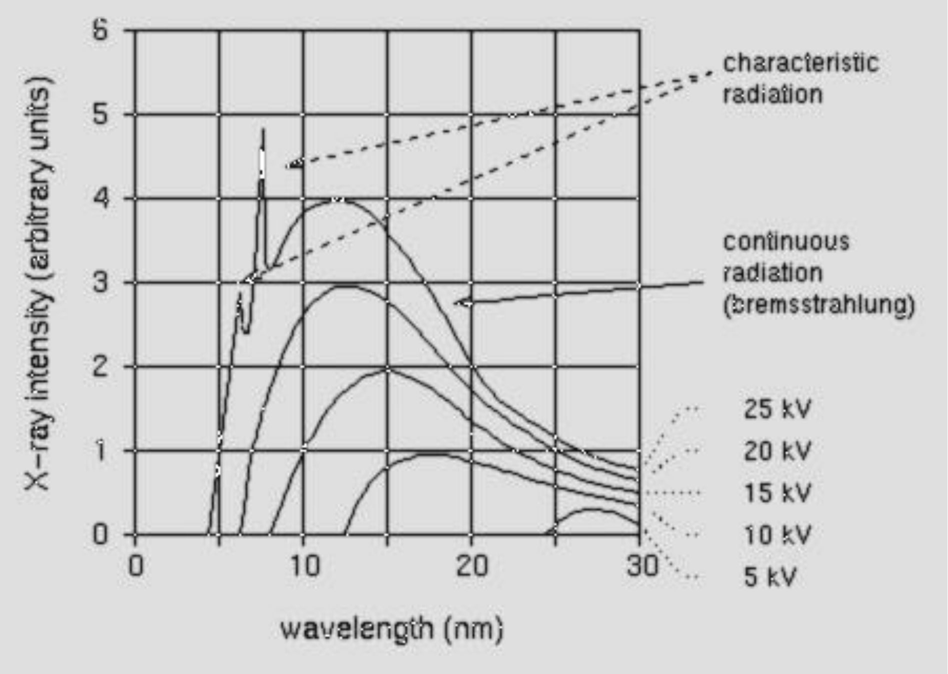


Fig (2.4): The graph shows the following features

- A continuous background of X-radiation in which the intensity varies smoothly with wavelength the background intensity reaches a maximum value as the wavelength increases, and then the intensity falls at greater wavelengths.
- Minimum wavelength which depends on the tube voltage. The higher the voltage the smaller the value of the minimum wavelength.
- Sharp peaks of intensity occur at wavelengths unaffected by change of tube voltage.

2.6: Absorption of X-rays

X-rays which enter a sample are scattered by electrons aroundthe nucleus of atoms in the sample. The scattering usually occurs in various different directions other than the direction of the incident X-rays,even if photoelectric absorption does not occur. As a result, the reduction in intensity of X-rays which penetrate the substance is necessarily detected.When X-rays with intensity$I_o$penetrate a uniform substance, the intensity$I$after transmission through distance x is given by:

$$I = I_o e^{-\mu x} \quad (2.6)$$

Here, the proportional factor μ is called linear absorption coefficient. Which is dependent on the wavelength of X-rays, the physical state (gas, liquid, and solid) or density of the substance, and its unit is usually inverse of distance. However, since the linear absorption coefficientμ is proportional to density ρ , (μ ⁄ρ)becomes unique value of the substance, independent upon the state of the substance. The quantity (μ ⁄ρ)of is called the mass absorption coefficient and the specific values for characteristic X-rays frequently-used are compiled. Equation (2.6)can be re-written as (2.7) in terms of the mass absorption coefficient.

$$I = I_o e^{-(\frac{\mu}{\rho})\rho x} \quad (2.7)$$

Mass absorption coefficient of the sample of interest containing two or more elements can be estimated from (2.8) using the bulk density,ρ, and weight ratio of w_j for each element j:

$$\left(\frac{\mu}{\rho}\right) = w_1 \left(\frac{\mu}{\rho}\right)_1 + w_2 \left(\frac{\mu}{\rho}\right)_2 + \cdots = \sum_{j=1} w_j \left(\frac{\mu}{\rho}\right)_j \quad (2.8)$$

Absorption of X-rays becomes small as transitivity increases with increasing energy(wavelength becomes shorter).However,if the incident X-ray energy comes close to a specific value (or wavelength) .the photoelectric absorption takes place by ejecting an electron in K-shell and then discontinuous variation in absorption is found. Such specific energy (wavelength) is called absorption edge. It may be added that monotonic variation in energy (wavelength) dependence is again detected when the incident X-ray energy is away from the absorption edge [9].

