Development and characterization of a laboratory based X-ray diffraction imaging system for material and tissue characterization

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Abstract

Soft tissues feature a degree of short-range order, giving rise to diffraction patterns with broader peaks than crystalline materials. For this reason, an X-ray diffraction system (XRD) for characterization of soft tissue has less stringent requirements in terms of momentum transfer resolution than one aimed at characterizing crystalline materials. We present results on the characterization of two energy-dispersive XRD systems. The first was based on conical collimation at 5.9$^\circ$ and the second was based on linear collimation at varying angles between 2$^\circ$ and 10$^\circ$. The systems include a CdTe detector and a W-anode X-ray source. The angular resolution was measured as a function of sample thickness and scattering angle. Preliminary results confirm the effectiveness of the method for the characterization of biological tissue, showing insensitivity to small changes in angular acceptance and sample thickness, also showing it is possible to combine scattering data obtained at different angles.

Keywords: Energy-dispersive X-ray diffraction; small angle scattering; tissue characterization

1. Introduction

Scattering of X-rays is present in all imaging techniques. Initially it was dealt with as a source of image degradation and blurring. More recently, however, the ability of coherently scattered X-rays to reflect intrinsic variations in the molecular structure of the material under test has been demonstrated (Evans et al., 1991). This is due to interference effects that depend on the inter-atomic distances (Castro et al., 2004, 2005; Round et al., 2005; Cunha et al., 2006; Pani et al., 2007; Oliveira et al., 2008; Sidhu et al., 2009). These studies used X-ray scattering for tissue and material characterization typically in the angular range 2$^\circ$-10$^\circ$ because coherent scatter is predominant with respect to Compton scatter. Within this range, it is a strong function of the scatter angle. In the past, energy-dispersive X-ray diffraction (EDXRD) techniques have proven to be of great use in distinguishing between materials on the basis of
scatter spectrum shape and relative intensity (Harding et al., 1990 and Kidane et al., 1999). However, the systems developed so far are not viable for clinical use because of the long measurement times.

In the present work, an EDXRD imaging system has been set up with the aim of characterizing soft tissue. The final objective will be to test the extent to which simultaneous acquisitions at different scatter angles can be performed in order to reduce the acquisition time. Reducing the acquisition time would be crucial for using the EDXRD system in a clinical environment (operating theatre or histopathology lab) for performing computed tomography (CT) images of "ex-vivo" tissue samples.

In this study, we evaluate different measurement geometries, focusing in particular on determining the extent to which the counting statistics can be increased without altering the diffraction patterns. Increased statistics may be obtained by increasing the angular acceptance or by summing data acquired at different angles. From results obtained, we expect that other influencing factors as, for instance, the detector energy resolution and the geometry of the front collimator could have a direct effect on the quality of the scattering data.

2. Experimental system and methods
2.1 Experimental set up
A laboratory-based X-ray diffraction imaging system using an energy dispersive approach was developed for the present work. We used a conventional polychromatic X-ray beam and measured the diffraction patterns at a fixed scattering angle. The source used was a high intensity tungsten target Comet MXR 225/22 X-ray tube system with a high voltage power supply (Gulmay CP225, Shepperton). The focal spot size of the X-ray tube is 1 mm. During the present measurements, both tube voltage and current were kept constant at 70 kV and 30 mA. Figure 1 shows a schematic diagram of the experimental set up, showing one of the two types of collimator investigated herein (see below).

![Fig. 1. Schematic diagram of the X-ray diffraction set up (top view).](image-url)
The scattered photons were detected using an x- and gamma ray Cadmium Telluride (CdTe) detector (Amptek, XR-100T-CdTe). The detector area and thickness were 3 mm x 3 mm and 1 mm, respectively; its efficiency was greater than 80% in the energy range 3 keV - 80 keV with an energy resolution of 1.2 keV FWHM at 122 keV (Amptek Inc., 2010). The signal, after amplification by a PX2T-CdTe Power Supply with Analog Pulse Shaping amplifier, was analysed by an ADC-Multi Channel Analyser (MCA) system (Toivel D-spect). The MCA was controlled by spectroscopy analysis software (SpectLab) through a USB interface. The detector was shielded with a 3 mm thick Pb sleeve in order to reduce the background of scattered radiation from the surroundings. For both types of diffraction collimator that were investigated, the detector was put in contact with the collimator exit.

