Radiation Physics and Chemistry 80 (2011) 779–781

Contents lists available at ScienceDirect

Radiation Physics and Chemistry

journal homepage: www.elsevier.com/locate/radphyschem

X-ray scattering for the determination of fat content in dairy products

Wael M. Elshemey

Biophysics Department, Faculty of Science, Cairo University, Giza, Egypt

ARTICLE INFO

Article history:
Received 8 October 2010
Accepted 10 March 2011
Available online 17 March 2011

Keywords:
X-ray scattering
Fat content
Dairy products

ABSTRACT

The scattering of X-rays from biological samples has been shown to produce characteristic profiles, which depend on their molecular structure. The highly ordered fat molecules in an adipose tissue result in a relatively sharp scattering peak at 1.1 nm^{-1} with a scattering profile, which is considerably different from the scattering profile of a water-rich tissue. The latter is characterized by a broad scattering peak at about 1.6 nm^{-1}. A biological sample consisting of a mixture of both adipose and a water-rich tissue is expected to show a scattering profile, which is directly linked to the relative contribution of each component and would reflect the percentage by volume of each component in the mixture. In this work, X-ray scattering profiles of a number of dairy products and water are measured. The values of two selected X-ray scattering characterization parameters (I_1/I_2% and areas A_1/A_2% of the scattering peaks at 1.1 and 1.6 nm^{-1}, respectively) are plotted against the fat content of each of the measured dairy samples. Results show a strong linear dependence of each of the X-ray scattering parameters and the fat content of the investigated dairy products. These results suggest a possible use of such technique as a new, simple and straight forward method for determination of fat content of dairy products that would join and support the currently available techniques.

1. Introduction

The scattering of X-rays from biological samples has been extensively studied by many authors for the purpose of tissue characterization. Kosanetzky et al. (1987) presented X-ray scattering profiles of a number of biological samples including water, muscle, liver, kidney, fat, and brain gray and white matter. They pointed out that soft tissues produce similar scattering profiles characterized by a single broad scattering peak as they are all dominated by scattering from water, whereas, fat produces a considerably different profile characterized by a relatively sharp diffraction peak due to the spatially high degree of order of fat molecules in an adipose tissue. Evans et al. (1991) presented early X-ray scattering measurements highlighting the differences between the scattering profiles of adipose tissue and breast carcinoma. The use of a polyenergetic source seemed to affect the shape of their measured profiles. Kidane et al. (1999) showed that breast carcinoma could be characterized on the basis of the shape of X-ray scattered spectrum (from 1.0 to 1.8 nm^{-1}) and the relative intensities of the adipose and fat free peaks at 1.1 and 1.6 nm^{-1}, respectively. They referred to the fact that carcinoma is characterized by the lack of isolated pockets of fat within its mass and hence would give rise to low-intensity scattered signals at 1.1 compared to 1.6 nm^{-1}. They added that peak height analysis of such measurements is sensitive to the fat content of different tissues. The last decade witnessed several other contributions marking valuable developments in the area of characterization of breast cancer based on the characteristic nature of X-ray scattering from adipose and soft tissue (Royle et al., 1999; Poletti et al., 2002; Castro et al., 2004; Ryan and Farquharson, 2004, 2007; Cunha et al., 2006; Griffiths et al., 2007, 2008; Oliveira et al., 2008; Theodorakou and Farquharson, 2008; Elshemey and Elsharkawy, 2009; Bohndiek et al., 2009; Elshemey et al., 2010).

This work makes use of the difference in the X-ray scattering profiles of fat and water-rich component of dairy products for determination of their fat content. The declaration of the total fat content is of special interest for nutritional and legislative purposes (Evers et al., 2000). It is an important indication of quality, both economically and physiologically (Badertscher et al., 2007). The established reference methods for the determination of fat content in dairy products, such as Acid-Butyrometrie (Gerber) method and the RÖse Gottlieb method has been reported to suffer from several practical and precision problems (Evers et al., 2000; Badertscher et al., 2007). This stimulated a search for different methods, which may either overcome some of the disadvantages of the standard methods or may offer an acceptable alternative that is more practical in specific situations. Beattie et al. (2004) presented a method for determination of fat content in butter using Raman spectroscopy. Dukhin et al. (2005) utilized acoustic spectroscopy for fat content measurements in a wide variety of dairy products. Xin et al. (2006) employed a laser

E-mail address: waelelshemey@yahoo.com

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doi:10.1016/j.radphyschem.2011.03.010

light scattering technology for the determination of fat content in milk.