2.7:Filters

Many x-ray diffraction experiments require radiation which is as closely monochromatic as possible. However, the beam from an x-ray tube operated at a voltage above V_K contains not only the strong K_α line but also the weaker K_β line and the continuous spectrum. The intensity of these undesirable components can be decreased relative to the intensity of the K_α line by passing the beam through a filter made of a material whose K absorption edge lies between the K_α and K_β wavelengths of the target metal. Such a material will have an atomic number 1 or 2 less than that of the target metal. A filter so chosen will absorb the Kβ component much more strongly than the Kα component, because of the abrupt change in its absorption coefficient between these two wave lengths, in which the partial spectra of the unfiltered and filtered beams from a copper target (Z = 29) are shown superimposed on a plot of the mass absorption coefficient of the nickel filter (Z = 28). The thicker the filter works on the lower the ratio of intensity of Kβ to Kα in the transmitted beam. But filtration is never perfect, of course, no matter how thick the filter and one must compromise between reasonable suppression of the Kβ component and the inevitable weakening of the Kα component which accompanies it.

Table 1-1 shows the filters used in conjunction with the common target metals, the thicknesses required, and the transmission factors for the Kα line. Filter materials are usually used in the form of thin foils. If it is not possible to obtain a given metal in the form of a stable foil, the oxide of the metal may be used. The powdered oxide is mixed with a suitable binder and spread on a paper backing.

In practice it is found that a reduction in the intensity of the Kα line to about half its original value will decrease the ratio of intensity of Kβ to Kα from about 1\5 in the incident beam to about 1\500 in the transmitted beam ; this level is sufficiently low for most purposes [10].

2.8: Interaction of X-Rays with Matter

X-rays interact with matter in three main ways, through photoabsorption, Compton scattering, and Rayleigh scattering. The strength of these interactions depend on the energy of the X-rays and the elemental composition of the material, but not much on chemical properties because the X-ray photon energy is much higher than chemical binding energies. Photoabsorption or photoelectric absorption is the dominant interaction mechanism in the soft X-ray regime and for the lower hard X-ray energies. At higher energies, Compton scattering dominates.

2.8.1 Photoelectric absorption

The probability of a photoelectric absorption per unit mass is approximately proportional to Z^3/E^3, where Z is the atomic number and E is the energy of the incident photon. This rule is not valid close to inner shell electron binding energies where there are abrupt changes in interaction probability, so called absorption edges. However, the general trend of high absorption coefficients and thus short penetration depths for low photon energies and high atomic numbers is very strong. For soft tissue photoabsorption dominates up to about 26 keV, photon energy where Compton scattering takes over. For higher atomic number substances this limit is higher. The high amount of calcium (Z=20) in bones together with their high density is what makes them show up so clearly on medical radiographs.

A photoabsorbed photon transfers all its energy to the electron with which it interacts, thus ionizing the atom to which the electron was bound and producing a photoelectron that is likely to ionize more atoms in its path. An outer electron will fill the vacant electron position and produce either a characteristic photon or an Auger electron. These effects can be used for elemental detection through X-ray spectroscopy or Auger electron spectroscopy.

2.8.2 Compton scattering

Compton scattering is the predominant interaction between X-rays and soft tissue in medical imaging. Compton scattering is an inelastic scattering of the X-ray photon by an outer shell electron. Part of the energy of the photon is transferred to the scattering electron, thereby ionizing the atom and increasing the wavelength of the X-ray. The scattered photon can go in any direction, but a direction similar to the original direction is a bit more likely, especially for high-energy X-rays. The probabilities for different scattering angles are described by the Klein–Nishina formula. The transferred energy can be directly obtained from the scattering angle from the conservation of energy and momentum.

2.8.3 Rayleigh scattering

Rayleigh scattering is the dominant elastic scattering mechanism in the X-ray regime. Inelastic forward scattering gives rise to the refractive index, which for X-rays is only slightly below.

2.9: X-ray Detectors The two most common types of X-ray detector used in the laboratory for powder diffraction (excluding the case of X-ray film) are the scintillation and the gas-filled detectors, both of which are described below.