2.2 Diffraction collimators
Two types of diffraction collimator have been used in present study:
(a) A 5.9 ° conical collimator consisting of a brass block of dimensions 28.62 mm x 30 mm x 30 mm with a truncated conical core insert of dimensions 14.1 mm (small base diameter), 19.9 mm (large base diameter) (Fig. 2a). The insert is located co-axially within the collimator body and a selection of collimator gap sizes (90 µm, 180 µm and 1.6 mm) have been selected for study. The centre of the scattering volume of the target was determined by scanning a thin (1.3 mm) caffeine sample at different positions in front of the collimator. The optimum position of the sample along the central axis was determined to be the one giving rise to the greatest value of scattering signal intensity. Caffeine, having sharp peaks at well-defined values of momentum transfer, was also used for angular alignment of the collimator, which was assumed to be aligned when caffeine scattering profiles obtained from both sides of the conical insert were coincident. In present study, the optimum location of the sample was at 6.6 cm from the scatter collimator face nearest to the sample. The angular resolution of this system was 0.23° and 1.4° for collimator gap sizes 90 µm and 1.6 mm respectively. It was calculated using the procedure presented by (Pani et al., 2009) and when converted into momentum transfer resolution it ranged from 0.118 nm⁻¹ to 0.471 nm⁻¹ and 0.02 nm⁻¹ to 0.08 nm⁻¹ for collimator gap sizes 1.6 mm and 90 µm respectively in the momentum transfer values range from 0.5 to 2 nm⁻¹. This is because angular resolution is not a constant function of momentum transfer.
(b) A multi-angle collimator consisting of a brass block of thickness 12 mm containing three pairs of circular holes of diameter 1.5 mm, corresponding to three different angles 4.88°, 6.5° and 8.2° (Fig. 2b) and also a central hole that is used for alignment. The collimators were individually mounted on an optical bench that allows movement along the central axis and rotation of the scattering collimators. The centre of the sample scattering volume, for each angular position, was determined using the same procedure as described above for the case of the conical collimator. In this case the centres were found to be at 5.8 cm, 10.4 cm and 14 cm from the face of the scattering collimator nearest to the sample, corresponding to 4.9°, 6.5° and 8.2° respectively. The conical collimation will allow us to greatly increase the speed of data acquisition if we had a large-area detector (single-element or pixellated). With a pixellated one we could use several concentrical cones and integrate angle by angle the relevant pixels. With the multi-angle collimator, one can use simpler, cheaper single-point, small-area detectors. Multiple detectors would allow simultaneous acquisition of spectra at different angles. The current collimator was designed with different centres of scattering volume for each different angle for ease of manufacturing. Future designs will have a coincident centre of the scatter volume for all scatter angles. The angular resolution of this system was 0.77° for scatter angles 4.9° and 6.5° while for the scatter angle 8.2° it was 0.79°. It was calculated using the procedure presented by Pani et al., (2009). Due to the non-linear relationship between momentum transfer and scatter angle when the angular resolution is converted to momentum transfer resolution it is a non-constant function of momentum transfer in our case. It ranged from 0.0784 nm⁻¹ to 0.3136 nm⁻¹, 0.0639 nm⁻¹ to 0.2555 nm⁻¹ and 0.048 nm⁻¹ to 0.192 nm⁻¹ for angles 4.9°, 6.5° and 8.2° respectively in the momentum transfer range from 0.5 to 2 nm⁻¹.

Fig. 2. Schematic drawing for the scattering collimators: (a) conical collimator (b) Multi-angle collimator.
2.3 Samples
The X-ray diffraction system was tested using materials of well defined scattering profiles including caffeine, nylon, polycarbonate and perspex. The plastic samples (nylon, polycarbonate and perspex) were prepared in the form of parallel-sided, polished blocks of different thicknesses. All samples were scanned at the position of the diffractometer corresponding to each angular position described above. The acquisition time was 3 minutes for the conical collimator and 5 minutes for the multi-angle collimator.

