J. Mountain Res. P-ISSN: 0974-3030, E-ISSN: 2582-5011 DOI: https://doi.org/10.51220/jmr.v19-i2.55
Vol. 19(2), (2024), 533-544

Insights Through Crystals: Exploring X-Ray Diffraction Techniques

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Received: 20.8.2024; Revised: 27.12.2024; Accepted: 28.12.2024

Abstract: X-ray diffraction (XRD) is a non-destructive analytical technique pivotal in unraveling the crystalline structure of materials. This paper explores the foundational principles of XRD, emphasizing its historical evolution, theoretical framework, and instrumentation, including X-ray sources, collimators, monochromators, and detectors. Key concepts such as Bragg's Law and the interference function are detailed to illustrate how diffraction patterns yield insights into atomic arrangements. The versatility of XRD across diverse fields—ranging from material science and pharmaceuticals to food technology and forensics—is underscored, demonstrating its role in determining crystallinity, phase identification, and structural refinement. The paper also highlights advanced methods, including the Laue and transmission techniques, which cater to specific research needs. While XRD offers unparalleled accuracy for crystalline materials, its limitations in analyzing amorphous substances, sample size requirements, and resolution constraints are discussed alongside potential solutions through complementary technologies like electron microscopy. This comprehensive review underscores XRD's indispensable role in modern research and industrial applications, emphasizing its adaptability through ongoing technological advancements. By bridging fundamental principles with practical applications, this paper provides a robust resource for understanding and leveraging XRD in scientific investigations.

Keyword: XRD • Bragg's Law • Instrumentation • Method • Application • Limitation

Introduction

Wilhelm Conard Roentgen found an image on November 8, 1895, that was cast from his cathode rays generated and extended well beyond the cathode rays' probable range (known as an electron beam). While studying cathode rays he noticed a mysterious glow from a fluorescent screen near his experimental set even though it was shielded. He termed these rays "X-rays" due to their mysterious nature L (Zolotoyabko *et al* 2014). Max Van Loue postulated in 1912, that a beam of x-rays moving through a crystal might cause diffraction which would result in the development of an image on a photographic plate positioned perpendicular to the X-ray path. This finding provided a fresh method for investigating the fine structure of materials. As a result, X-ray crystallization developed. A non-destructive technique called XRD can yield comprehensive details in about the material's chemical composition, crystal structure, and physical characteristics. One popular method for determining sample arrangement is X-ray diffraction (Seeck and Murphy 2015). The crystalline phases affect the diffraction pattern in an X-ray diffractometer. Its foundation is a crystalline sample with monochromatic X-ray constructive interference. An extensive range of materials including thin film coating, ceramic, solar cells, semiconductors, metals,

minerals, polymers, plastics, and medicine can be identified using x-ray diffraction, a non-destructive approach (Bunaciu *et al,* 2015). A crystal resilient to size, shape, degree of crystallinity stress nearly any other characteristic pertinent to the sample's fundamental structure can all be rapidly identified using XRD analysis. When electrically charged molecules with sufficient energy slow down they generate X-rays or smaller wavelength electromagnetic radiation. The electromagnetic waves generated by the X-ray scattering (XRD) method are collimated and directed toward a non-material sample. Interaction between the incoming rays and the sample creates a diffracted beam which is then detected, processed, and counted (Chatterjee *et al* 2010). Each phase of a material generates a distinctive diffraction pattern due to its specific chemistry and atomic arrangement. The resulting patterns are plotted by measuring the strength of diffracted rays that are scattered at various angles of the substances. The basic X-ray scattering (XRD) is a form of light with wavelength on the order of nanometer. Radiation from substances with structure at that length scale may disperse X-rays, creating an interference pattern with different strengths.

Objectives of the Study

This review comprehensively underscores XRD's vital role in modern research and industrial applications and emphasizes its application using ongoing technological advancements. The review focuses on the fundamental principles of XRD with its practical applications and provides a robust resource for understanding and leveraging XRD in scientific investigations.

Methodology

From databases like Scopus and PubMed, various studies were used to discuss the theory, instrumentation and application of XRD technique in various scientific investigations. The studies were systematically and critically discussed after planning the outline of the article. The efforts were made to keep the discussion concise and useful for larger groups of multidisciplinary researchers working on different scientific disciplines.

