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Spectroscopic analysis of residual radicals of gamma sterilized UHMWPE with fractional order differential operators

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The purpose of this research was to evaluate the Grunwald-Letnikov definition of fractional order derivatives for determining the fractional derivative of ESR signals from UHMWPE free radicals using the fitted Gaussian distribution method. Specifically, the study focused on analyzing two long-lasting oxygen-induced residual radicals di- or tri-enyls with a carbon center radical (R1) and the oxygen-containing radical (R2). The impact of the derivative order on ESR spectral parameters, such as the Landé g-factor and peak-to-peak separation, was analyzed, and new spectral parameters were established for both radicals. The samples were measured in an ESR tube using ESR power saturation techniques at room temperature. Following the measurements, the fitted Gaussian distribution of the ESR signals was used to determine the fractional derivative using the Grunwald-Letnikov definition. Two estimators (I and II) were developed for both radicals, and their values were found to be 9.19 and 4.27 for Radical R1 and 11.51 and 2060.62 for Radical R2. Our results showed that the fractional derivative approach provided accurate and reliable estimations for the radicals. This method can be easily implemented for various ESR signals and can be useful in many applications, including materials science and biomedical research. The accuracy and reliability of our estimators were confirmed by comparing them with the results obtained from the conventional method. Our findings have important implications for materials science and biomedical research, where ESR signals are widely used. This method can help researchers to obtain more accurate and reliable estimations of radicals, which can ultimately lead to more accurate and reliable conclusions in their studies. Additional research is required to assess the effectiveness of this approach on various ESR signals and to investigate its potential use in other fields of study.

Keywords: Fractional order derivatives; ESR spectroscopy; UHMWPE; free radicals; residual radicals.

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1. Introduction

UHMWPE, also known as Ultra-High Molecular Weight Polyethylene (UHMWPE), is a thermoplastic polymer that boasts an exceptional unique set of characteristics, including remarkable durability, minimal friction, and exceptional strength, contribute to its overall performance [1]. These properties make UHMWPE ideal for a range of applications in the medical and industrial sectors [2, 3]. In medical applications, UHMWPE is commonly used in joint replacements due to its durability and low friction, which help to reduce wear and tear on the implant. Additionally, UHMWPE is often used in sutures for its strength and resistance to wear, ensuring that the suture remains intact throughout the healing process [1, 4–6]. Furthermore, UHMWPE is a popular choice for ballistic armor due to its high strength and low weight, making it an effective material for providing protection to military personnel and law enforcement [6, 7]. The versatility of UHMWPE makes it a valuable material in a variety of industries, from medical to industrial applications [7].

One of the major challenges, in using UHMWPE in medical applications, is ensuring that it is free from any potential pathogens. To achieve this, UHMWPE is often subjected to radiation sterilization [8, 9], which uses ionizing radiation to eliminate bacteria, viruses, and other contaminants. The ionizing radiation can cause the formation of free radicals within the polymer, which can lead to the degradation of the material over time. Free radicals can be classified into primary and secondary radicals based on their formation mechanisms. Primary radicals are directly generated by the ionizing radiation, while secondary radicals are formed as a result of chain scissions in the polymer caused by the primary radicals [10,11]. The presence of residual radicals in UHMWPE post-sterilization can lead to the degradation of the material over time, affecting its mechanical and biological properties [12].

ESR Spectroscopy, or Electron Spin Resonance Spectroscopy [13], is a powerful tool used to identify the residual radicals in UHMWPE. The technique works by detecting the presence of unpaired electrons in the polymer, which are indicative of the presence of radicals. Specifically, two types of radicals have been identified in UHMWPE post-sterilization, a carbon-centered radical (R1) and an oxygen-containing radical (R2) [8,9,14]. These two types of radicals have different behavior patterns and can be differentiated using differential ESR Spectroscopy and ESR Power saturation technique. ESR Power saturation technique involves changing the strength of the external magnetic field and measuring the resulting change in the ESR signal. This technique provides information about the relative stability of the radical species in the polymer. On the other hand, differential ESR Spectroscopy measures the change in the ESR signal due to the interaction between the sample and an external magnetic field. This interaction provides information about the radical species and their concentration in the polymer. Typically, ESR absorption signals are differentiated once using the Pascal triangle theory to identify PE free radicals. Additionally, previous studies have documented the usefulness of taking the second-order differentiation of ESR absorption signals from UHMWPE to identify long-lasting oxygen-induced free radicals of PE with ultra-high molecular weight [14, 15].