2.4 Data corrections
The raw data were corrected as follows:
(a) The energy values corresponding to the diffraction profile were converted to momentum transfer values using the relationship:

\[ x = \frac{E}{hc} \sin(\frac{\theta}{2}) \]  

where \( E \) is the energy of the incident photons, \( h \) is the Planck constant, \( c \) is the speed of light and \( \theta \) is the scattering angle.
(b) The background due to the scattering of photons from the sample holder and the surroundings was subtracted from the experimental raw data.
(c) The acquired spectra were normalised by the incident spectral shape as obtained from Cranley et al., (1997).
(d) The experimental data was corrected for the attenuation of X-rays through the sample using attenuation data created by Nowotny (1998).

3. Results and discussion
3.1 Conical collimator
3.1.1 Angular acceptance
The choice of angular acceptance of the diffraction system concerned examination of the opposing effects of a small gap (eg. 90 µm), allowing good momentum transfer resolution or of a large gap (eg. 1.6 mm), allowing higher statistics for the same acquisition time. The current work looked at materials such as perspex and polycarbonate which generally show one or more broad peaks (Klug and Alexander, 1974) and materials which feature a greater degree of short range order, including caffeine, nylon and polyethylene. The results, shown in figure 3, demonstrate reproducible diffraction patterns for the materials with a higher degree of short-range order as well as for less structured materials. For the former, the peak width
increases for the larger collimator gap sizes; the increase in the peak width between the collimator gap sizes 1.6 mm and 90 µm was found to be of the order of 0.04 nm\(^{-1}\) for caffeine 1.3 mm and 0.1 nm\(^{-1}\) for polyethylene 5.5 mm, while differences in peak width between the diffraction patterns of the less structured materials are less perceptible suggesting the possibility of using broad collimation without loss of information. The increase in the peak width between the aperture 1.6 mm and 90 µm was found to be of the order of 0.01 nm\(^{-1}\) for both polycarbonate 7.9 mm and perspex 2 mm.

In the present results for nylon (figure 3(d)) a triple-peak diffraction pattern is observed (with strong peaks at 0.78 and 1.18 nm\(^{-1}\), and smaller peak at 1 nm\(^{-1}\)) in agreement with Poletti et al., (2002) while others (Kosanetzky et al., 1987; Harding et al., 1990) only detected a double-peaked diffraction pattern. The peak position at 1.18 nm\(^{-1}\) is in agreement with that observed by Kosanetzky et al., (1987) and Poletti et al., (2002). Similarly, the peak position of polyethylene at 1.19 nm\(^{-1}\) is in agreement with that observed by Kosanetzky et al., (1987) and Poletti et al., (2002). Similarly the perspex peak position at 0.77 nm\(^{-1}\) is in good agreement with that represented by Poletti et al., (2002), Kosanetzky et al., (1987), Tartari et al., (1998) and Bradley et al., (1989). The results obtained confirmed that the amount of scatter from all investigated samples in the case of collimator gap size of 1.6 mm is considerably higher and the difference in peak width between the scattering data in the case of collimator gap sizes of 1.6 mm and 90 µm is not crucial. This means the collimator gap size of 1.6 mm gives better statistics in shorter time without any significant loss in the resolution particularly for less structured materials when compared to that of 90 µm as for the same acquisition time we get much lower statistics.
3.1.2 Sample thickness

In order to investigate the values of sample thickness above which differences in peak broadening of scatter signatures becomes non-negligible, samples of different thicknesses (between 1 and 10 mm) were scanned at different angular acceptances, as shown in figure 4. The effect of sample thickness on peak broadening can be explained on the basis of drawings shown in figure 5. Increasing the sample thickness increases the range of scatter angles that lie within the angular acceptance of the collimator. In case of collimator gap size 1.6 mm, a variation in both nylon and perspex peak widths of the order of 0.08 nm\(^{-1}\) and 0.02 nm\(^{-1}\) respectively is found, while in case of 90 µm collimator gap size, these variations for nylon
and perspex peak widths were found 0.04 nm\(^{-1}\) and 0.01 nm\(^{-1}\) respectively. It must be pointed out that the situation will be different when moving from more structured materials (samples used for testing the system) to biological ones which feature broader peaks where in this case the resulting data will be less affected by the angular acceptance. This suggests the possibility of scanning samples of greater thickness without further corrections such as deconvolution by the angular resolution of the system.

