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# Third-Order Nonlinear Optical Properties of Hibiscus Sabdariffa Dye Using Z-scan Method

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**Abstract**

This study delves into the characterization of nonlinear optical properties of Hibiscus Sabdariffa Roselle through the application of the Z-scan method. Molecular vibration properties were determined using Fourier-transform infrared-ray spectroscopy (FTIR), which aids in identifying chemical bonds by generating an infrared absorption spectrum. Additionally, the absorbance spectrum of the sample was measured using UV-vis Spectroscopy. The research focused on elucidating the nonlinear susceptibility $\chi^3$ and intensity-dependent refractive index n2 of Hibiscus Sabdariffa dye solutions within the nanosecond domain at 532 nm. The experimental approach employed Z-scan and optical limiting techniques to quantify the changes in $n_2$, nonlinear absorption coefficient (β), and the real and imaginary parts of $\chi^3$ concerning the concentration of the natural dye extract. The findings revealed that nonlinear refraction played a pivotal role in shaping the third-order nonlinearity of Hibiscus Sabdariffa dye solutions. This effect led to a substantial optical limiting behavior, showcasing the potential of Hibiscus Sabdariffa as a candidate for nonlinear optical applications. The outcomes of this study contribute valuable insights into the third-order nonlinear optical properties of natural dye extracts and their applicability in optical limiting devices. The calculation of the third-order nonlinear optical properties, derived from the data of nonlinear absorption and refractive index, for roselle diluted in distilled water, the third-order nonlinear optical properties range from -3, while for roselle diluted in ethanol, the range spans from -3. These prove show the reversible absorption behavior of nonlinear optical of Hibiscus Sabdariffa Roselle Dye.

## 1. Introduction

The exploration of nonlinear optical characteristics in organic materials has garnered significant attention in recent times due to its potential applications in various industries, including photonics, telecommunications, and optical signal processing. The development of advanced photonic devices relies heavily on understanding nonlinear optical processes, such as self-phase modulation and two-photon absorption (TPA). Natural-source organic dyes have emerged as promising candidates for nonlinear optical studies, offering ecological advantages over traditional synthetic counterparts [1].

Optical characterization divides into two, linear and nonlinear optical. First, linear optical properties happens when light interacts with the material but does not cause any significant changes in its structure. These properties cause absorption, transition, and reflection to happen. The absorption spectrum in materials produces valuable insight into the energy levels and electronic 5 transitions that occur within the dye molecules. Secondly, nonlinear optical properties happen when a high-intensity light passes through materials, significant changes in optical properties due to the excitation of a nonlinear process [2]. This nonlinear occurrence is two-photon absorption and optical limiting usually the focus of study nowadays.

This occurrence is important in developing more useful devices that require efficient light manipulation, such as optical switches and frequency converters. One method that can be used to measure nonlinear optical properties is the Z-scan. The technique provides useful data on a sample's nonlinear refraction and absorption properties. Measurement of fluctuations in transmitted or reflected light intensity with respect to the sample's position along the beam propagation direction is the underlying notion behind the Z-scan approach [3]. This technique calculates the intensity-dependent transmission, which calculates nonlinear refraction and nonlinear absorption quickly [4].

This research aims to shed light on the potential utilization of organic dye derived from the calyces of Hibiscus sabdariffa in nonlinear optical applications. The study will focus on examining the two-photon absorption coefficient and nonlinear refractive index using the Z-scan method. The focus of this contribution, the Hibiscus Sabdariffa dye, exhibits NLO properties because of a high population of delocalized -electrons within their anthocyanin skeleton, which results in a significant amount of third harmonic generation and two-photon absorption phenomena, including the alluring intensity dependent refractive index and optical limiting [5].

