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Synthesis, characterization, and nonlinear optical properties of some new series of S-(5-aryl-1,3,4-oxadiazol-2-yl) 2-chloroethanethioate derivatives

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Abstract: In the present investigation, some novel S-(5-aryl-1,3,4-oxadiazol-2-yl)2-chloroethanethioate (**3a**–**3e**) derivatives were synthesized and their impact on optical properties was studied. They have also been characterized by elemental analysis and various spectroscopic methods including FTIR, ^{1}H NMR, ^{13}C NMR, and UV-Vis techniques. The nonlinear refractive indexes of **3a**–**3e** were also measured in dichloromethane via Z-scan method using a continuous wave diode-pumped laser at 532 nm wavelength. The nonlinear refractive coefficient of compounds was obtained from 10^{11} m^2/W order. Regarding the appropriate nonlinearity of these compounds, they could be considered good candidates for biooptical and photonic applications. All the synthesized compounds (**3a**–**3e**) have also been evaluated for their antimicrobial and antifungal activities. The bioactive assay showed that the synthetic compounds displayed variable inhibition zones against tested bacterium *Escherichia coli* and fungus *Aspergillus fumigatus* in comparison to enrofloxacin and amphotericin as reference drugs, which are normally used for treating such infections.

Key words: Synthesis, S-(5-aryl-1,3,4-oxadiazol-2-yl)2-chloroethanethioate, nonlinear optics, Z-scan technique, diode-pumped laser

1. Introduction

Recently, the detection of new materials with significant nonlinearity and optical properties has gained much attention from scientists, especially physicists and chemists. Nonlinear refraction may originate from many different physical sources like electronic molecular, electrostriction, or thermal ones.[1] During the last two decades several branches of materials such as organic molecules,[2,3] organic dyes,[4] π-conjugated organometallic compounds and their derivatives,[5–7] and nanocomposites[8,9] have been investigated because of their exceptional optical properties. Heterocyclic compounds are the largest classical division of medicinal chemistry studies and some other sciences due to their biological activities and industrial importance. Oxadiazole is a versatile heterocyclic nucleus that can be considered as a simple five-membered heterocycle containing two nitrogen and one oxygen atoms. The oxadiazoles exist in different isomeric forms such as 1,2,5-, 1,2,3-, 1,2,4-, and 1,3,4-oxadiazoles. Among them, 1,3,4-oxadiazole derivatives are the most studied ones because of their various chemical and pharmaceutical activities and adaptable intermediates, due to the fact that the thiol group on the oxadiazole ring undergoes nucleophilic substitution reaction easily. The compounds possessing 1,3,4-oxadiazole cores have a broad spectrum of biological properties including antibacterial,[10] antifungal,[11] analgesic and

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antiinflammatory,[12,13] antiviral,[14] anticancer,[15] hypoglycemic,[16] and antimalarial[17] properties. Development of novel chemotherapeutic agents is an important and challenging task for medicinal chemists. The agents originating from heterocyclic compounds are antibiotic-resistant, have good biological properties, and are effective compounds against multidrug-resistant microbial infection. Remarkable nonlinearity arises from donor-acceptor (D-A) electron groups. In this research the effect of donor-acceptor groups in five-ring members was studied comparatively. Some of the investigated materials regarding D-A groups are acetylenes[18] and hetero aromatic rings.[19,20]

Different methods like four-wave mixing, ellipse rotation, and Z-scan technique were used to determine the nonlinearity of materials,[21–23] among which Z-scan is the latest and simplest method for determining both the sign and magnitude of nonlinear coefficients. Since oxadiazole substitutes are known in today's scientific world, their nonlinear optical (NLO) properties are very promising for biooptical applications and use in the field of optoelectronic, such as in optical switching and optical limiting.[24,25]

In the present research, some novel S-(5-aryl-1, 3, 4-oxadiazol-2-yl) 2-chloroethanethioates (**3a**–**3e**) were synthesized and their influence on the optical properties was studied. The new synthesized heterocyclic derivatives have been characterized by elemental analysis and various spectroscopic methods including FTIR, ^{1}H NMR, and ^{13}C NMR techniques. The linear optical properties were also analyzed by UV-Vis spectrophotometer.

In our previous works we reported the synthesis and antibacterial properties of a new series of thioglycoside heterocyclic derivatives of 1,2,4-triazole-5-thiones and 1,3,4-oxadiazole-5-thiones.[26–29] Following the design and synthesis of 1,3,4-oxadiazole moieties linked to choloroactylchloride, the synthesis and nonlinear optical properties of a series of new S-(5-aryl-1,3,4-oxadiazol-2-yl) 2-chloroethanethioate derivatives (**3a**–**3e**) have been reported along with the related antimicrobial activity.