Observations and Discussion

The XRD technique's historical perspective, fundamental concepts to advancement of the technique with application in different scientific domains were extracted and systematically discussed as follows.

Theory of Diffraction

Diffraction effects arise when waves contact with periodic structures when the crystals' wavelength and periodicity are similar in magnitude. While it is simple to generate X-rays at wavelengths that correspond to crystal unit cell size, and electrons or for diffraction investigations on crystals, neutrons of the right energy can also be employed, the width of the atom is (1 Å), and unit cells have multiple Å in dimension. This denotes that crystals are micron-sized or bigger and are made up of billions of unit cells that exhibit long-range order due to their periodic repetition in all three dimensions (Bunaciu *et al* 2015). Crystalline materials differ from crystalline ones due to their short-range organization, like glasses. The X-ray diffraction diagram will show any alteration from perfect order because the "quality" of diffraction effects in XRD is heavily dependent on the rigorous and undisturbed periodicity of atoms. The theoretically limitless perfect crystal may deviate from even tiny crystallite sizes (Chatterjee *et al* 2010). Additional atom replacements are common in solid solutions, with small geometrical atom variations to best place due to within strain.

The atom form factor

X-rays are electromagnetic waves having wavelengths of 10 nm−10 pm. Most of the electrons generate a nearly spherical wave with a wavelength similar to the incident rays when those waves interact with an atom's charges because of its electrical fields (Fewster *et al* 2014). When considering the overall number of electrons in an atom, the amplitude of this outgoing wave is precisely proportional to that number. "Poor" X-ray

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scatterers are light elements with few electrons, such as carbon or oxygen; "good" scatterers are heavier elements, such as lead. On detection limits, this effect has a major influence. The atom form ratio (f) defines the amplitude of such a distributed wave. The scattering angle affects the amplitude of the outgoing wave and, consequently, the atom form factor because of interference within individual atoms especially larger ones (2θ).

Bragg's Law

Through the use of X-rays, XRD identifies the arrangement or form of a molecule. Techniques for X-ray resolution are based on the elastic scattering of X-rays from structures with length-range order. Crystals exhibit diffractive effects on X-rays due to the similarity between their wavelength and the interatomic spacing. A crystal's regular three-dimensional atomic arrangements cause some X-ray beams to constructively interfere, producing the distinctive X-ray diffraction pattern that is used to determine the crystal's structure. These reinforced diffracted X-rays behave as though they were "reflected" from a family of planes within the crystal that was subsequently named after W.L. Bragg (Fig. 1). These atoms later form the rows that make up the crystal structure. A concept of diffraction termed Braggs Law was formulated by William Henry Braggs in 1915. According to the law, a crystal surface will reflect an equal angle of scattering (θ) when an x-ray is incident on it, according to the law "Constructive interference will occur when the path difference (d) equals a whole number (n) of wavelengths."

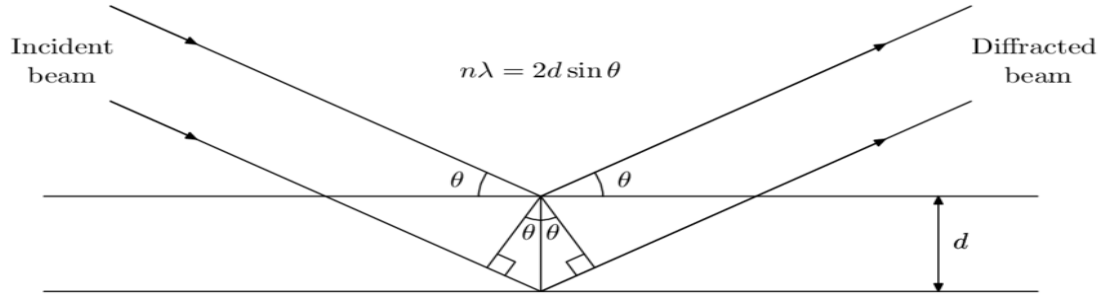

Fig. 1: Understanding Bragg's equation for X ray

Bragg's equation is expressed as nλ = 2d Sinθ; where, (d) is the interatomic space, (n) is the number of integers, (λ) is the wavelength of x-rays, (θ) represents the x-ray angle of incidence

Derivation of Braggs equation

In the Fig. 2, the beams coincide in phase when the incidence angle is equal to the reflecting angle. The incident beams are parallel to each other until they get to point (z). When they get to point z, they hit the surface and start to climb. At point B, the second beam disperses. The total distance traveled by the second beam is equal to AB plus BC. The extra distance is known as the integral multiple of the wavelength.