## 1.1 Preparation of Hibiscus Sabdariffa Roselle Dye

Purchase dried Hibiscus Sabdariffa calyces from an online shop (Baker and Flavorist). The calyces were chopped into small pieces using a food processor. The already chopped roselle calyces were diluted in chosen solvent. In this study 96% ethanol and distilled water were chosen. Ethanol and distilled water were used to improve the solubility of dye molecules, help in a more concentrated extraction procedure, and preserve the quality of the dye solution. Ethanol's transparency, refractive index, and solubility properties make it a suitable choice for such measurements, as it minimizes unwanted effects and allows researchers to focus on the nonlinear response of the sample being studied [6]. The properties of the dye and the necessary extraction efficiency influence the choice of solvent. Ethanol and distilled water are also suitable for the subsequent method of measuring linear optical characteristics using UV-vis spectroscopy, Fourier-transform infrared-ray spectroscopy (FTIR) and Z-scan technique in the following steps [7].

These mix solutions were soaked in beaker at room temperature for two hours with concentrations of 0.02g/ml, 0.04g/ml and 0.12g/ml for ethanol and distilled water separately. These solutions will undergo stirring process while soaking. Afterwards the solution was filtered using filter paper. These dye solutions were stored in cold places and away from sunlight.

In Z-scan analysis, each solution was put within a 1 mm cuvette. After characterizing the dye solutions using Fourier transform infrared (FTIR) spectroscopy, important information about their chemical structure was obtained. Several research has reported that organic dyes have large nonlinear optical properties as Maaza et al state. Hibiscus Sabdariffa's anthocyanin is glycosylated in the 3- position with the chemical structure given as delphinidin-3-O-(2-O-d-xylopyranosyl)-d glucopyranoside (R=OH,1), cyanidin3-O-(2-O-dxilopyranosyl)-d-glucopyranoside (R=H, 2) [8] as shown in Fig. 1. The dye solutions' linear absorption coefficients (α) were determined using UV-vis spectroscopy, specifically the U-3900H Spectrophotometer. The absorbance values were measured at a wavelength of 532 nm, and these measurements were used to calculate the linear absorption coefficients (α) for each solution. The resulting values have been compiled in Table 1 for reference.

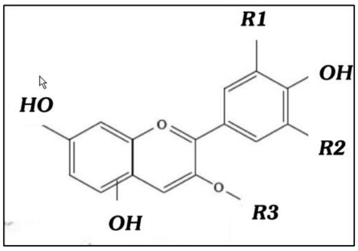


**Fig. 1** *The chemical structure of Hibiscus Sabdariffa Roselle dye. [11]*

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## 1.2 Linear absorption, α optical response

The linear absorption coefficients (α) of the dye solutions were ascertained using UV-vis spectroscopy, employing the U-3900H Spectrophotometer. The linear absorption coefficients (α) for every solution were determined by measuring the absorbance values at a wavelength of 532 nm. For reference, the values that were obtained have been assembled in Table 1. Linear optical absorption from UV-vis can determine the limit of light that can be absorbed by the sample. The absorbance value of the reference is higher than the sample. The reference and the sample are interchanged. The sample is very diluted and close to the absorbance of the reference [9]. The method measures the amount of light that enters a sample and compares it to the amount of light that enters a reference sample.

**Table 1** *List of value of (α) at 532 nm.*

| Sample | Concentration (g/ml) | Linear absorption coefficient, α ($cm^{-1}$) |
|---|---|---|
| Diluted in Ethanol | 0.02 | -3.8468 |
| | 0.04 | -3.6587 |
| | 0.12 | -3.2705 |
| Diluted in distilled water | 0.02 | -3.8383 |
| | 0.04 | -2.6764 |
| | 0.12 | -3.1657 |

