Consortium for Educational Communication

Module on

X-ray spectroscopy (XRS)

By

Afshan Hamdani

Department of Food Science and Technology,

University of Kashmir, Srinagar

Text:

Introduction to X-ray:

X rays are short-wavelength electromagnetic radiations that can undergo various interactions with matter. Such interactions yield data which, when appropriately analyzed, can provide useful information on the materials irradiated. Their discovery dates back to 1895 when Roentgen working in dark German laboratory discovered X-rays accidentally, while doing some basic research in physics using a cathode ray tube (like in the back of your TV or computer monitor). He found the device he was using produced X-rays and by doing basic scientific research on it with no specific practical goal, Wilhelm Roentgen discovered one of modern medicine's most useful diagnostic tools and won the first Nobel Prize in physics in 1901.

X-rays exhibit several properties that make these radiations suitable for wide range of applications from medical science to industry. These include their non-charged behaviour, the property of travelling in straight lines with the speed of light and a tendency to induce fluorescence in the materials like calcium tungstate and zinc sulphide. In addition, the specific properties of X-rays with respect to their application, is their high penetration power which gives them an ability to penetrate the solid materials of considerable thickness and the property that these can expose photographic films. However, depending upon the material densities the power of penetration is seen to vary. Their short wavelength allows high resolution imaging if appropriate optical components are available.

Theory of production of X-rays:

The process of production of X-rays can be understood with the help of an example. It is assumed that an electron travelling at a very high velocity strikes an atom. Since, it has high energy; the electron can overcome the negative charge due to the electrons around the nucleus and penetrate into the inner levels of the atom. During this process, electron will lose some of its energy as it requires work to push through the negatively charged field. However, if the electron has sufficient energy so that it is not repelled, it may transfer a part of its remaining energy to an electron in the K-shell around the nucleus causing it to be ejected. This creates a vacancy in a very low energy level that electrons from the other orbitals will try to fill. That is, after the removal of an inner electron by an energetic photon provided by a primary radiation source, an electron from an outer shell drops into its place. There are a limited number of ways in which the process can occur, the main transitions being an L□K called K_α transitions or M□K transition is called K_β. However, since L-shell electrons are most readily available, they usually succeed in filling this vacancy. When this L-electron drops from its higher energy which is a less stable position to lower energy which is a more stable position, it loses a large amount of energy. Similarly, if the original electron entering penetrates the L-shell, the X-rays produced will be called as L X-rays, an M□L transition is called L_α while as N□L transition is called L_β and so on. Furthermore, since each element has different arrangement of electrons about its nucleus and thus with a different set of energies, each element emits X-rays that are characteristic to that particular element in terms of wavelength and energies. X-rays are generated by following methods:

- Bombarding metal target with a beam of high energy electrons.
- Exposing a substance to a primary beam of X-rays to generate a secondary beam of X-rays of lower energy, whenever required.
- Using a radioactive source emitting X-rays during its decay process.
- From a synchrotron radiation source.

Interaction of X-ray radiations:

- Production of white radiations:

When a high-energy electron beam is incident upon a specimen, the interaction with the sample leads to the emission of a broad-wavelength band of radiation called continuum or white radiation. This white radiation is produced due to the deceleration of the impinging high-energy electrons by the atomic electrons of the elements making up the specimen. Most commercially available spectrometers utilize a sealed X-ray tube as an excitation source that contains a source of electrons and, an anode. The broad band of white radiation produced by this type of tube is ideal for the excitation of the characteristic lines from a wide range of atomic numbers. In general, the higher the atomic number of the anode material, the more intense the beam of radiation produced by the tube.

- Photoelectric effect:

In addition to electron interactions leading to the production of white radiation, there are also electron interactions which produce characteristic radiation. If a high-energy particle, such as an electron, strikes a bound atomic electron, and the energy of the particle is greater than the binding energy of the atomic electron, it is possible that the atomic electron will be ejected from its atomic position, departing from the atom with a certain amount of kinetic energy. Where the excitation causing particles are X-ray photons, the ejected electron is called a photoelectron and the interaction between primary X-ray photons and atomic electrons is called the photoelectric effect. As long as the vacancy in the shell exists, the atom is in an unstable state and can regain stability by transference of an electron from one of the outer orbitals to fill the vacancy.