2. Materials and methods

A variety of dairy products with different fat contents (in g/100 g) are purchased from the Egyptian local market, preserved at 4 °C and measured within few days. The purchased samples are yoghurt (2.5 g/100 g), Egyptian kareesh Cheese (7 g/100 g), feta light cheese (12 g/100 g), Egyptian white cheese (17 g/100 g), Labnah dairy product (18 g/100 g), spread cheese (27 g/100 g), cheddar cheese (30 g/100 g), spread cheese (33 g/100 g), spread cheese (45 g/100 g), milk cream (80 g/100 g), butter (88 g/100 g), and ghee (99 g/100 g). The values inside the brackets are the fat contents as printed on the product label of each sample. These values are found well matched with those determined by the National Nutrition Institute of Egypt (2006) for the fat contents of food stuff in the Egyptian market.

For X-ray scattering measurements, a dairy product is smeared into the circular depression (0.1 cm in depth and 2.5 cm in diameter) of the horizontal aluminum sample holder of a Schimadzu (XRD 6000) X-ray diffractometer. A distilled water sample is also measured by carefully adding fine drops until the depression in the sample holder is filled. The diffractometer employs a Cu target, which produces 8.047 keV CuKα collimated X-rays using a built-in Ni filter. Measurements are carried out at 40 kV and 30 mA in a θ–2θ step mode with a step of 0.25°. Data are plotted against the momentum transfer parameter; $x(\mathrm{nm}^{-1})=(\sin\theta/2)/\lambda(\mathrm{nm})$, where θ is the scattering angle and λ is the wave length of incident X-ray beam.

3. Results and discussion

Fig. 1 presents the X-ray scattering profiles of water and six dairy products of different fat contents. All graphs are tail normalized by dividing each point in a graph by a value which makes the tail of this graph coincides with that of water. This is in order to be able to compare the differences in the scattering profiles and peak amplitudes of the investigated samples. From the graph one can notice the gradual change in the peak amplitudes at 1.1 compared to 1.6 nm^{-1} with the change in the fat content of the different samples. The water sample (zero fat content) shows the standard X-ray scattering profile of water with a broad peak at about 1.6 nm^{-1} and extremely low amplitude at 1.1 nm^{-1}. The yoghurt (2.5 g/100 g) sample exhibits increased amplitude at 1.1 nm^{-1} compared to water probably due to the presence of small amount of fat. Further increase in the fat content of the dairy product as in feta light cheese (12 g/100 g) results in further increase in the peak amplitude at 1.1 compared to 1.6 nm^{-1} where the two peaks turn to be of almost the same amplitude. The same trend continues for the other samples starting from spread cheese (33 g/100 g) and passing by milk cream (80 g/100 g) and butter (88 g/100 g) up to ghee (99 g/100 g) which records the maximum difference in amplitude between the fat peak at 1.1 compared to the peak at 1.6 nm^{-1}. This is clearly due to the presence of the highest fat content in ghee compared to all other samples.

The graphs in Fig. 1 together with the rest of the measured samples is further analyzed in order to examine the possible dependence of an X-ray scatter parameter on the fat content of a dairy product. The ratio of amplitudes, I_1/I_2% (Fig. 2) and areas A_1/A_2% (Fig. 3) of the scattering peaks at 1.1 and 1.6 nm^{-1}, both exhibit a strong linear dependence on the fat content of dairy products ($R^2=0.97$ and 0.98, respectively). The area under 1.1 nm^{-1} peak is determined as the region starting from

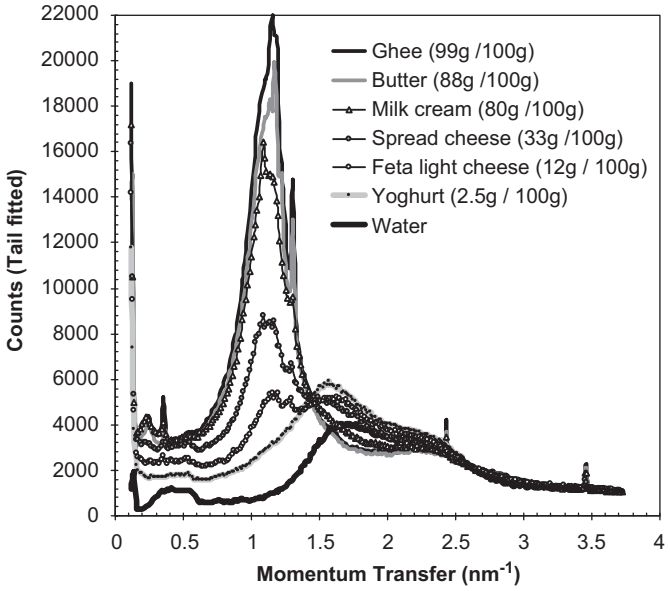

Fig. 1. X-ray scattering profiles of water and some dairy products. Figure legend shows the fat content of each sample.