2.9.1: Scintillation Detectors

In the scintillation counter, the conversion of X-ray photons into an electrical signal is a two-stage process. The X-ray photon collides with a phosphor screen, or scintillator, which forms the coating of a thallium-doped sodium iodide crystal. The latter produces photons in the blue region of the visible spectrum. These are subsequently converted to voltage pulses by means of a photomultiplier tube attached directly behind the scintillator. The number of electrons ejected by the photocathode is proportional to the number of visible photons which strike it, which in turn is proportional to the energy of the original X-ray photon.Due to a large number of losses, the energy resolution of the detector is poor, and as such it cannot be used to resolve X-ray photons due to Kα and Kβ radiation. However, it has a very high quantum efficiency and a very low dead time making it the ideal detector for the point intensity measurements required for step-scanning diffractometers.

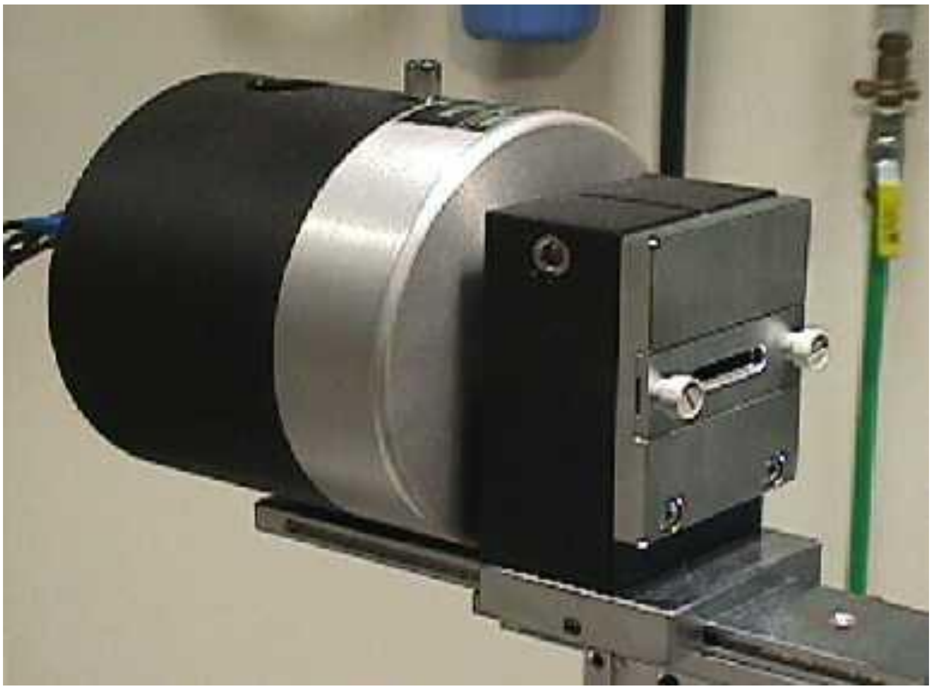


Fig (2.5): show the Scintillation Detectors

2.9.2: Gas-filled Detectors

The second type of detector commonly used in the laboratory is the gas-filled detector. This detector works on the principle that X-ray photons can ionize inert gas atoms such as argon or xenon into an electron (e-) and ion (e.g. Ar+) pair. The ionization energy required to eject an outer electron is low (10-20 eV) compared to the energy of the X-ray photon (8 keV) so that one X-ray photon can produce several hundred ion pairs. A wire placed inside the detector is set to a potential of about 1,000 V. This accelerates the electrons of the ion pair towards the wire causing further ionization and an enhanced signal by gas amplification. The burst of electrons on the wire is converted into a voltage pulse which is then shaped and counted by the electronics. In order to minimise the dead time of the system, a quenching gas such as methane (CH_4) is mixed with the inert gas (e.g. 90% Ar: 10% CH_4)[15].