## 1.3 Nonlinear optical characterization

Z-scan measuring technique nonlinear optical properties at 532 nm in the nanosecond domain, the intensity-dependent refractive index $n_2$ and nonlinear susceptibility $v^3$ of Hibiscus Sabdariffa dye solutions are reported. They have calculated the nonlinear absorption coefficient (β), nonlinear refractive index $n_2$, and the real and imaginary third-order susceptibility components ($x^3$). From open aperture z-scan data, the nonlinear absorption coefficient (β) can be estimated by [10]:

$$\beta = \frac{\Delta\phi_2\lambda}{2\pi L_{eff}} \tag{1}$$

While $n_2$ is the nonlinear refractive index and I is the intensity of the laser beam from closed aperture

$$n2 = \frac{\Delta\phi_0\lambda}{2L_{eff}l_0} \tag{2}$$

The change of n2, β, and the real and imaginary sections of $X^3$ concerning the concentration of natural dye extract have been measured using z-scan and optical limiting techniques. Where ΔT is the value of one trough from curve of open aperture. The third order nonlinear susceptibility $x^3$ define using equation:

$$X^3 = Re_{x^3} + Im_{x^3} \tag{3}$$

Where the real part obtained from nonlinear refractive index, $n_2$ from z-scan measurement with closed aperture. The behavior of the pre-focus transmission at minimum trough and post-focus transmission maximum peak behavior given the real magnetic susceptibility, $Rex^3$. The imaginary part determines from nonlinear absorption coefficient, β obtained from z-scan measurement [12]. As the sample moves toward the laser beam, the transmitted intensity detected by using a detector increases and decreases, forming a trough and peak. Therefore, a plot of normalized transmittance correlated with sample shift (z shift) is created. Real, $x^3$ and imaginary $x^3$ can be evaluated if their relation were defined as below:

$$Re_{x^3} = 10^{-4} \frac{\varepsilon_0 \, c^2 n_0^2}{\pi} \tag{4}$$

$$\mathrm{Im}_{x^3} = 10^{-2} \frac{\varepsilon_0 c^2 n_0^2 \lambda}{4\pi^2} \beta \tag{5}$$

where ε0 represents the vacuum permittivity, c is the speed of light in a vacuum.

The experimental setup utilizes a diode laser source that emits electromagnetic radiation with a wavelength of 532 nm, falling within the range between microwave and infrared wavelengths. Fig. 2 provides a schematic representation of the Z-scan experiment configuration.

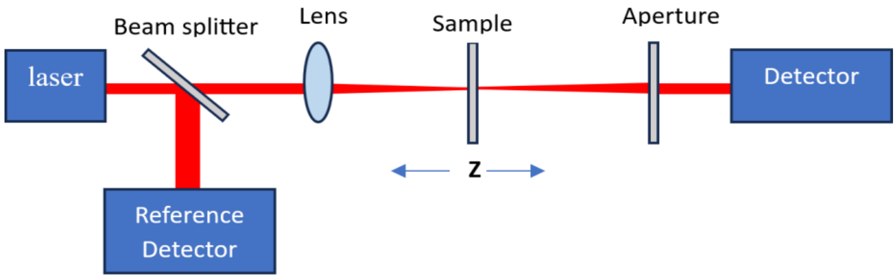



**Fig. 2** *Z-scan arrangement for the z-scan measurements of the various Hibiscus Sabdariffa Roselle dye extract solutions [13]*

The Z-scan experiment typically begins with a laser source providing a focused and collimated light beam. The wavelength of the laser is chosen based on the properties of the roselle sample. Then, a beam expander controls the diameter and divergence of the laser beam to ensure a well-defined and stable input beam. Roselle dye solution was placed in a glass cuvette (1mm). The sample was positioned on a translation stage that allows movement along the z-axis (the direction of propagation of the laser beam). As the sample moves through the beam's focal region, the material's nonlinear effects cause changes in the transmitted light. Next, a photodetector that was connected to an oscilloscope measured the intensity of the transmit ted light as a function of the position of the sample along the z-axis. The changes in intensity provide information about the material's nonlinear absorption and refraction properties. Lastly, the oscilloscope collects and displays the data in real time, showing how the intensity of the transmitted light changes as the sample moves through the focal region. The shape and magnitude of the signal on the oscilloscope are analysed to extract information about the nonlinear optical properties of the material. The data collected from the oscilloscope includes the transmitted intensity profile as a function of the position of the sample along the z-axis. Analyzing this data allows researchers to determine the material's nonlinear absorption and nonlinear refraction coefficients, providing valuable insights into its optical properties under the influence of intense laser light [8]