- Absorption Factor

During their transit through matter x-rays suffer from an attenuation of intensity caused by their absorption. The Beer-Lambert, well known from optics, is used to describe this absorption effect. The intensity of X-rays say 'I_0' that enter into the sample will be exponentially damped to an amount equal to I_0 exp (−2lμ) after travelling a path of '2l' through the sample. The parameter 'μ' is named the linear attenuation coefficient and depends upon the wavelength of the radiation used, the chemical composition of the sample and its density. The observed variation in the scattered intensity is one of the dominant effects of the absorption factor on a diffraction pattern.

X-ray instrumentation:

The major parts of the X-ray instrumentation include an X-ray source, a sample holder, an X-ray monochromator and a detector. A common X-ray source generates X-rays by bombarding a heavy metal target with high energy electrons. The choice of heavy metal controls the range of energy of the emitted X-rays. For instance, a tungsten (W) target produces higher energy X-rays than a silver (Ag) target. In general, the higher the atomic number of the anode material (target metal), the more intense beam of radiation is produced by the tube. Most of the energy that powers this bombarding process is lost as heat which necessitates the cooling of target electrode.

X-ray source:

The purpose of the X-ray source is to supply X-ray radiation to the sample so that either X-ray absorption or fluorescence experiments can be carried out. Most commercially available spectrometers utilize a sealed X-ray tube as an excitation source. These tubes typically employ a heated tungsten filament as cathode that acts as a source of electrons. It contains a heavy block of copper to which the metal target to be bombarded is either platted or embedded. Target metals include tungsten, chromium, copper, molybdenum, rhodium, iron, cobalt etc. These are adjusted in the form of a layer of pure metal that acts as the anode. X-ray generation by this process is, however, an inefficient process in which much of the energy is wasted in the form of heat. Due to this, the cooling of an X-ray source becomes necessary. Modern equipments employ highly sensitive transducers in which no cooling is necessary. Figure-10 given below gives a schematic representation of a cooling tube.

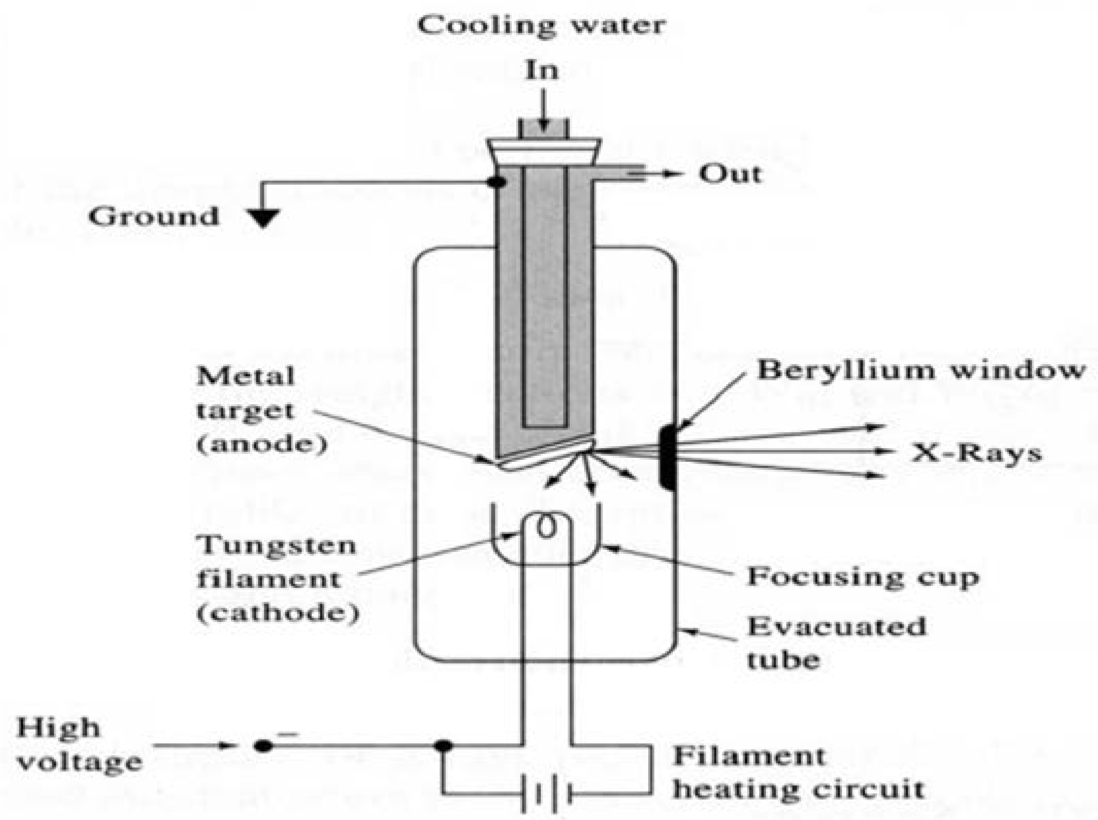

Fig. 10: Schematic diagram of a cooling tube.