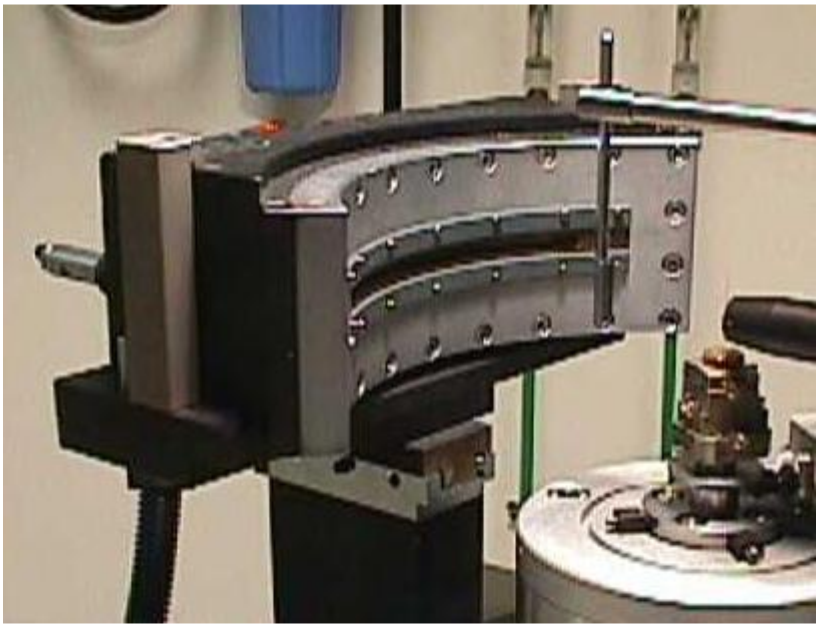


Fig (2.6): Gas-filled Detectors

Chapter three

X-Ray Diffraction

Diffraction refers to various phenomena which occur when a wave encounters an obstacle or a slit. It is defined as the bending of light around the corners of an obstacle or aperture into the region of geometrical shadow of the obstacle [5]. In classical physics, the diffraction phenomenon is described as the interference of waves according to the Huygens–Fresnel principle. These characteristic behaviors are exhibited when a wave encounters an obstacle or a slit that is comparable in size to its wavelength. Similar effects occur when a light wave travels through a medium with a varying refractive index, or when a sound wave travels through a medium with varying acoustic impedance. Diffraction occurs with all waves, including sound waves, water waves, and electromagnetic waves such as visible light, X-rays and radio waves. Since physical objects have wave-like properties (at the atomic level), diffraction also occurs with matter and can be studied according to the principles of quantum mechanics. Italian scientist Francesco Maria Grimaldi coined the word "diffraction" and was the first to record accurate observations of the phenomenon in 1660.

3.1:Diffraction of x-ray

X-ray diffraction is a tool for the investigation of the fine structure of matter. This technique had its beginning in Von Laue’s discovery in 1912, that crystals diffract X-rays, the manner of the diffraction revealing the structure of the crystal. Using this technique, a clear cut idea can be obtained about the arrangement and spacing of atoms in crystalline materials. X-ray diffraction has provided a wealth of important information to science and industry [10].By the X-ray crystallographic studies the determination of unit cell dimensions, lattice type of crystal, the interplanar spacing of lattice planes and miller indices of the reflection planes are possible [11, 12]. Crystalline solids consist of regular arrays of atoms, ions or molecules with interatomic spacing's of the order of 100 pm. For diffraction to take place, the wavelength of the incident light has to be of the same order of magnitude as the spacing's of the grating. Because of the periodic nature of the internal structure, it is possible for crystals to act as a three-dimensional diffraction grating to light of a suitable wavelength.Diffraction patterns are obtained by automatic scanning (Scattering angle ($2\theta = 0\sim10$ deg).X-ray diffraction is the only convenient and hence widely used physical procedure for the complete determination of molecular structure. Crystallographic study of materials depends on the nature of crystals. Studies of crystal faces and their intercepts on crystal axes show that all known crystals can be classified into seven systems. Particular axial lengths and axial angles characterize each system. Depending on these values the crystals are classified as cubic, tetragonal, orthorhombic, trigonal, hexagonal, monoclinic and triclinic.This is obtained by allowing the X-rays of required wavelengths to fall on the crystal.The interference pattern will be created because of the scattering of these rays from neighboring atoms produces diffraction, where this diffraction obeys to the Bragg's law.

3.2: Bragg's law

W.L.(Lawrence) Bragg noted that X-ray diffraction behaves like 'reflection' from the planes of atoms within the crystal and that only at specific orientations of the crystal with respect to the source and detector are X-rays 'reflected' from the planes. It is not like the reflection of light from a mirror, as this requires that the angle of incidence equals the angle of reflection, and this is possible for all angles. With X-ray diffraction,the reflection only occurs when the conditions for constructive interference are fulfilled [13].Therefore the condition for diffraction at any observable angle 2θ is

$$\lambda \leq 2d \qquad (3.1)$$

Figure (3.1) illustrates the Bragg condition for the reflection of X-rays by a crystal. The array of black points in the diagram represents a section through a crystal and the lines joining the dots mark a set of parallel planes with Miller indices $hkl$and interplanar spacing d_{hkl}.