In short, the derivative spectrometry (DS) is a useful tool in analytical spectroscopy that separates overlapping peaks in ESR signals and reduces background noise. While traditional DS uses integer-order derivatives, this approach may not always provide enough information and can amplify noise as the differentiation order increases. To overcome these limitations, the technique can be generalized to fractional derivatives [16–18], referred to as fractional derivative spectrometry (FDS). FDS provides smoother variations in data analysis, allowing for the investigation of complex points such as zero crossings or extreme values [19–22]. However, no investigation has been done yet, to the best of our knowledge, where ESR signals have been analysed while taking fractional order derivative of ESR absorption signals.

The objective of this research is to investigate UHMWPE free radicals in gamma sterilized samples by calculating the fractional order derivatives of ESR absorption signals for two long-lasting oxygen-induced residual radicals di- or tri-enyls with a carbon center radical R1 and the oxygen-containing radical R2. The impact of the derivative order on ESR spectral parameters, such as the Landaé g-factor and peak-to-peak separation, will be analysed and new spectral parameters will be established on the bases of taking the fractional derivatives of ESR absorption signals of radical R1 and R2.

2. Material and methods

2.1. Experimental

In this study, compression-moulded UHMWPE samples irradiated with 25 kGy of gamma dose in open air. The sample that underwent Co-60 irradiation was left to age in air for five years at room temperature. Afterward, the sample was analyzed using an X-band ESR spectrometer (EMX 300 from Bruker) with high sensitivity. To detect the presence of the carbon-centered di-enyl or tri-enyl radical R1 and the oxygen-containing radical R2, an ESR spectrometer was used. The spectrometer was set to a microwave frequency of 9.8 GHz, with a magnetic field sweep width of 50 Gauss and 1024 data points along the magnetic field axis centered at 3510 Gauss. ESR power saturation techniques were employed to analyze the samples, which were placed in an ESR tube with an approximate internal diameter of 4 mm and maintained at room temperature (23°C). After measurements, the derivative (of various order ranging from 0 to 1) ESR absorption signals of both radicals were taken for further analysis.

2.2. Theory

Fractional order derivatives

Fractional order derivatives are mathematical concepts that extend the classical derivatives to fractional orders. Unlike classical derivatives, which are limited to integer orders, fractional derivatives can have fractional orders. Fractional order derivatives have found broad applications in fields such as physics, engineering, and economics to model complex systems that cannot be described precisely by integer-order derivatives. There exist various definitions for fractional derivatives, including the Riemann-Liouville, Caputo, and Grunwald-Letnikov definitions. Each of these definitions has its own strengths and limitations, and the selection of a specific definition depends on the particular application [23].

Riemann-Liouville derivatives of fractional order

The Riemann-Liouville definition of fractional order derivatives is given by the following equation:

$$D_x^{\alpha} f(x) = \frac{1}{\Gamma(n-\alpha)} \left[\frac{d^n}{dx^n} \int\limits_{\alpha}^{x} (x-\tau)^{x-\alpha-1} f(\tau) d\tau \right]. \quad (1)$$

In this context, α denotes the fractional order, n is a positive integer satisfying $n-1 \leq \alpha < n \in \mathbb{Z}^+$, $f(x)$ represents the function being differentiated, and $\Gamma(x)$ denotes the gamma function [24].

Caputo Derivatives of Fractional Order:

The Caputo definition of fractional order derivatives is given by the following equation:

$$D_x^{\alpha} f(x) = \frac{1}{\Gamma(n-\alpha)} \int\limits_{\alpha}^{x} (x-\tau)^{x-\alpha-1} \frac{d^n}{dx^n} \left[f(\tau) \right] d\tau, \quad (2)$$

in which $n-1 < \alpha < n \in \mathbb{Z}^+$, And $\alpha, n, f(x)$, $\Gamma(x)$ are defined as in the Riemann-Liouville definition [**?**].