X-ray monochromators:

The function of a monochromator is to produce a monochromatic beam of radiation. X-ray monochromators consist of a pair of collimators, one serving as slit and other as a dispersing agent. These collimators consist of a series of closely packed metal plates that absorb all the radiations except the parellel beams. Modern X-ray monochromators also employ a microprocessor controlled motors that help in automatic and independant driving of crystal and the detector devices, eliminating the use of gear based systems.

X-ray monochromators are sometimes used in combination with X-ray filters, particularly in the applications where X-ray tubes with narrow wavelength range are required. These are obtained by the use of filters and monochromators. One of the example of the filters is the zirconium filters shown in figure-11.

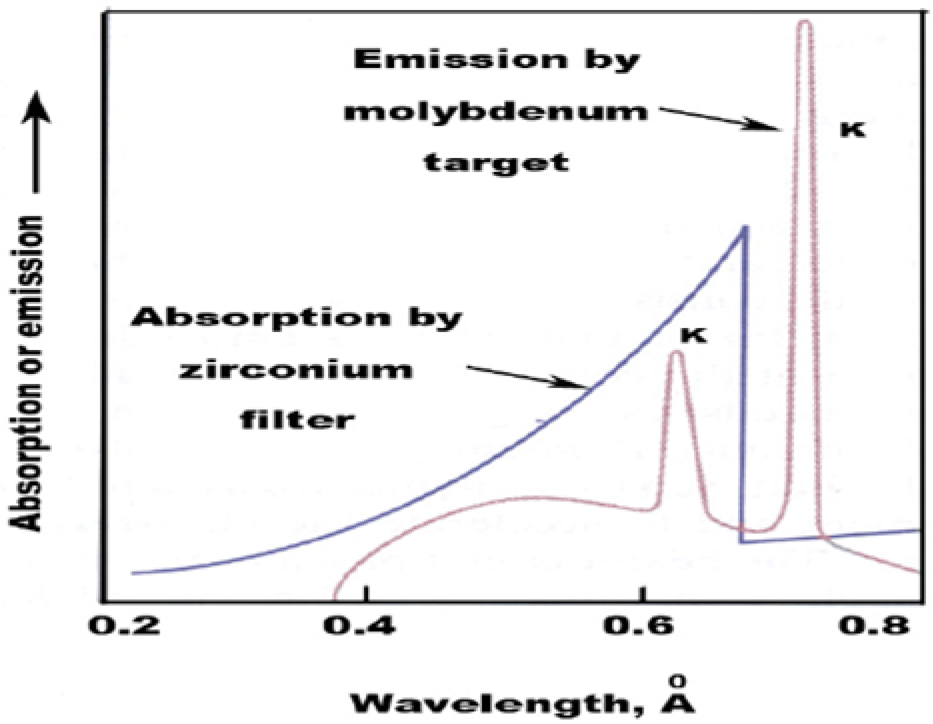

Fig. 11: Filters for X-ray

X-ray detector:

An X-ray detector is a transducer that converts X-ray photon energy into voltage pulses. These oftenly employ photo-cathodes that convert flashes of light into electrical pulse that can be amplified and counted. Detectors historically have been based on silicon semiconductors, in the form of lithium-drifted silicon crystals, or high-purity silicon wafers. These work on the principle of photo-ionization in which the entering X-ray photon interacts with the active detector material producing number of electrons. The current produced by these electrons is converted to a voltage pulse by a capacitor and a resistor. In this way, each entering X-ray photon produces a digital voltage pulse. An ideal detector should possess three important qualities:

- Sensitivity: It should be sensitive to the appropriate photon energies so that all the given range of wavelengths or energies is detected.
- Proportionality: The voltage pulse produced in the detector as a result of the interaction with the X-ray photon should be proportional to the energy of photon entering the detector.

- Linearity: It is a property in which the output pulses are produced at the same rate by a detector at which the X-ray photons enters the same.

Different types of X-ray techniques: Based on the properties of X-rays, a number of analytical methodologies have been developed over years, which include:

- X-ray emission spectroscopy (XES).
- Auger emission spectroscopy (AES).
- X-ray fluorescence spectroscopy (XFS).
- Electron spectroscopy (ES).
- X-ray absorption (XRA).
- X-ray diffraction spectroscopy (XRD).