![Graphs showing scattering spectra](image)

**Fig. 4.** The scattering spectra of different thicknesses of materials at two angular acceptances cases (a) and (b) are Nylon spectra, (c) and (d) are Perspex spectra. Cases (a) and (c) are spectra obtained for collimator gap size of 1.6 mm while cases (b) and (d) are spectra obtained for angular acceptance of 90 µm. All data are normalized with respect to the maximum intensity.
3.2 Multi-angle collimator

3.2.1 Samples of different thicknesses at different angles

The present study is aimed at comparing data acquired at different angles. Combining them would allow reduction in acquisition times in a diffraction CT system providing that, after corrections for the spectral shape of the beam, diffraction patterns acquired at different angles are equivalent. The scattering spectra in figure 6 for data obtained at different angles, shows a discrepancy in peak position and width for caffeine (for instance, the peak positions for caffeine 8.8 mm at angles 4.9°, 6.5° and 8.2° are 0.67 nm⁻¹, 0.66 nm⁻¹ and 0.69 nm⁻¹ respectively while the variation in peak width between angles 4.9° and 8.2° is 0.1 nm⁻¹). For other less structured materials, such as polycarbonate, discrepancies are less apparent (for polycarbonate 2.1 mm, the peak positions at angles 4.9°, 6.5° and 8.2° are almost the same (0.77 nm⁻¹) while the variation in peak width between angles 4.9° and 8.2° is 0.02 nm⁻¹). These results suggest that combining data at different angles is practicable for soft tissues that feature intrinsically broader peaks than materials with a high degree of order such as caffeine.

Fig. 5. Schematic drawing represents the centre of scatter volumes as shaded areas resulting from the intersection between scatter collimator and source axes. The thick sample gives a range of scattering angles higher than that of the thin one.
5. Conclusion
The properties of an energy-dispersive X-ray diffraction system for two different scattering collimators have been represented. The angular acceptance study showed negligible differences in peak width between the scattering data obtained in the case of the three investigated angular acceptances, the increase in the peak width between the angular acceptances 1.6 mm and 90 μm was found to be of the order 0.01 nm⁻¹ for less structured materials. Because we will be dealing with materials with intrinsically broad peaks we will not likely to be affected by these differences. The sample thickness study at different angular acceptances showed the sample thickness to have only small effect on peak broadening which suggests using a collimator gap size of 1.6 mm for better statistics in shorter acquisition time without any significant loss in the information.

Fig. 6. The scattering spectra of various samples of various thicknesses: (a) Polycarbonate 2.1 mm (b) Polycarbonate 9.5 mm (c) Caffeine 1.3 mm and (d) Caffeine 8.8 mm at different scattering angles. Data are normalized with respect to the maximum intensity.
The effect of several different scattering angles on the peak position and width has also been studied. The variation in peak width at different angles can be neglected for the materials and thicknesses investigated. We expect similar thicknesses in our future clinical system. Studies of biological tissue, featuring intrinsically broader peaks than the plastic materials, demonstrate that it would be possible to directly sum diffraction patterns acquired at different angles without need for complex corrections.

Work is now in progress to develop the system. In particular, we seek to improve the quality of measured data, especially at low scattering angles, through design of a new primary collimator of a smaller diameter. Low scattering angle allows a certain momentum transfer range to be investigated with higher energy spectra, hence limiting the need for attenuation corrections.

An optimised system is intended to provide for medical applications such as measuring the diffraction signals from human breast tissues. This will be integrated with a scanning system in order to acquire diffraction CT images in different geometries.

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References