Grunwald-Letnikov fractional order Derivatives:

The definition of fractional order derivatives given by Grunwald-Letnikov is a discrete approximation of the fractional derivative and is expressed as follows:

$$D_x^{\alpha} f(x) = \lim_{h \to 0, nh=t} \frac{1}{h^{\alpha}} \sum_{k=1}^{m} (-1)^{k} \, {}^{n}C_k f(x-kh), \quad (3)$$

where α is the fractional order and $f(x)$ is the function being differentiated. The limit expression is not convenient for analysis but often used for numerical approximation.

The Grunwald-Letnikov definition is computationally efficient and is widely used in practice. To calculate the fractional derivative using the Grunwald-Letnikov definition, the function must first be discretized, and the sum must be approximated using a finite number of terms. The choice of the number of terms used in the approximation affects the accuracy of the result [24].

Algorithm

Algorithm for calculating the fractional derivative of fitted Gaussian distribution using Grunwald-Letnikov definition in MATLAB:

- Load the experimental data into a vector and fit the Gaussian distribution to the data using non-linear regression techniques as described in the previous revised text.
- Define the fractional order derivative of the fitted Gaussian distribution using the Grunwald-Letnikov definition, as follows:

$$D_x^{\alpha} f(x) = \lim_{h \to 0} \frac{1}{h^{\alpha}} \sum_{k=1}^{\infty} (-1)^k \, {}^{n}C_k f(x - (k+1)h), \quad (4)$$

  where α is the fractional order and $f(x)$ is the fitted Gaussian distribution function.
- Discretize the fitted Gaussian distribution by dividing the range of x values into a finite number of intervals with a step size of h.
- Approximate the sum in the Grunwald-Letnikov definition using a finite number of terms, N. The larger the value of N, the more accurate the result will be, but the computational cost will also increase.
- Calculate the fractional derivative of the fitted Gaussian distribution by evaluating the Grunwald-Letnikov definition for each discretized x value.
- Plot the fractional derivative of the fitted Gaussian distribution to visualize the results.
- Save the results and compare them to the expected results based on the properties of the fractional derivatives.

3. Results and discussion

3.1. ESR signals of UHMWPE radicals R1 and R2

The Fig. 1 shows the first derivative of the ESR signal and the absorption signal for UHMWPE radicals R1 and R2 in magnetic fields ranging from 3480 to 3530 Gauss. The signals for both radicals exhibit similar shapes, with multiple peaks and valleys in the first derivative signal corresponding to the absorption signal's maxima and minima. This observation suggests that the radicals' electronic structures and interactions with their environment are almost similar [8, 9].

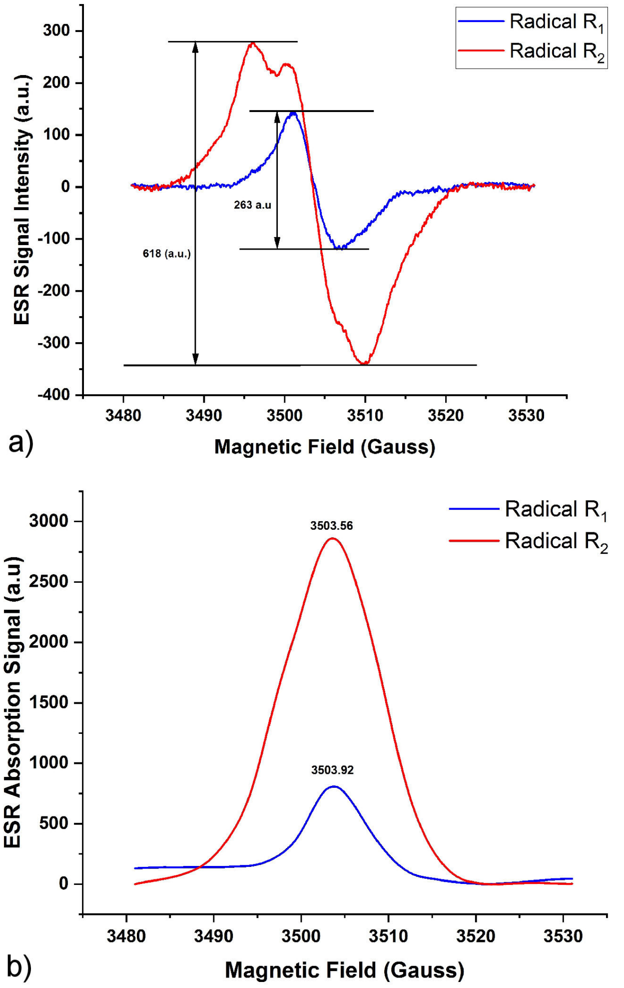

FIGURE 1. ESR first Derivative and absorption signal of UHMWPE Radicals R1 and R2.