## 2. Results and discussion

### 2.1 Fourier transform infrared (FTIR)

The FTIR spectra of roselle dye using distilled water and ethanol reveals its functional group as shown in Table 2 The strong peak at 1639 $cm^{-1}$ for Hibiscus sabdariffa, Roselle diluted with distilled water correlates to the C-O stretching, representing the carbonyl group for ketone structure [11]. The CC stretch corresponds to the peak at 2141 $cm^{-1}$. The O-H stretching, the hydroxyl group in roselle dye, is responsible for the wide absorption at peak 3366 $cm^{-1}$.

**Table 2** *Functional group and bond position of Roselle solution diluted in distilled water*

| Concentration (dilute in distilled water) (g/ml) | Functional group class | Bond position ($cm^{-1}$) |
|---|---|---|
| 0.02 | Carbonyl compounds $C = O$ | 1637.111, 10140 |
| | Alkynes $\equiv C - H$ | 3331.859, 5.04 |
| 0.04 | Carbonyl compounds $C = O$ | 1636.801 ,11.536 |
| 0.12 | Carbonyl compounds $C = O$ | 1637.000, 11.748 |

Figure 3 shows the functional group of roselle dye solutions diluted in ethanol. The aromatics group with C-H stretching vibration is responsible for the peak at 879 cm-1 in the FTIR spectra. The esters group, which has C-O stretching vibrations, is responsible for the two strong peaks at 1046 cm-$^1$ and 1087 cm$^{-1}$. The C=O stretching vibration confirms the presence of the ketones functional group at 1646 cm$^{-1}$. The absorption at peaks 2889m-$^1$2976cm-$^1$, and 3366 cm$^{-1}$ are attributed to the O-H stretching, which is the presence of hydrogen bonding in roselle diluted with ethanol solvent [11].

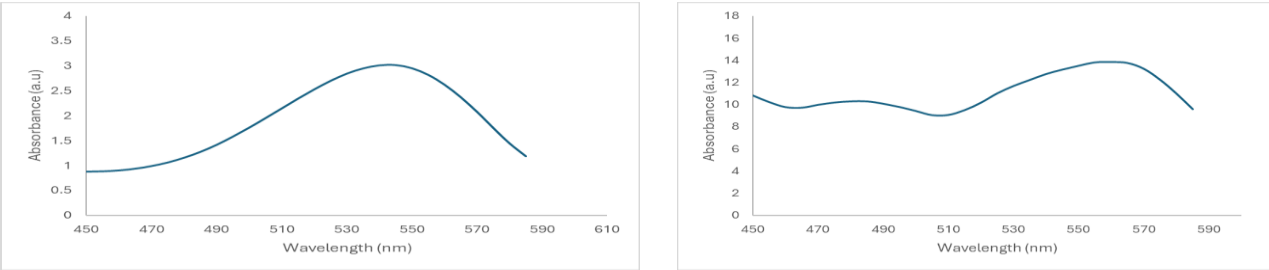
**Table 3** *Functional group and bond position of Roselle solution diluted in Ethanol*

| Concentration (dilute in Ethanol) | Functional group class | Bond position ($cm^{-1}$) |
|---|---|---|
| 0.02 | Carboxylic Compounds $O - H$ | 1736.535, 3.142 |
| 0.04 | Carboxylic Acids $O - H$ | 2973.160,1.901 |
| | Carboxylic Compounds $O - H$ | 1654.999,1.190 |
| | Ester Group $C - O$ | 1086.988,2.072 1044.676,7.388 |
| | Aromatics group $C - H$ | 879.348,2.572 |
| 0.12 | Carboxylic Acids $O - H$ | 2972.824, 1.884 |
| | Ester Group $C - O$ | 1087.035, 2.183 1044.740, 7.490 |
| | Aromatics group $C - H$ | 879.463, 2.766 |