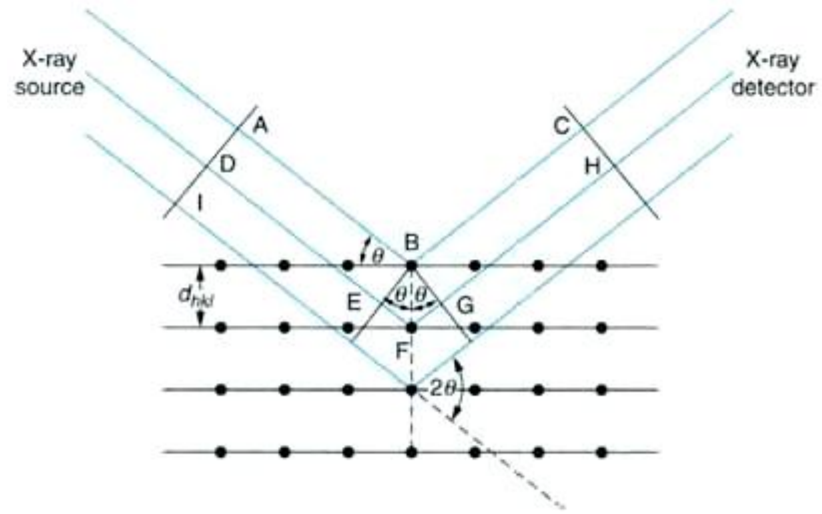


Fig (3.1):Bragg reflection from a set of crystal planes with a spacing d_{hkl}

A parallel beam of monochromatic X-rays ADI is incident to the planes at an angleθ_{hkl}. The ray A is scattered by the atom at B and the ray D is scattered by the atom at F. For the reflected beams to emerge as a single beam of reasonable intensity, they must reinforce, or arrive in phase with one another. This is known as constructive interference, and for constructive interference to take place, the path lengths of the interfering beams must differ by an integral number of wavelengths. If BE and BG is drawn at right angles to the beam, the difference in path length between the two beams is given by:

Difference in path length$= EF + FG$

But

$$EF = FG = d_{hkl} \sin \theta_{hkl} \qquad (3.2)$$

So

Difference in path length $= 2d_{hkl} \sin \theta_{hkl}$ (3.3)

This must be equal to an integral number, n, of wavelengths. If the wavelength of the X-rays is λ, then

$$n\lambda = 2d_{hkl} \sin \theta_{hkl} \qquad (3.4)$$

This is known as the Bragg equation, and it relates the spacing between the crystal planes, d_{hkl}, to the particular Bragg angle,θ_{hkl}at which reflections from these planes are observed (mostly the subscript hkl is dropped from the Bragg angle θ without anyambiguity as the angle is unique for each set of planes).

When n=1, the reflections are called first order, and when n=2 the reflections are second order and so on. However, the Bragg equation for a second order reflection from a set of planes hkl is

$$2\lambda = 2d_{hkl}\sin\theta_{hkl} \qquad (3.5)$$

which can be rewritten as

$$\lambda = 2\frac{d_{hkl}}{2}\sin\theta \qquad (3.6)$$

Equation 2.3 represents a first order reflection from a set of planes with interplanar spacing $\frac{d_{hkl}}{2}$The set of planes with interplanar spacing$\frac{d_{hkl}}{2}$has Miller indices $2h\ 2k\ 2l$.Therefore, the second order reflection from hkl is indistinguishable from the first order reflection from $2h\ 2k\ 2l$, and the Bragg equation may be written more simply as

$$\lambda = 2d_{hkl}\sin\theta \qquad (3.7)$$

3.3: Determination of crystal system

In the study of crystal systems, the relationship between the interplanar space, d and Miller indices h, k, l are used. Crystals belonging to regular or cubic system are built up on three equal axes at right angles. In this case axial lengths a = b = c and axial angles $\alpha = \beta = \gamma = 90°$. In this case the relation d and Miller indices (h, k, l) is given as

$$\frac{1}{d^2} = \frac{h^2+k^2+l^2}{a^2} \qquad (3.8)$$

$$d^2 = \lambda^2/4sin^2\theta$$

$sin^2\theta = \frac{\lambda^2(h^2+k^2+l^2)}{4a^2}$(3.9)

(h^2 + k^2+ l^2) will be constant. On measuring Bragg angles, the values of $Sin^2\theta$ will be obtained. It is found to be an integral multiple of λ^2 / $4a^2$ which is a constant.