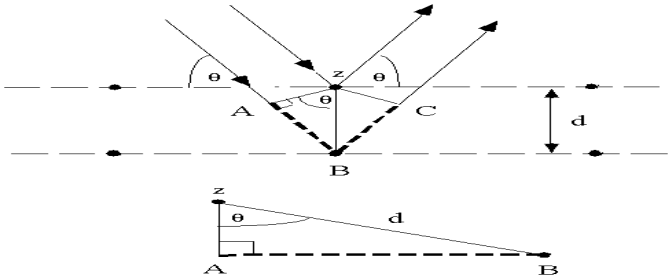

Fig. 2 Derivation of Braggs equation for X ray

nλ = AB + BC

also know that AB = BC

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for $n\lambda = 2AB$ (use equation 1)
The hypotenuse of the right triangle Abz is denoted by d. The angle θ opposite is AB.
According to equation 2,
$AB = d\sin\theta$
Substituting equation 2 in equation 1
$n\lambda = 2d\sin\theta$
The formula for Bragg"s law is given above.

The Interference function

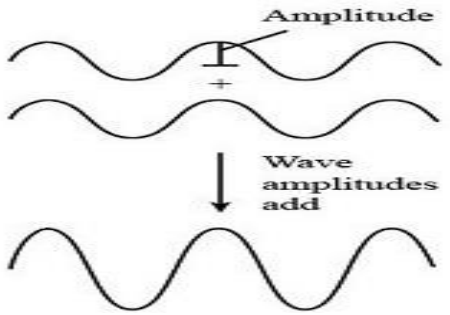
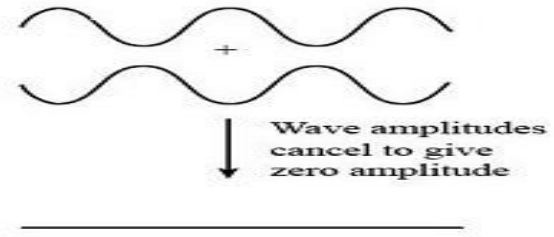


Fig. 3 (a) Constructive interference



(b) Destructive interference

One could argue that, if the phase delay is near λ, then even tiny departures from the ideal Bragg angle θ should result in a substantial intensity, indicating some but not all of the constructive interference occurring. This is usually the case when the crystals are appropriately small. Destructive interference arises from a place within a big crystal that results in a λ/2 phase delay when the phase delay is not exactly λ at an angle of 2θ. Sharp peaks are thus produced in big crystals when there is even a slight departure from the Bragg angle due to interference canceling. However, for tiny crystals, the peaks enlarge. The interference function models this crystal size effect.

$$\mathbf{S = (\sin\pi hN)^2 / N (\sin\pi h)^2}$$

Where (h) is the index of reflection and (N) is the total number of atoms in a unit cell in the crystal.

Limitation of Bragg's Law

1. Braggs law states that the structure of the crystal is symmetrical and can be extended forever. The majority of crystals have flaws, dislocations, and other imperfections that can alter the diffraction pattern.

2. Only crystalline materials are subject to Bragg's law. Analyzing amorphous materials or materials with extremely small crystallite sizes is not possible with it.

3. The incident X-rays wavelength has a significant impact on Bragg's law. Shorter wavelength X-rays can produce diffraction patterns with higher resolution, but they can also limit penetration depth into the sample and induce absorption effects.

4. Braggs law provides details about the crystal structure close to the sample surface; frequently below a few micrometers. This drawback limits the use of this technique to the investigation of unseen surfaces and layered systems.