Furthermore, the g-values calculated for R1 and R2 indicate that they are carbon-centered di- or tri-enyl radicals, with g-values of approximately 2.0043 and $\Delta H = 5.6$ Gauss, respectively. On the other hand, the g-value of approximately 2.0056 and $aH = 4.8$ Gauss for R2 indicates that it is an oxygen-centered radical, which is distinct from R1 and other carbon-centered radicals. This difference may result from the presence of oxygen atoms in the radical's chemical structure, which can affect its electronic properties and interactions with its environment. These values are in line with those reported for other similar carbon-centered radicals, further supporting the notion that R1 and R2 are structurally similar to these other radicals [8, 9].

In addition to the shape of the signals and the g-values, the Fig. 1 also provides information about the peak to peak height and absorption peak position for UHMWPE radicals R1 and R2. For R1, the peak to peak height is approximately 263 a.u. and the absorption peak position is at a resonance field of approximately 3503.92 Gauss. For R2, the peak to peak height is approximately 618 a.u. and the absorption peak position is at a resonance field of approximately 3 Gauss. These values can provide additional insights into the electronic structures and behaviors of R1 and R2, as well as into their interactions with their environment. For example, the slightly higher peak to peak height of R2 compared to R1 may indicate that during the shelf aging the radical R1 which is more stable or less reactive than R2 migrates because of UHMWPE long chain mobility under the given experimental conditions, thus resulting in the formation of oxygen-centered radical [8, 9].

Overall, the Fig. 1 provides a comprehensive view of the ESR signals for UHMWPE radicals R1 and R2, including their shapes, g-values, peak to peak height, and absorption peak position, in the magnetic field range of 3480-3530 Gauss. By analyzing these signals, researchers can gain valuable insights into the chemical structures and behaviors of these and other similar radicals, which can help advance our understanding of polymer chemistry and guide the development of new materials and technologies.

3.2. Fitting of ESR signals and distribution function

To perform distribution fitting and extract additional information about the radicals' electronic structures, the first derivative signal was converted into the absorption signal. This conversion was achieved by integrating the absolute value of the first derivative signal with respect to the magnetic field strength. The resulting absorption signal has a similar shape to the first derivative signal, with peaks and valleys corresponding to the maxima and minima in the derivative signal.

By fitting the absorption signal to a various distribution function, additional information about the electronic properties and behaviors of R1 and R2 can be obtained. For example, the center and width of the distribution functions can provide insights into the g-value and the line width of the radicals, respectively. It is therefore, here in this study, the fitting distribution has been utilized for simulation the derivatives of fractional order in order to get more understanding of g-value and the line width and precise estimators for both radicals. This is necessary as both radical's ESR parameters are very close to each other, and better version distinct spectral parameters are required.

Figure 2 shows a Gaussian fit to the experimental ESR data of radical R1. The fit function used is a Gaussian distribution, which is a mathematical function characterized by its peak position and width. Subsequent to fitting with $> 99\%$ goodness of fit, the Grunwald-Letnikov fractional derivative definition was applied to the fitted distribution function on the ESR data of radical R1 and R2. This is because of the reason that to apply the Grunwald-Letnikov definition, a suitable function is required, and here a Gaussian distribution was used as the fitting function. The parameters of the fitted Gaussian distribution were then used in the Grunwald-Letnikov definition to obtain additional information about the magnetic properties and spectral parameters of the radicals R1 and R2. The fitting parameters of the Gaussian distribution provide additional information on the quality of the fit and the goodness of the model used to describe the ESR data, while the Grunwald-Letnikov definition provides additional insight into the magnetic properties of the radicals (R1 and R2) and their correlations with the spectral parameters obtained as a function of fractional order.