X-ray absorption and emission are quite simple because they consist of very few lines of spectra. X-ray emission spectrum of an element may be obtained by using the sample itself as the target element, however, it is not convenient for all type of samples.

Applications of X-rays:

- Food industry applications:

The ability of X-rays to traverse through matter gives them the tendency to reveal hidden contaminants or defects. This has led to their extensive use in manufacturing industries for quality control inspection. The technique has been successfully employed for the inspection of food products to detect defects and contamination.

- Luggage inspection for concealed foods:

X-rays are used for the detection of food items concealed within a container, generally luggage. This application of the X-rays is particularly important and crucial to protect agricultural crops from invasion by foreign pests. Research efforts have shown that automatic shape recognition software combined with dual energy imaging is an effective tool for detecting many concealed food products.

- Packaged foods:

X-ray inspection has traditionally been implemented to the greatest extent in the food packaging industry. Products that are packaged in bottles or cans are ideally suited for high speed X-ray inspection. Such units routinely process and inspect more than 20 samples per second. The inspection using X-rays is highly suitable for such products as there is the possibility of contamination of the product by metals, plastics, glass, bone fragments, etc. in the processing plant environment. An advantage of X-ray analysis over the metal detector equipment is their ability to detect all kinds of contaminants.

- Poultry inspection:

Poultry inspection is another segment of the food industry that employs X-ray inspection on a routine basis. The inclusion of greatest interest in this field is the detection of bone fragments in the product that are often left behind after the de-boning process. X-ray systems have been also found effective for detection of heavier contaminants such as metal or rock, detection, but the detection of the softer material is usually hampered in this process due to the irregular shape and non-uniform thickness of the product.

- Grain inspection:

X-ray analysis is widely employed for the grain inspection. Most of this research effort has been devoted to the problem of insect infestation in wheat kernels. Although being very successfully for this application, bulk grain is still not routinely inspected using X-ray. The reason for this is the inability of current high speed X-ray systems to detect larvae at their earlier stages and the size of grain kernels that exceed the inspection speeds beyond the capability of even the fastest computers.

- Apples:

X-ray imaging of apples has been used for the detection of codling moth damage, watercore disease and core rot in apples. Detection of codling moth larvae in apples has been investigated using CT (computer tomography), film, as well as linescan X-ray systems. High quality images can be obtained using film and CT, but the techniques cannot be used for the bulk inspection. Watercore disease is a physiological disorder wherein fluid accumulates around the vascular bundles of the fruit, leading initially to sweetening but eventually to core rot. Efforts to detect watercore have also been reported using CT, film and systems. Recognition of the disease is based on darkening of the affected areas in X-ray images, presumably because of fluid filling. Results are sketchy, with severe cases easily identifiable.

- Tree nuts:

X-ray imaging has been used for inspection of pistachio nuts infested by the naval orange worm (NOW). Works conducted so far have achieve 98% recognition with less than 1% false positives in which good products were classified as bad on scanned film images. Similar technique is expected to be extrapolated to other products like almonds for the detection of NOW and the burrowing activity of the Pecan weevil.

- Other food products:

More limited research has been reported on a wide variety of food products. CT imaging has been used to determine maturity in tomatoes, monitor internal fruit changes in peaches during ripening, detection of core breakdown in pears and Woolly breakdown in nectarines. Linescan imaging has proven effective for detecting voids and rot in onions with more than 90% accuracy, as well as insect infestation in guava fruit. Other detection studies conducted using X-rays include inspection of seed weevil in mango, translucency in pineapple, hollow heart in potato, insect infestation in peaches and measure the micro-structure of meat emulsions.

- Other applications:

i. Elemental analysis: Certain areas of elemental analysis are still unique to X-ray fluorescence. Non-destructive X-rays fluorescence has been used for analysis of techtenium in solutions when currently no good chemical methods are available for the techtenium detection in different samples.

ii. Medical applications: In addition to the usual skeletal applications, radio isotope X-ray fluorescence methods are being used for analysis of biological samples such as those provided by biopsy of human aorta or tissue sections. X-ray employing a cadmium source is being used for analysis of tumor growth studies and studies involving atherosclerosis.

iii. Others: X-rays are used for the analysis of rock samples for detection of different elements. The technique is used for analysing alloy composition; presence of lead and bromine in aviation fuels; calcium, barium and zinc in lubricating oils; iron, copper and zinc in rice samples and pigments in plant samples.