5. Although diffraction angles can be easily calculated using Bragg's law, figuring out crystal structures from diffraction patterns can be challenging, particularly for materials with complicated crystal symmetries or numerous phases.

Instrumentation of X-ray diffraction

When crystalline substances are subjected to X-rays, interference arises between the wavelengths that the atom scatters. Depending on how the waves overlap, there are two types of interference (Fig. 3). In the same direction, two waves traveling can overlap and merge to create larger waves, a phenomenon known as constructive interference. Destructive interference happens when at their crest and trough, two waves traveling in an identical direction coincide.

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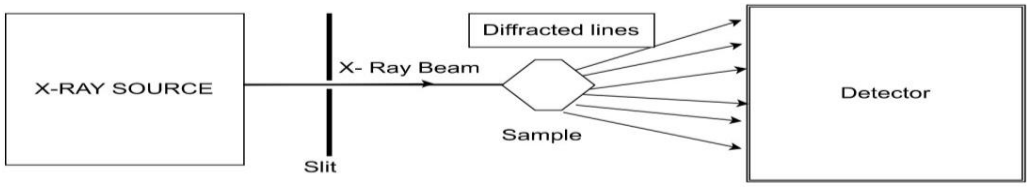

Fig. 4 Instrumental diagram of X-ray Diffraction

The instrumentation of XRD contains the following components (Fig. 4).

- Production of an X-ray source
- Collimation
- Monochromator – i) Filter ii) Crystal monochromator
- Detector – i) Photographic plate ii) Counter method

Production of X-rays:The acceleration of a charged particle produces X-rays (Fig. 4). In XRD electrons with high velocity strike a metal target and generate x-rays. The large accelerating voltage between the anode and cathode as well as the metal target (Cu, Al, Mo and Mg) and a hot W filament source. The desired target metal-containing block of Cu is the anode, which is cooled by water. Consequently, each X-ray tube needs to have

a) An electron source.

b) A voltage that accelerates quickly.

c) A target that is made of metal.

Each X-ray tube contains two electrodes: one that is usually maintained at the ground's potential, and another which is kept at a high negative potential to produce a diffraction pattern. This high potential is often between 30,000 and 50,000 volts. Any electrically charged particle with adequate kinetic energy can quickly slow down an X-ray. Typically, electrons are employed for this function.

Collimator: It's a device that makes waves or particle beams smaller. Narrowing can mean making the beam's spatial cross section smaller or making the motion more aligned in one direction (i.e., producing collimated lights or parallel rays) (Bunaciu *et al* 2015). The x-ray produced permits the desired substances to flow between closely spaced metal plates that are only slightly apart in order to produce a narrow, concentrated beam of radiation. This device absorbs all the X-rays except for the narrow beam that passes through the opening. The collimator narrows and parallelizes randomly directed X-rays. Pitches sizes vary from 400 microns to 6 microns.

Monochromator: This optical device transmits a limited band of light and other radiation wavelengths that may be mechanically selected from a greater range of wavelengths that are accessible at the point of entry (Cain 2020). The function of the monochromator is to filter the lights entering the spectrophotometer so that only certain wavelengths are permitted to pass through. Monochromatization is broadly classified into two categories;

a) Interference filter

b) Crystal monochromators

a) Interference filter: An appropriate filter can be inserted to partially monochromatize an X-ray beam. An absorber of unwanted radiation, a filter is a material window that lets through the necessary wavelengths of radiation. Interference filters are made of clear quartz or glass substrates with many optical layers put on them. The thickness of the optical layers determines the filter's unique performance. The aim of low energy is to be removed by subsequent filtration.

b) Crystal Monochromator: A Monochromator is made out of an appropriate crystalline substance that is placed inside the X-ray beam such that Bragg's equation may be used to satisfy the angle of reflection planes for the needed wavelength. The materials used to make monochromators include quartz, lithium fluoride, NaCl, and other materials.

Detector: These devices are used for measuring the spectrum, flux, and other properties of X-rays. The

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detectors split into two main categories: Various digitization technologies, such as imaging plates or flat, have largely superseded imaging detectors like photographic plates and x-ray film. These devices are known as counter techniques.