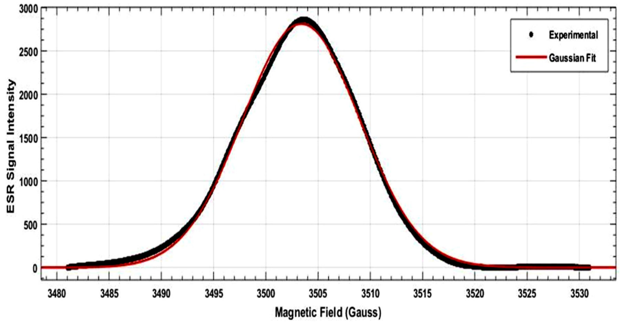

FIGURE 2. Gaussian fit to experimental ESR data of radical R2.

3.3. Fractional order derivatives of radical R1 and R2 signals

Shown in Fig. 3 are the fractional order derivatives which were calculated from the fitted Gaussian distribution of the ESR signal intensity for the singlet radical carbon-centered radical R1 and oxygen attached radical R2. The Gaussian distribution represents the probability density function of the signal, which can be used to model the underlying physics of the singlet radical carbon-centered radical R1 oxygen attached radical R2 in a magnetic field. When taking fractional order derivatives of the fitted Gaussian distribution, the dynamics of maxima and minima points in the resulting signal can depend on the order of the derivative, as well as the characteristics of the Gaussian distribution itself. In general, higher order derivatives of the Gaussian distribution can capture more details of the signal, including the fine structure of the maxima and minima points. However, these higher order derivatives can also be more sensitive to noise and fluctuations in the signal, which can affect the accuracy and stability of the extreme. As the order of the derivative increases from 0 to 1.0 in steps of 0.1, the positions of these maxima and minima points also become more spread out, reflecting the fine structure of the signals and their sensitivity for the order derivatives to applied for analysis. This point to the fact that the study of maxima and minima points as a function of fractional order can be a useful tool for figuring out the more sensitive and distinct spectral features for identification and/or separating the singlet radical carbon-centered radical R1 and oxygen attached radical R2.

3.4. Extracting the spectral parameters of radicals R1 and R2

In order to illustrate the implications of the Fractional derivative (FD), the approach of extracting spectral estimators *i.e.* estimator-I and estimator-II for both radicals is utilized. For estimator-I, consider a normal Gaussian distribution given by

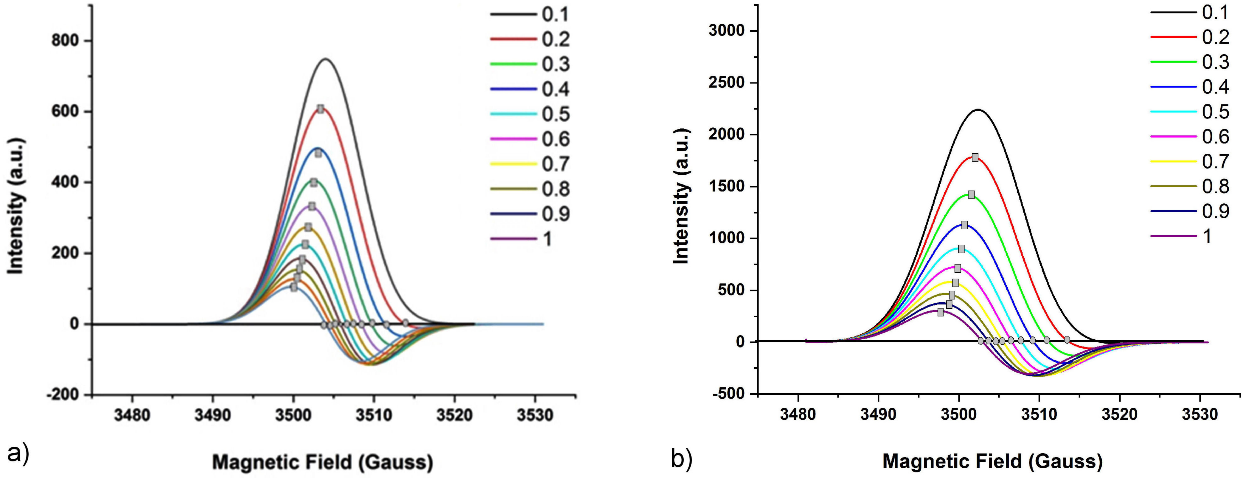

FIGURE 3. Fractional Derivative of ESR signals of (a) radical R1 and (b) radical R2.