## 2.2 UV-vis spectroscopy

The absorption spectra of Hibiscus sabdariffa, Roselle dye samples diluted in distilled water and ethanol were measured with a UV-Vis Spectrophotometer [12]. Roselle dye extracted using distilled water yielded a light red colored solution as opposed to roselle extracted with ethanol, which yielded a deep red colored solution. According to the table below, the greatest peak of roselle diluted with distilled water absorbs wavelengths ranging from 400 to 600 nm. The peak of the absorption spectra of Hibiscus Sabdariffa Roselle sample diluted in ethanol and distilled water were 540 nm in the range of 450 nm to 600 nm. This absorption range of 400 nm to 600 nm confirmed the existence of roselle anthocyanin pigment. Roselle can potentially be a useful supplier and colorant due to its high anthocyanin content [13].

Fig. 3 depicts the absorption spectrum obtained using UV-Vis for the Hibiscus Sabdariffa Roselle dye solution diluted in ethanol and distilled water as solvent. The results indicate that the optical absorption spectrum peak is just above 460 nm, with an absorbance value slightly below 3 in ethanol while below 13 in distilled water. This suggests a significant level of light absorption by the substance at this wavelength. The concentration of Roselle dye and the solvent (distilled water or ethanol) impact the solution's peak absorbance wavelength, photon energy, and absorption coefficient. Generally, higher Roselle dye concentrations result in higher absorption coefficients, and the choice of solvent can also affect the absorption characteristics [14].




**(a) Ethanol** **(b) Distilled water**

**Fig. 3** *The absorption spectrum of Roselle diluted in (a) ethanol (b) distilled water using UV-Vis Spectroscopy*

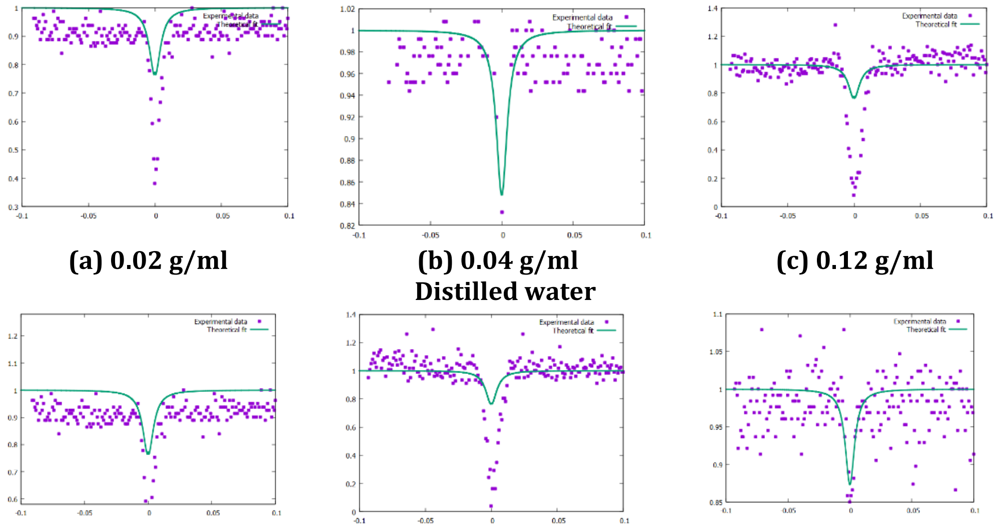
## 2.3 Nonlinear absorption ($\beta$) by Open aperture of Z-scan.

The open aperture z-scan measurements were crucial in discerning the nonlinear optical behavior of the studied Hibiscus Sabdariffa dye. To distinguish between saturable absorption and reverse saturable absorption, it was imperative to analyze the transmittance data as a function of increasing input laser intensity.