Types of Detectors

i) Photographic Film: The photographic film approach records the whole diffraction patterns on a single film, it is mainly used in diffraction research. A plane or cylinder of film is used to capture the location and strength of the x-ray beam. After being exposed to X-rays, the film is processed. Density unit D is used to express the developed film's blackening.

$D = \log I_0 / I$; where I_0 represents incident intensity, I stand for transmitted intensity and D which is the total energy that causing the film to blacken, is measured using a densitometer.

ii) Geiger Muller tube counter-argon or other inert gas fills the Geiger tube. The 800–1500V positive voltage of the center wire anode is maintained. An avalanche of electrons is produced and travels toward the center anode as a result of the electron being accelerated by the potential gradient and ionizing a large number of argon constituents.

iii) Scintillation Detector: Through the process of scintillation, these types of detectors transform x-ray photons into visible light. A significant quantity of sodium iodide crystal is activated in a scintillation detector with a relatively small amount of thallium. A photomultiplier tube detects the visible light pulses that are produced when an X-ray strikes a crystal. A scintillation detector is a valuable tool for measuring lower wavelength X-rays. Among the crystals used in scintillation detectors are phenol, sodium iodide, and anthracene as well as naphthalene. Although they are frequently found in older XRD instruments, scintillation detectors have generally been replaced by more advanced technologies because of their comparatively inferior sensitivity and resolution.

iv) Semi-conductor Detector: When x-rays fall on silicon lithium detector an electron and a hole. Pure silicon is made up of a thin film of lithium metal plated onto one end. The amount of x-ray intensity falling on a crystal is measured by the voltage generated.

Voltage of pulse = q / c. where c is the detector capacity and q is the total charge that is deposited on the electrode.

X-Ray Diffraction Method

There are several XRD methods which are generally used for investigating the internal structure and crystal structures of various solid compounds.

Laue Methods

The structure of crystalline materials can be studied using a method called the Laue method, which is sometimes referred to as Laue diffraction or Laue crystallography. It bears Max von Laue's name, who was awarded the Nobel Prize in Physics in 1914 for discovering it (Eckert 2012). In the Laue method, a single crystal is exposed to an X-ray beam (Fig. 5).

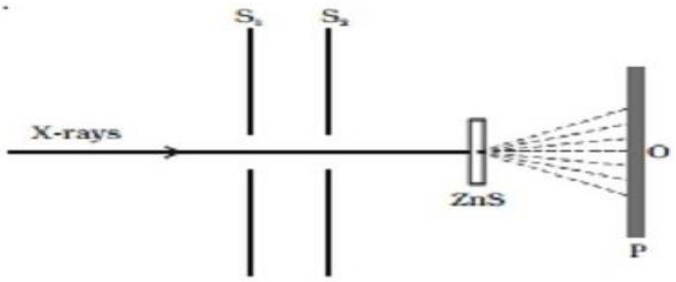

Fig.5 (a) Laue experimental setup

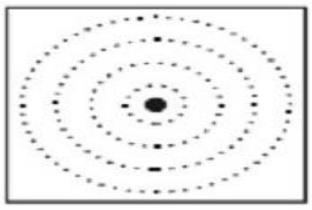

Fig. (b) Laue spot

White radiation, usually from an unfiltered X-ray tube, was used in this procedure. This lands on a solitary, stationary crystal. Thus, for each diffraction plane, the diffraction angle θ is fixed, and any

particular wavelength will be diffracted at that value of θ. Since the wavelength of the X-ray creating a particular region is unknown, this method is only helpful for confirming the orientation and quality of the crystal. It cannot be utilized for any quantitative study. The periodic arrangement of atoms in the crystal causes the X-rays to scatter in different directions when they interact with the crystal lattice (Eckert 2012). A digital detector or photographic film can be used to detect the diffraction pattern created by the dispersed X-rays. Information on the arrangement of atoms within the crystal lattice, including the distance between atomic planes and the crystal's orientation, can be found in the ensuing diffraction pattern. Scientists can ascertain the crystal structure, including the atomic locations, symmetry, and unit cell dimensions, by examining the diffraction pattern. Because the Laue method offers information on the complete crystal lattice in a single exposure, it is very helpful for figuring out the structure of big and complex crystals. In solid-state physics, chemistry, materials science, and structural biology, it is frequently utilized to investigate the atomic structure of various materials.