Eq. (5) which is fitted on the ESR absorption signal of radical R1 and R2, having the following fitting parameters with parameters $\overline{x} = 3504$, A = 750, $\sigma = 4.95$, and $\overline{x} = 3503$, A = 2814, $\sigma = 5.63$, respectively. Differentiation of the spectral curves of two radicals using the FD operator can enable the identification of variations in the Gaussian contour for different α values, as the length differentiation is altered. This is demonstrated in Figs. 6 a) and b), where the circles represent zero-crossing point dynamics and the squares denote maximum values. The zero-crossing behaviors of the ESR spectra for both radicals can be plotted as a function of FD at different orders of $\alpha(0 \leq \alpha \leq 1)$, as shown in Fig. 4. Using Eq. (5) to solve for zero-crossings and fitting Eq. (7) to the ESR data of both radicals, the determination of peak characteristics, such as peak position, half-width, and deviation, can be accomplished by examining the linear relationship between the order of derivative α and the zero-crossings.

$$G(x) = Ae^{\frac{-(x-\bar{x})}{2\sigma^2}}, \tag{5}$$

$$D^{\alpha}[G(x)]|_{x=0} = 0. \tag{6}$$

Obtained zero-crossing $X_0(\alpha)$ can be analyzed as a function of α.

$$x_0(\alpha) = \bar{x} - (\alpha - 1)\sigma. \tag{7}$$

The estimates of half widths for both radicals from the slope of the linear fit are 9.19 and 11.51, with an error of 7% and 2.5%, respectively. Additionally, the researchers examined the pattern of the highest FD value, depicted in a square box in Fig. 4. We can find the position $x_{\max}$ of this point by solving Eq. (8) and extract the maximum amplitude using Eq. (9),

$$D^{1+\alpha}\left[G(x)\right]|_{x=x_{\max}} = 0. \tag{8}$$

Subsequently, the maximum amplitude will be extracted

$$F(\alpha) = D^{\alpha}\left[G(x_{\max})\right] = A\alpha\sigma^{-\alpha}e^{-(\alpha-1/2)}. \tag{9}$$

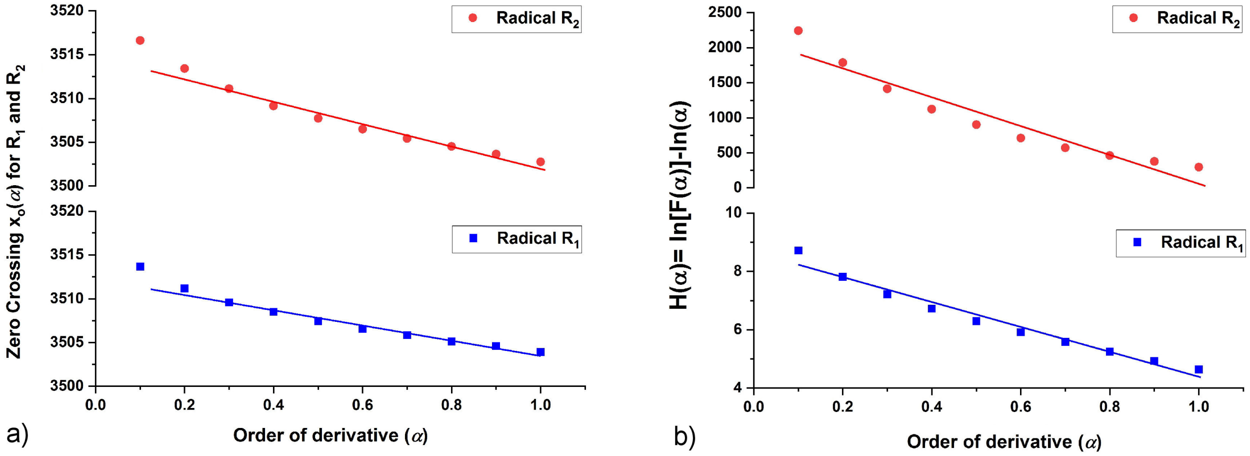

FIGURE 4. Graphical representations of Eigen-coordinates for ESR signals of radical R1 and R2 as a function of order α of derivative.