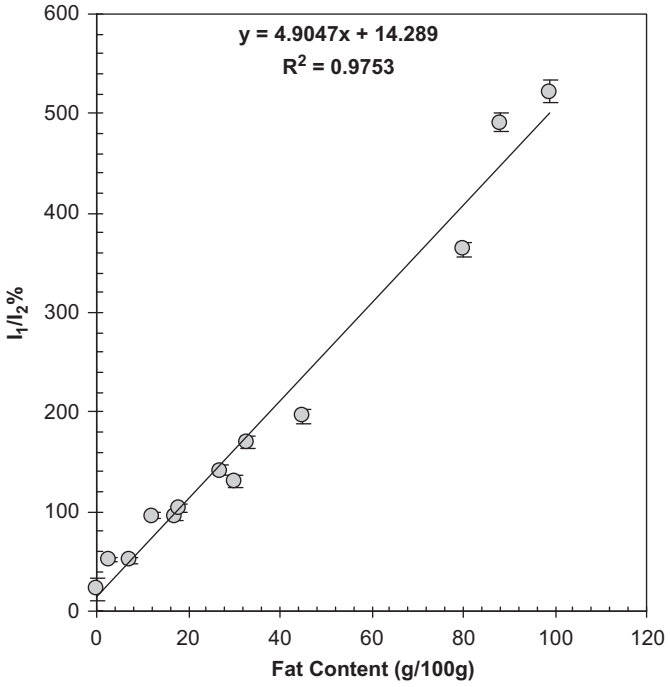

Fig. 2. Variation of the amplitudes of 1st scattering peak at 1.1 nm^{-1} relative to the 2nd scattering peak at 1.6 nm^{-1} (I_1/I_2%) plotted versus the fat content of each sample. The straight line passing by the data points represents the best linear fit.

momentum transfer value of 0.5 nm^{-1} up to a momentum transfer value of 1.4 nm^{-1}, which corresponds to the trough of the valley between the two peaks. The area under the 1.6 nm^{-1} peak is calculated as the region starting from the trough up to a momentum transfer value of 3.7 nm^{-1}. The calculation of area under curve started at momentum transfer value of 0.5 nm^{-1} in order to avoid the region where forward scattered photons may contribute to the measured X-ray scattering profile.

The slope of the fitting straight line in Fig. 2(4.90) for the I_1/I_2% parameter being greater than that of the other characterization parameter (1.42 for A_1/A_2%) may indicate greater sensitivity of the

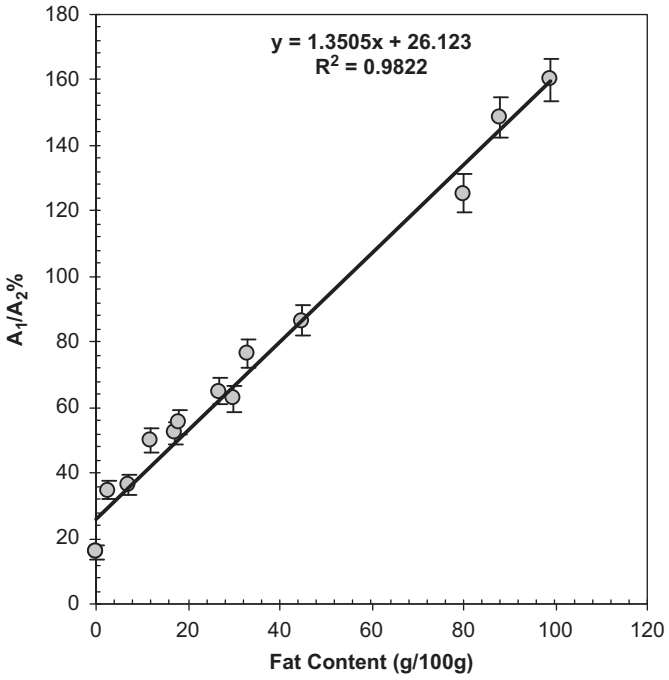

Fig. 3. Variation of the area under peak of 1st scattering peak at 1.1 nm^{-1} relative to the 2nd scattering peak at 1.6 nm^{-1} (A_1/A_2%) plotted versus the fat content of each sample. The straight line passing by the data points represents the best linear fit.

I_1/I_2% parameter towards the changes in fat content of dairy products. It should be noted that although the variation in the intensity ratio is more sensitive to the fat content changes (Fig. 2) than the areas under the peaks (Fig. 3), the uncertainty in identifying the precise peak intensity is evident from the fact that the points in Fig. 2 are somewhat far from the best fit line than their (Poisson) statistical errors. On the other hand, Fig. 3 shows that the areas in the chosen regions of interest are consistent with the Poisson errors in the data. Nevertheless, the linear dependence on fat content of the two parameters is convincing and indicates that the discussed method would probably be a promising tool for the determination of fat content in dairy products.

4. Conclusion

The present work introduces a novel method for the determination of fat content in dairy products. The method is simple and does not require a fat extraction step. In order to determine where the present method stands compared to the currently available methods a future inter comparison study dedicated for such purpose may be conducted.

Acknowledgment

The author would like to acknowledge the support of Rehab Hassan, Samar Hamad, and Shahenda Mohamed, the 4th year Biophysics students at the Faculty of Science, Cairo University, Egypt.

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