Transmission Laue Method

In this method, a thin film is placed behind the crystal to record the beams that pass through it. In the cone of Laue reflection, the transmitted beam defines one side (Fig. 6a). The location of the diffraction spots appears on an ellipse when the image reaches the cone. Since the film is flat, incident beam perpendiculars are partially transmitted through the sample and strike the film. Crystals can be oriented using it in solid-state investigations. Analyzing the symmetry of a single crystal is another use for it. The study of preferred orientation sheets, particularly those limited to lower diffraction angles, is well suited for it.

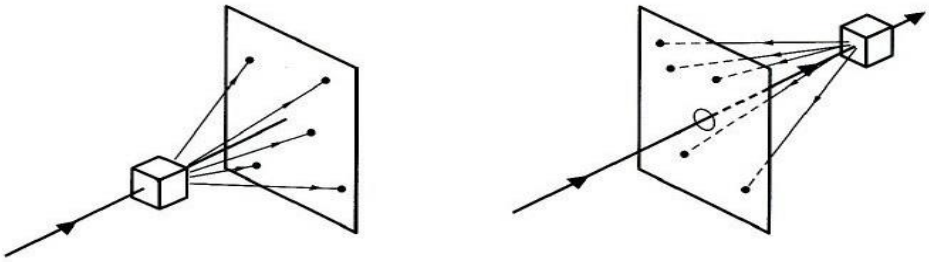

Fig. 6 Single crystal diffraction Laue method: (a) Transmission (b) back reflection Laue methods

Back Reflection Laue Method

The back reflection Laue method involves placing the materials in the crystal and the source of x-rays (Fig. 6b). Records are made of the fracturing beam in the reverse direction. The beam that is transmitted defines one side of the Laue reflection cone. Diffraction spots typically lie on a hyperbola, where the film meets with the cone. It is comparable to the transmission approach, but the only way to investigate thick and huge specimens is through back reflection (Eckert 2012).

Disadvantages of the Laue Method

a) The spots' positions permit one to identify the perspective of the crystal. Special charts may be used to index, or assign, each location to a specific plane.

b) This method is also helpful for measuring the size and shape of crystals to determine the quality of them.

c) A big crystal is required.

d) The Laue method is primarily applicable to single crystals. It cannot be used for polycrystalline or amorphous materials, which do not produce well-defined diffraction pattern.

Application of X-ray diffraction

a) XRD is used for analyzing thin films for application in coating, electronics, and optics and involves understanding their composition and

properties. It can ascertain the thin film layer's thickness and crystallinity once it is put on the substrate.

b) In the food sector, XRD helps produce products with consistent levels of sweetness, texture, and flavor. These properties are dependent on the ratio of crystalline to amorphous constituents, which can be readily observed by XRD. Among the most dependable techniques for assessing the powdered quality of dried fruit juices in XRD (Purohit *et al* 2019).

c) Phase identification is one of the most frequent methods used to evaluate the purity of a sample. It is possible to determine the presence of an impurity in a sample by comparing the diffraction pattern that was acquired from it with a reference pattern that is known. A pure sample should only show diffraction peaks matching to the phases that are expected, but impurities may add new peaks or change existing peaks.

d) atomic arrangement in a crystalline material. The atoms in the crystal lattice scatter X radiation when they get into touch with a sample of crystals. Bright areas known as diffraction peaks generate a diffraction pattern as a result of the dispersed X-rays interfering with one another. These peaks stand for particular angles and levels of constructive interference.

e) Polymorph screening, determining crystallinity, and characterizing medicinal molecules are all done in the pharmaceutical business using XRD. It contributes to preserving the effectiveness and stability of pharmaceuticals.

f) XRD is also used in forensic analysis to determine trace pieces of evidence such as minerals, crystals, and crystalline compounds found at crime scenes or on artifacts relevant to criminal investigations. By comparing the diffraction patterns of unknown substances to database of known drug structures, forensic scientist can determine the identity and purity of the substance.