Upon careful analysis of the data obtained from the hibiscus sabdariffa roselle sample diluted with distilled water and ethanol, it became evident that the optical nonlinearity observed was primarily attributed to reverse saturable absorption [15]. This phenomenon is characterized by a decrease in transmittance as the absorption coefficient increases when subjected to escalating laser intensities.

**Open aperture data**

Ethanol




**(a) 0.02 g/ml** **(b) 0.04 g/ml** **(c) 0.12 g/ml**

**Distilled water**

**(d) 0.02 g/ml** **(e) 0.04 g/ml** **(f) 0.12 g/ml**

**Fig. 4** *Data analysis of open aperture (a) 0.02 g/ml Roselle + Ethanol,(b) 0.04 g/ml Roselle + Ethanol, (c) 0.12 g/ml Roselle + Ethanol, (d) 0.02 g/ml Roselle + Ethanol, (e) 0.04 g/ml Roselle + Ethanol, (f) 0.12 g/ml Roselle + Ethanol*

The presented figure illustrates the open data from various roselle concentrations (0.02g/ml, 0.04g/ml and 0.12g/ml), demonstrating a clear trend of reverse saturable absorption as shown in Fig. 4. As the input laser intensity increased, the transmittance exhibited a decreasing pattern, indicating a simultaneous increase in the absorption coefficient. This distinctive behavior is a hallmark of reverse saturable absorption and reinforces the notion that the nonlinear optical response is a consequence of the hibiscus sabdariffa roselle dye itself, rather than the choice of solvent used in the dilution process [16].

These findings are significant in the context of potential applications of hibiscus sabdariffa-derived organic dyes in nonlinear optical devices. The observed reverse saturable absorption behavior suggests that the Hibiscus Sabdariffa dye may find utility in optical limiting applications, wherein it effectively attenuates intense laser irradiation [17]. Moreover, the fact that the nonlinearity is intrinsic to the dye and not influenced by the solvent enhances the reliability and reproducibility of these optical characteristics.

In conclusion, this investigation into the third-order nonlinear optical properties of Hibiscus Sabdariffa using the Z-scan method has unveiled a distinctive reverse saturable absorption behavior. The results underscore the potential of hibiscus sabdariffa roselle as a viable and environmentally friendly source for nonlinear optical applications, contributing to the development of sustainable photonic technologies [18]. Further research may explore optimization strategies and delve deeper into the specific mechanisms underlying the observed nonlinearities, paving the way for enhanced utilization of natural dyes in advanced optical devices.