For a hexagonal system a = b ≠ c and α = β = 90°, γ = 120°, $Sin^2\theta$ in this case is

$sin^2\theta = \lambda^2/3a^2(h^2 + hk + k^2) + (\lambda^2 l^2)/4c^2)$(3.10)

Density and number of molecules per unit cell of the complex have been calculated using the formula

$D = nM/VN$(3.17) (3.11)

Where D is the density of the complex, n the number of molecules in the unit cell, N is the Avogadro number, V is volume of unit cell and M is the molecular mass of the complex. The relative intensity of each peak can be Calculated using the equation, 100 (I/I_0), where I is the intensity of diffracted beam and I_0 is the intensity of the incident beam.

3.4 Diffraction methods

Diffraction can occur whenever the Bragg law $\lambda = 2dsin\theta$, is satisfied.This equation puts very stringent conditions on λ and θ for any given crystal. With monochromatic radiation, an arbitrary setting of a single crystal in a beam of x-rays will not in general produce any diffracted beams. Some way of satisfying the Bragg law must be devised, and this can be done by continuously varying either λ or θ during the experiment. The ways in which these quantities are varied distinguish the three main diffraction methods:

diffraction methods		
Laue method	Variable	Fixed
Rotating-crystal method	Fixed	Variable
Powder method	Fixed	Variable

3.4.1: Laue method

The Laue method was the first diffraction method ever used, and it reproduces von Laue's original experiment. A beam of white radiation, the continuous spectrum from an x-ray tube, is allowed to fall on a fixed single crystal. The Bragg angleθ is therefore fixed for every set of planes in the crystal, and each set picks out and diffracts that particular wavelength which satisfies the Bragg law for the particular values of d and θ involved. Each diffracted beam thus has a different wavelength.

3.4.2: Rotating-crystal method

In the rotating-crystal method a single crystal is mounted with one of its axes, or some important crystallographic direction, normal to a monochromatic x-ray beam. A cylindrical film is placed around it and the crystal is rotated about the chosen direction, the axis of the film coinciding with the axis of rotation of the crystal.

3.4.3: Powder method

Powder diffraction is a scientific technique using X-ray, neutron, or electron diffraction on powder or microcrystalline samples for structural characterization of materials. [5] An instrument dedicated to performing such powder measurements is called a powder diffractometer. A finely ground crystalline powder contains a very large number of small crystals, known as crystallites, which are oriented randomly to one another. If such a sample is placed in the path of a monochromatic X-ray beam, diffraction will occur from planes in those crystallites which happen to be oriented at the correct angle to fulfill the Bragg condition.

The diffracted beams make an angle of 2θ with the incident beam. Because the crystallites can lie in all directions while still maintaining the

Bragg condition, there flection's lie on the surface of cones whose semi-apex angles