g) The structures of biological substances, including lipids, proteins, and nucleic acids, are studied using XRD (Sayers *et al* 2017, Semalty 2014). In the case of proteins, X-ray crystallography has been particularly instrumental in determining their three-dimensional structures. Similarly, XRD has been used to study the structures of nucleic acid such as DNA and RNA, elucidating their double helix arrangements and interactions with proteins.

h) The XRD method is used to determine molecular structures, assess polymorphism (differing crystal structures of the same material), and investigate intermolecular interactions. In disciplines like organic chemistry and materials chemistry, this is especially crucial (Lee 2017).

i) In material science, XRD is widely frequently used to determine a solids crystal structure. This involves examining how atoms or molecules are arranged inside a crystal lattice, determining which phases are present in a sample, and examining flaws or imperfections in materials.

j) Insights into reaction processes, active sites, and catalyst stability can be gained by using XRD to examine the atomic structure of catalysts and catalytic reactions. Examining materials for energy conversion, storage, and environmental applications is another beneficial use of it (Lee 2017).

Limitations of XRD

a) Amorphous materials, lacking long-range order, do not form a characteristic diffraction pattern, making them challenging to analyze via XRD alone.

b) XRD typically requires a crystalline sample of sufficient size and uniformity. Small or poorly homogenized samples may produce weak diffraction pattern, impacting the accuracy and reliability of the analysis.

c) XRD may have limitations in detecting trace phases or components within a sample particularly when they are present in low concentration.

d) It might be difficult to identify peaks and restrict resolution when sample complexity or ones with very close crystal planes generate conflicting diffraction patterns.

e) Proper sample preparation is crucial for accurate XRD analysis. Inadequate sample preparation such

as improper grinding or mounting can introduce artifacts or alter the sample's crystalline structure leading to incorrect outcomes.

f) XRD provides valuable information about crystal structures; it does not always provide direct information about the arrangement of atoms within crystal lattices. Neutron diffraction or electron microscopy these techniques may be necessary for complete structural determination.

g) XRD primarily probes the bulk structure of the material. Surface effects such as roughness or contamination can influence the diffraction pattern and may require additional technique to characterize.

Role of XRD in the analysis of Crystallinity

The degree of crystallinity, crystal structure, type of crystalline regions, and lattice parameters are all determined by XRD analysis (Semalty *et al* 2010). These details aid in understanding the physical, chemical, and biological characteristics of each compound by correlating a chemical's structure to its function (Semalty 2014). When the diffraction pattern of the X-ray from the sample is established as a function of the scattering angle, the crystalline and amorphous nature of the sample can be determined by XRD analysis (Suryanarayanan and Rastogi, 1995). In a study, DTX-loaded nanoparticles were developed and characterized. The sharp peaks in pure DTX between 2◦and 20◦ showed the crystallinity of DTX, but in nanoparticles, DTX changed to an amorphous form with no clear peaks or hump observed from 10 to 22◦ (Kulhari *et al* 2014). In another study, the X-ray analysis showed that it has clear crystal patterns, EC has an amorphous nature, and At-NPs have some crystal features of At but with less intensity, indicating that Act's crystal structure changes to an amorphous structure when made into nanoparticles (Shaker *et al* 2021). The XRD patterns of a study by Muthu and Singh showed that risperidone, PCL, and Pluronic F-68 all had sharp peaks, indicating their crystalline nature. In the risperidone-loaded PCL nanoparticles, the peaks for risperidone were less intense, suggesting it had become less crystalline and more amorphous. Mixing risperidone with Pluronic F-68 also reduced its crystallinity, changing its structure during the preparation of the nanoparticles (Muthu and Singh 2008). Another study performed X-ray diffraction analysis to further confirm the crystalline state of the quercetin samples. The X-ray study showed the crystalline nature of quercetin with clear and sharp peaks, while the quercetin nanoparticles had fewer and less intense peaks, indicating the decreased crystallinity of the drug in the nanoparticles (Kumar *et al* 2015). In a study, the XRD of pure ERS and empty formulations showed an amorphous structure, while before processing, MOX was in a crystalline state, and the NPs' spectra no longer had strong peaks. This showed that there was very little free drug present in crystalline form on the surface of NPs and that MOX was molecularly distributed throughout the polymeric matrix (Yurtdaş-Kirimlioğlu *et al* 2018). According to Ramasamy *et al 2014* the absence of a distinctive peak for the SLN formulations shows the drug is fully mixed. This finding implies that the drug is either distributed molecularly or exists in an amorphous form within the lipid matrix crystal lattice (Ramasamy *et al* 2014). By measuring the smallest unfaulted regions or coherently scattering domains of the material, XRD can also provide information on the size of the particles (Basaran 2017). In general, XRD peaks are dependent on crystallite size as they indicate the crystalline nature at a specific value in the 2θ range, but in a study of Repaglinide-loaded polymer nanoparticles, the characteristic Repaglinide peak overlapped with the interference of the coated polymer. It was found that the XRD signal of the encapsulated drug was barely detectable, suggesting that the drug was dispersed at the molecular level in the polymer matrix and thus no distinct crystallites were found in the drug-containing matrix (Lekshmi *et al* 2012). In another study, the pure drug carbamazepine and its polymeric nanoparticles were analyzed by XRD. The crystalline nature of pure drug was indicated by a clear sharp single peak. A notable change was