**Closed aperture z-scan**

Ethanol

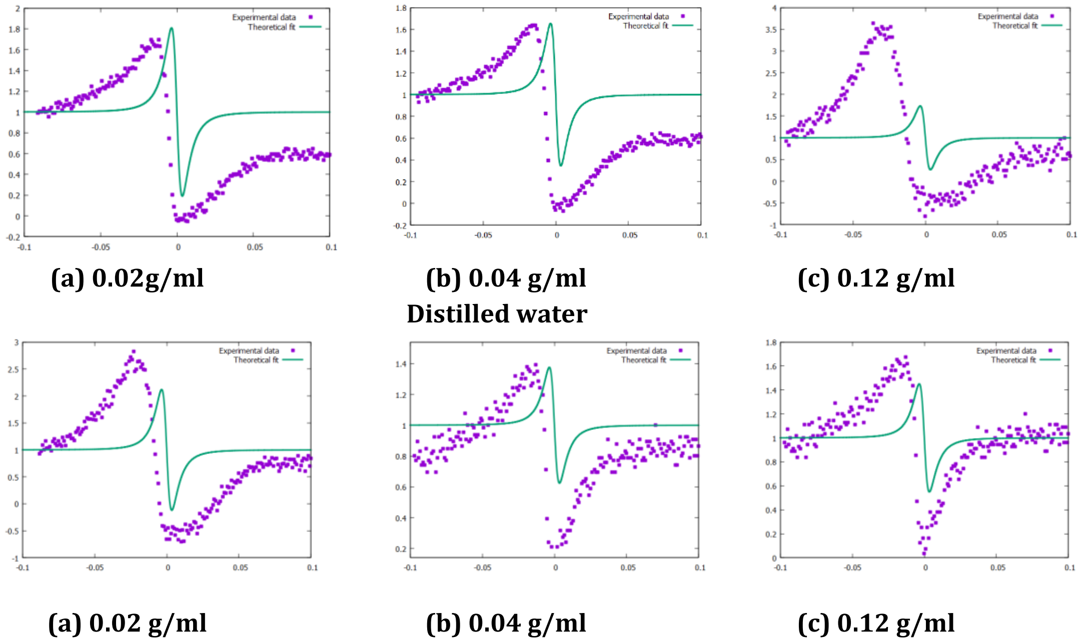



**Fig. 5** *Data analysis of closed aperture (a) 0.02 g/ml Roselle + Ethanol, (b) 0.04 g/ml Roselle + Ethanol, (c) 0.12 g/ml Roselle + Ethanol, (d) 0.02 g/ml Roselle + Ethanol, (e) 0.04 g/ml Roselle + Ethanol, (f) 0.12 g/ml Roselle + Ethanol*

The linear absorption coefficients (α) of the dye solutions were ascertained using UV-vis spectroscopy, employing the U-3900H Spectrophotometer. The linear absorption coefficients (α) for every solution were determined by measuring the absorbance values at a wavelength of 532 nm. For reference, the values that were obtained have been assembled in Table 5.

The closed aperture z-scan measurements on the hibiscus sabdariffa roselle samples, when diluted with distilled water and ethanol, reveal an intriguing asymmetric behavior, indicative of reverse saturable absorption. As demonstrated in the Fig. 5, the differential peak-to-valley transmission and the breadth of associated peaks increase with higher dye concentrations (0.02g/ml, 0.04g/ml and 0.12g/ml), suggesting a concentration-dependent impact on the nonlinear optical response. An exemplary simulation curve, depicted by the dotted line superposed on the z-scan, facilitates the estimation of the refractive index n2 and the third-order nonlinear optical properties of the different hibiscus sabdariffa liquid solutions [19].

The values obtained, as shown in Table 4, indicate a trend of 1 to 2 for roselle diluted in ethanol and a trend of 2 for roselle diluted in distilled water. This trend suggests that as the dye concentration increases, more thermally agitated particles come into play, amplifying the observed nonlinear effects. The negative sign in the closed z-scan profiles signifies the dominance of the thermotic effect in these conditions. The observed nonlinear optical behavior is attributed to a thermal origin, given the temperature dependency of the refractive index. This aligns with comparable behavior demonstrated in a continuous mode (cw Nd:YAG) [20]. The presence of a thermal lens alters the phase of the propagating beam, contributing to the observed nonlinearities.

**Table 4** *Nonlinear absorption coefficient* $(\propto) km^{-1}$ *of sample diluted in ethanol and distilled water.*

| Concentration (diluted in ethanol) | Nonlinear absorption (a.u) |
|---|---|
| 0.02g/ml | 2.29719 |
| 0.04g/ml | 1.3123 |
| 0.12g/ml | 2.3067 |
| **Concentration (diluted in distilled water)** | **Nonlinear absorption (a.u)** |
| 0.02g/ml | 2.3057 |
| 0.04g/ml | 2.3446 |
| 0.12g/ml | 2.4115 |

The subsequent analysis of the refractive index *n*2 of hibiscus sabdariffa roselle samples, measured with the closed aperture of the z-scan, further supports the thermal influence. From table 5, the negative values in the range of -5 to -6 for roselle diluted in distilled water and -8 to -1 for roselle diluted in ethanol suggest a strong thermal optic effect. The limitation in increasing light intensity, to avoid triggering the thermal optic effect, underscores the sensitivity of the material to external stimuli.