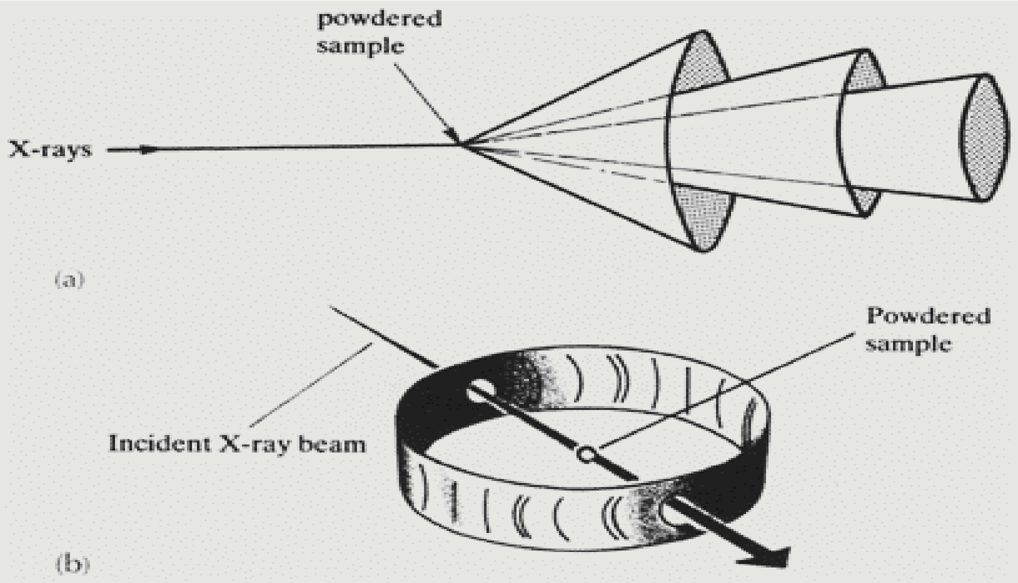


Figure (3.2) :(a) Cones produced by a powder diffraction experiment; (b) experimental arrangement for a Debye-Scherrer photograph.

are equal to the deflection angle 2θ (Figure (3.2.a). In the Debye-Scherrer photographic method, a strip of film was wrapped around the inside of a X-ray camera (Figure (3.2.b) with a hole to allow in the collimated incident beam and a beam stop to absorb the undiffracted beam. The sample was rotated to bring as many planes as possible into the diffracting condition, and the cones were recorded as arcs on the film. Using the radius of the camera and the distance along the film from the

Centre, the Bragg angle 2θ, and thus the d_{hkl} spacing for each reflection can be calculated. Collection of powder diffraction patterns is now almost always performed by automatic diffractometers, using a scintillation or CCD detector to record the angle and the intensity of the diffracted beams, which are plotted as intensity against 2θ. The resolution obtained using a diffractometer is better than photography as the sample acts like a mirror helping to refocus the X-ray beam [14]. The data, both position and intensity, are readily measured and stored on a computer for analysis. A diffraction pattern also be used to determine and refine the lattice parameters of a crystal structure.The particle size of the powder can also be determined by using the Scherrer formula, which relates the particle size to the peak width.

The Scherrer formula is

$$t = \frac{0.9\lambda}{\sqrt{\beta_M^2 - \beta_S^2} \cos\theta} \tag{3.18}$$

Where λ is the x-ray wavelength, B_M is the observed peak width, B_S is the peak width of a crystalline standard, θ is the angle of diffraction.

Chapter four

Result and discussions

4.1: ExperimentalTechniques

In order to achieve the objectives of this project, certain experimental techniques were used. The sample was characterized using X-ray diffraction (XRD).Our discussion here is only limited to samples in powdered form with many randomly arranged crystals.As well known, a crystal consists of set of highly ordered parallel planes with specific spacing between them. These separations act as diffraction slits to incident x-ray beams. The diffracted beams will interfere either constructively or destructively depending on whether the path difference between them is an integer or half odd integer multiple of the X-ray wavelength (λ), respectively. If the bath differences between these two extreme cases, there will exist beams with lower amplitude. These beams are cancelled from beams from other planes. This cancellation will occur as far as there are many planes. However in a very fine crystallite (i.e. the domain of the solid that has the same structure as a single crystal [], this cancellation will not occur for angles near Bragg's angles, at which constructive interference occur, and broadening will be noticed in the peaks. Hence there is a connection between the size of a crystallite and this broadening. This connection can be formulated by an equation called Scherrer formula as mentioned in **section [3]** .When the constructive x-ray beams are accumulated and detected using a diffractometer, they will produce the finger-print x-ray pattern that is characteristic to the structural composition of the material. Hence, the x-ray diffraction pattern can provide useful information about the crystal structure and the crystallite size of the material.

In our work, X-ray diffraction (XRD) patterns were obtained using a standard Philips diffractometer and scintillation counter with an exit beam of. Cu-k_α (λ=1.5406 Å) radiation was used in 10^0 - 100^0, 2θ- range in steps of 0.02^0 and counting time of 0.04s per step. The resulting XRD patterns were then analyzed using the “Winfit” software[16]. Another software “powdercell”[17] of a chosen theoretical crystallographic structure to fit the experimental obtained XRD pattern.