2. Results and discussion

2.1. UV-Vis measurements

The electronic spectra of five samples in the 400–800 wavelength range using UV-Vis spectrophotometer are illustrated in Figure 1. Linear absorption spectra of the samples were associated with the color of organic compounds. Considering that our compounds are white and orange crystals, their absorption spectra are close to each other. The absorption values of compounds at $\lambda = 532\,nm$ are 0.028, 0.008, 0.020, 0.084, and 0.018, respectively.

The linear absorption coefficient α is determined by the following equation:

$$\alpha = \frac{1}{L} LnT. \tag{1}$$

In this equation, L is the thickness of cell equal to 1 cm and T is the transmittance of light from the cuvette.

2.2. Z-scan measurements

The Z-scan technique was proposed by Sheik-Bahae et al. in 1989.[23] Figure 2 illustrates the closed aperture Z-scan graphs of five samples at one incident intensity of laser. The intensity is the same and an aperture of the photodiode at far field has linear transmittance of about 0.1. The curve shows a minimum transmittance (valley) before focus and a maximum transmittance (peak) after focus that indicates formation of a self-defocusing effect. This effect is represented by the negative nonlinear refractive coefficient (n_2). Therefore, the shape of the graph

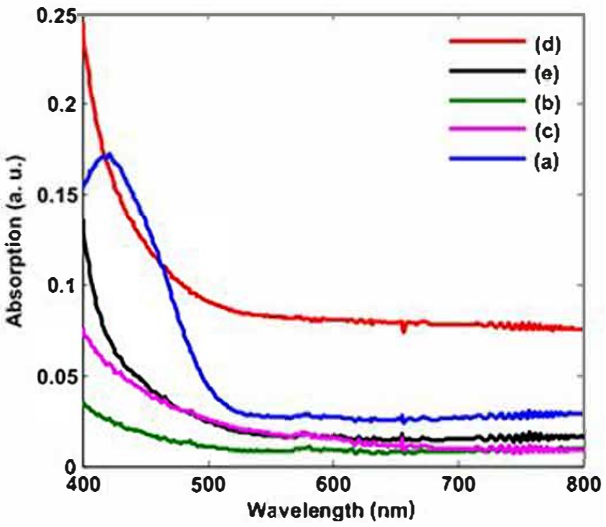

Figure 1. Absorbance spectra of (**a**) Ar = phenyl, (**b**) 2-chlorophenyl, (**c**) Ar = furyl, (**d**) 2-nitrophenyl, and (**e**) 3-nitrophenyl in CH_2Cl_2.

immediately reveals the sign of the refractive index. Because of the CW laser, the temperature rises (ΔT) in the laser spot, which leads to the change in refractive index so that $\Delta n = (dn/dT)\Delta T$, with dn/dT being the thermooptic coefficient of the sample. dn/dT comes from the density change with temperature in the solution. It is also noted that all the sample solutions have negative refractive index. In turn, this provides evidence that the nonlinear refractive index might be related to thermal collection in the samples. In addition, compound **d** has the largest absorbance at 532 nm among the five compounds, and also the magnitude of n_2 in **d** is higher than in others, which gives further evidence that the thermal effect is a dominant mechanism for the nonlinear refractive index. Variations of normalized transmittance for all of the samples were recorded by photodiode.

Using the peak–valley differences (ΔT_{PV}), variations in the phase shift can be calculated by the following equations:

$$\Delta T_{pv} \simeq 0.406\ (1S)^{0.25}\ |\Delta\phi_0| \tag{2}$$

$$\Delta\phi_0 = kn_2 I_0 L_{eff} \tag{3}$$

Here, S is the linear transmittance of aperture of about 0.1 and $\Delta\phi_0$ is variation of phase, $k = 2\pi/\lambda$ is the wave vector, $I_0 = P_0/\pi\omega_0^2$ is the input intensity of the laser, and $L_{eff} = 1e^{\alpha l}/\alpha$ is the effective length of the sample. Calculated values of n_2 are listed in Table 1. The theoretical fit according to Eq. (4) for all of the samples was drawn to prove the appropriate consistency between experimental data and theory.[30]

$$T(z) = 1\frac{4\Delta\phi_0(X)^2}{(1+(X)^2)(9+(X)^2)} \tag{4}$$

Here, $X = z/z_0$. Theoretical fits are plotted at ideal conditions without any errors in measuring the focal spot radius or linear absorption index. Furthermore, there are other uncertainties in estimating exact values, such as fluctuations of laser or voltage sources and contaminations on the cell. Therefore, there are some noises in the experimental curves. Open aperture measurements were recorded, too. Despite good refraction, these