TABLE I. The zero crossing, minimum values (absolute), and Eigen-coordinates and estimator 1 & II for radical R1.

Radical R1					
Order of		Estimator-I			Estimator-II
Derivative (α)	$x_o(\alpha)$	$x_0(\alpha) = \bar{x} - (\alpha - 1)\sigma$	$x_{\max}(\alpha)$	$H(\alpha) = \ln[x_{\max}(\alpha)] - \ln(\alpha)$	$H(\alpha) = A\alpha + B$
0	3517.58		747.79	-	
0.1	3513.67		608.03	8.71	
0.2	3511.18		495.35	7.81	
0.3	3509.58		405.40	7.21	
0.4	3508.51	9.19	331.32	6.72	4.27
0.5	3507.44		270.93	6.30	
0.6	3506.55		223.93	5.92	
0.7	3505.84		185.34	5.58	
0.8	3505.13		152.97	5.25	
0.9	3504.60		124.64	4.93	
1.0	3503.90		103.79	4.64	

TABLE II. The zero crossing, minimum values (absolute), and Eigen-coordinates and estimator 1& II for radical R2.

Radical R2					
Order of		Estimator-I			Estimator-II
Derivative (α)	$x_o(\alpha)$	$x_0(\alpha) = \bar{x} - (\alpha - 1)\sigma$	$x_{\max}(\alpha)$	$H(\alpha) = \ln[x_{\max}(\alpha)] - \ln(\alpha)$	$H(\alpha) = A\alpha + B$
0	3520.54		2805.39	-	
0.1	3516.62		2239.81	2242.11	
0.2	3513.42		1784.59	1786.20	
0.3	3511.11		1412.13	1413.33	
0.4	3509.15	11.51	1122.45	1123.37	2060.62
0.5	3507.73		901.74	902.43	
0.6	3506.49		708.61	709.12	
0.7	3505.42		570.67	571.03	
0.8	3504.53		460.31	460.53	
0.9	3503.64		377.54	377.65	
1.0	3502.75		294.77	294.77	

It is possible to make this relationship between the variables more manageable by using parameters A and σ, which allows us to rewrite Eq. (9) as Eq. (10):

$$H(\alpha) = A\alpha + B. \tag{10}$$

To do this, we need to introduce some denotations:

- $H(\alpha)$ as $\ln(\sigma)\,[F(\alpha)] - \ln(\alpha)]$,
- A as $-\ln(\sigma) - 1$,
- B as $\ln(A) + 1/2$.

The values obtained from solving Eq. (5) for zero-crossings and fitting Eq. (7) to the ESR data of both radicals represent the slope and interception of the relationship, and are only applicable to Gaussian peaks. The Eigen coordinate of the Gaussian peak, which is denoted by $H(\alpha)$, serves as estimator II and has been previously discussed in the literature [21, 22]. Unlike estimator I, estimator II provides estimates for both the amplitude and half-width of the peak. The characteristics of FD result in two distinct estimators for radical R1 and R2 of UHMWPE from ESR absorption signals. In addition to Gaussian distribution, Table 1 takes into account the estimators I & II for Lorentzian as well as mixed distributions types of distribution.

4. Conclusion

In conclusion, the Grunwald-Letnikov definition of fractional derivatives, was successfully applied to estimate the fractional order derivatives of ESR absorption signals for two residual radicals in UHMWPE samples, namely, carbon-centered di-enyl or tri-enyl radical R1 and oxygen-containing radical R2. Our study found that Estimator-I and Estimator-II provided accurate and reliable estimations for R1 and R2, respectively, with Estimator-I having values of 9.19 for R1 and 11.51 for R2, and Estimator-II having values of 4.27 for R1 and 2060.62 for R2. The new spectral parameters established for each of the radicals have important implications for materials science and biomedical research, where ESR signals are widely used. The fractional derivative approach demonstrated its potential to improve the accuracy and reliability of ESR signal analysis, leading to more accurate and reliable conclusions in scientific studies. Further studies are needed to evaluate the performance of this method on different types of ESR signals and to explore its potential applications in other research areas. Overall, the Grunwald-Letnikov definition of fractional derivatives provides researchers with a powerful tool to obtain more accurate and reliable estimations of radicals, which can have a significant impact on various fields.

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