4.2: Results and discussions

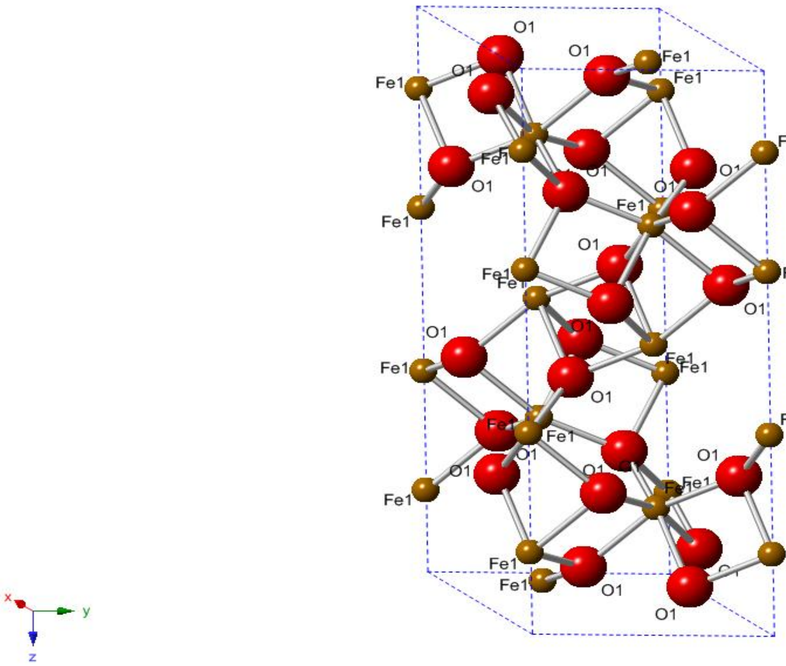


Figure 4.1: shows the structure of Fe_2O_3

Fig 4.2 shows the XRD pattern of Fe_2O_3, the intensity and peak position are in aagreement with the theoretical pattern

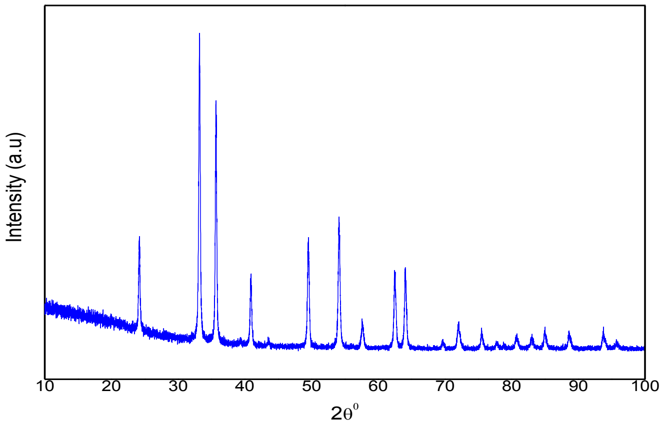


Fig 4.2: shows the XRD pattern of Fe_2O_3

Table 4.1 summarizes the results of XRD for the Fe_2O_3 sample. The value of the lattice parameter of the Fe_2O_3 sample is in agreement of those reported in literature.

Table 4.1: shows the Positions of the Fe_2O_3 atoms

No	name	crystal coordinates			Cartesian coordinates		
		X	Y	Z	x	y	z
1	Fe	0.0000	0.0000	0.3553	-1.2595	-2.1816	-1.9928
2	O	0.3059	0.0000	0.2500	0.2816	-2.1816	-3.4430

Table 4.2: show the angle between the atoms in Fe_2O_3 compound

No	atom1	atom2	atom3	Angle
1	O	Fe	O	78.2473
2	O	Fe	O	90.4960
3	Fe	O	Fe	86.4599
4	Fe	O	O	50.8764
5	Fe	O	O	92.8522
6	Fe	O	Fe	131.5694
7	Fe	O	O	114.7309
8	Fe	O	O	170.2638
9	Fe	O	O	42.3778
10	Fe	O	O	130.8739

Table 4.3: show the bond length between tow atoms

No	atom1	atom2	Distance	quant
1	Fe	O	2.1161	1

Table 4.4: shows the lattice parameters obtained using Powdercell program.

a/A	b/A	C/A	α	β	γ
5.0305	5.0305	13.7380	90	90	120

It is evidence from all results mentioned above the Fe_2O_3compound has hexagonal crystalline structure.

Conclusion

The structural properties of Fe_2O_3(99.9%) compound have been Studied using diffraction phenomena. The X-Ray data was refined by two software program (Win-fit and powdercell method) in order to find the lattice parameters of this compound. The crystal structure of $Fe_2O_3$was found to hexagonal with lattice parameters a=5.0305/A, b=5.0305/A^o, and c=13.7380/ A^o, and the angle are α=90, β=90 and γ=120^o.In Feature we can obtain on the other properties of this compound and used in different applications.

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