**Table 5** *Reflective index n2 (× 10-12cm*$^2$*/W) of sample diluted in ethanol and distilled water*

| Concentration (diluted in ethanol) | Reflective index n2 (× $10^{-12}$cm$^2$/W) |
|---|---|
| 0.02g/ml | -6.144 |
| 0.04g/ml | -4.971 |
| 0.12g/ml | -5.5772 |
| **Concentration (diluted in distilled water)** | **Refractive index n2 (× $10^{-12}$cm$^2$/W)** |
| 0.02g/ml | -8.494 |
| 0.04g/ml | -5.544 |
| 0.12g/ml | -3.420 |

The calculation of the third-order nonlinear optical properties, derived from the data of nonlinear absorption and refractive index, provides additional insights as show in Figure 6 below. For roselle diluted in distilled water, the third-order nonlinear optical properties range from -3, while for roselle diluted in ethanol, the range spans from -3. These values underscore the significant influence of thermal effects on the third-order nonlinear optical properties of hibiscus sabdariffa roselle [20].

**Table 6** *Third-order nonlinear* $X^3$*, of sample diluted in ethanol and distilled water*

| Sample | Concentration | Third-order nonlinear $X^3$ |
|---|---|---|
| Ethanol | 0.02g/ml | -3.8468 |
| | 0.04g/ml | -3.6587 |
| | 0.12g/ml | -3.2705 |
| **Sample** | **Concentration** | **Third-order nonlinear X3** |
| Distilled water | 0.02g/ml | -3.8383 |
| | 0.04g/ml | -3.1994 |
| | 0.12g/ml | -3.1657 |

Self-phase modulation, similar to what happened as in Henari et al state., is likely what created the diffraction type patterns with concentric ring structures that were seen during the z-scan studies and high peak intensities closer to the focus (z ± 0 mm) [21]. Therefore, the 1-mm quartz cell containing the dye solution was positioned distant from the focus for the optical limiting investigations that were carried out on the six Hibiscus Sabdariffa solutions [22]. Displaying the corresponding optical limiting profiles for laser beam input power within the range of 0–40 mW. Two main regions may be distinguished. The first, known as the linear regime, is where the output power follows Lambert-Beer law's linear relationship with the input power, while the second sector is where the output power passes over a plateau. When compared to the input power, the latter's value is extremely low and appears to not go over 3.0 mW for inputs up to 40 mW.

The z-scan technique, which has the benefits of simplicity and high sensitivity as well as enabling simultaneous measurement of the magnitude and sign of the nonlinear refractive index and the nonlinear absorption coefficient of the samples, was used to investigate the nonlinear optical properties in addition to the optical limiting. The procedure basically entails moving a sample across a Gaussian beam's focus and tracking variations in the far field intensity [21].

## 3. Conclusion

In conclusion various concentrations of hibiscus sabdariffa Roselle dye solutions were meticulously prepared using two different solvents, ethanol, and distilled water. The analysis conducted through UV–vis spectroscopy shows distinct absorption bands within the 460–660 nm range. Significantly, this range of absorption bands coincided with the laser excitation wavelength of 532 nm, utilized in the Z-scan method.

As the concentration increased during the open-aperture Z-scan data processing, an interesting Reverse Saturable Absorption (RSA) effect became apparent. This finding highlighted a non-proportional linear correlation between the dye concentration and the β values. While the Hibiscus Sabdariffa Roselle dye was shown to have a self-defocusing effect in the closed-aperture Z-scan characterization [23]

According to these results, hibiscus Sabdariffa Roselle dye solutions have potential uses in optoelectronics and advanced photonics [22], in addition to their use as optical limiters for safety purposes. This brings up new prospects for harnessing Curcumin's unique optical characteristics in cutting-edge.

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## Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

## Author Contribution

*The authors confirm contribution to the paper as follows:* ***study conception and design, data collection, methodology, analysis and interpretation of results:*** *Ainna Syafiqah Juma'in and Nurul Nadia Adnan*, Ganesan Krishnan*. All authors reviewed the results and approved the final version of the manuscript.*

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