found in the diffraction pattern of pure polymer and drug-loaded polymer nanoparticles. There was a variation in the intensity of the peak, which may be due to the dispersion of the drug at the molecular level leading to a lower level of detection. Furthermore, the slight disappearance of the carbamazepine peak indicated that the drug was trapped inside the polymer and also indicated the amorphous state of the encapsulated drug.

XRD is a helpful method to learn about the atomic structure of materials and confirm the development of nanoparticles (NPs). It provides average values and can identify chemical composition by comparing patterns with a database, but it doesn't work well for very small or non-crystalline materials. This technique is used to check if NPs are present, understand their structure, and find impurities. It also helps calculate the average size of the NPs using a specific formula based on the width of the strongest peak in the XRD data. For instance, a study by Bayat *et al* 2021, the XRD patterns of calcinated and non-calcinated zinc oxide nanoparticles showed clear peaks, indicating a hexagonal structure and confirming the purity of the samples without impurities. The XRD pattern of Zn nanoparticles showed no clear peaks, indicating they were mostly amorphous or too small to detect, making size calculation impossible. For magnesium oxide NPs, the cubic structure was revealed by the XRD pattern with clear peaks, confirming high purity and an average size of 69.6 nm, consistent with other studies. The XRD of silver NPs showed distinct peaks, confirming their crystalline nature and a face-centered cubic structure, with no impurities detected. The XRD pattern for Cu nanoparticles showed no clear crystalline peaks, indicating they are mostly amorphous or too small to detect, with some possible crystalline phase present. Similarly, the XRD pattern for Fe nanoparticles also lacked clear peaks, suggesting they were mainly amorphous, possibly due to a higher amount of amorphous particles or their small size. A study by Singh *et al* 2015 found that silver nanoparticles made from E. hybrida leaf extract had a specific crystal structure, showing clear patterns in XRD analyses, which confirmed they were crystalline and had a certain size. Another study by Younas *et al.* 2021 analyzed the XRD of silver nanoparticles, showing they had a spherical shape and were crystalline, with specific peaks indicating their structure and the presence of other compounds in the nanoparticles.

Conclusion

X-ray Diffraction (XRD) is a non-destructive, accurate method for determining the structure, phase identification, and crystal structure of materials. It is widely used in fields like material science, forensics, food, and thin film analysis, requiring minimal sample preparation. Recent advancements have made XRD devices more affordable and automated, broadening accessibility for research, process optimization, and quality control. While it faces challenges with amorphous materials, resolution, and surface sensitivity, integration with techniques like TEM, SEM, and spectroscopy enhances its capabilities. XRD remains pivotal in material analysis, driving progress across industries and fundamental research, especially in pharmaceuticals and forensic science.

Acknowledgment

Authors are thankful to UGC-DAE Consortium for Scientific Research, Indore, India for providing the research grant and fellowship under CRS project (CRS 2022-23/01702)

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