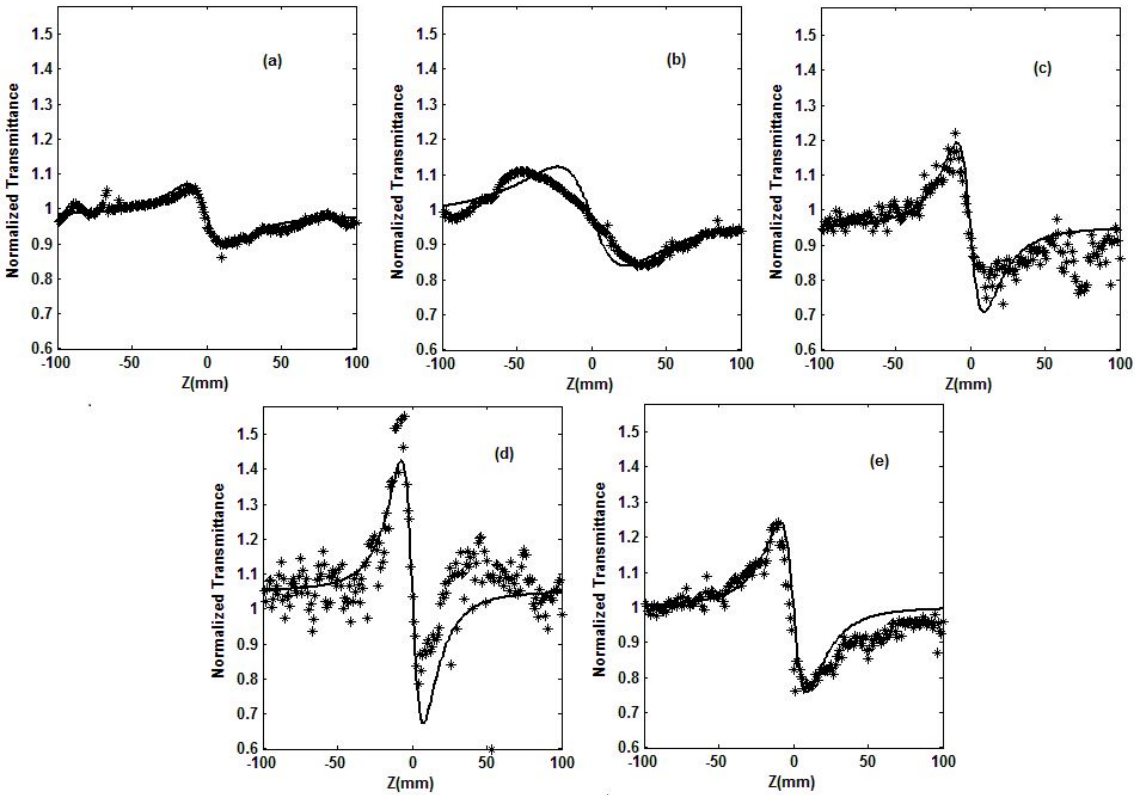

Figure 2. Closed aperture curves of five compounds containing (**a**) Ar = phenyl, (**b**) 2-chlorophenyl, (**c**) Ar = furyl, (**d**) 2-nitrophenyl, and (**e**) 3-nitrophenyl solved in CH_2Cl_2 at I_0= 89.36 kW/m^2 incident intensity of laser beam.

compounds do not have good absorption, so they are neglected. The comparison of normalized transmittance of five compounds as a function of position (z) is plotted in Figure 3.

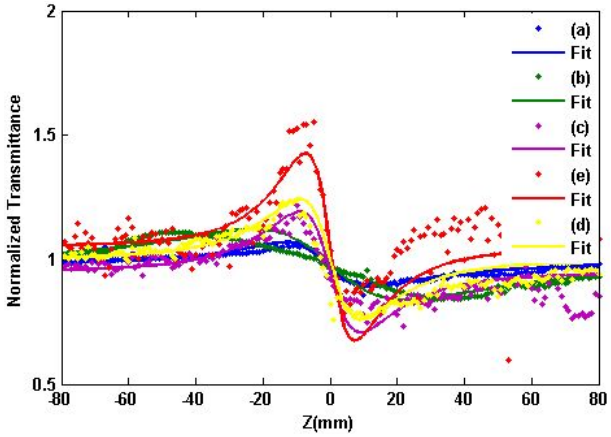

Figure 3. Comparison of normalized transmittance of five compounds containing (**a**) Ar = phenyl, (**b**) 2-chlorophenyl, (**c**) Ar = furyl, (**d**) 2-nitrophenyl, and (**e**) 3-nitrophenyl solved in CH_2Cl_2 at I_0= 89.36 kW/m^2 incident intensity of laser beam.

By comparing closed aperture curves of samples in Figure 3 at one intensity and results of calculations in Table 1, it was observed that the NLO of the new synthesized compounds (**a**–**e**) was increased directly by

Table 1. The measured values of the linear absorption coefficient $\alpha\left(cm^1\right)$, peak–valley difference ΔT_{PV}, and nonlinear refraction coefficients of five compounds containing (**a**) Ar = phenyl, (**b**) 2-chlorophenyl, (**c**) Ar = furyl, (**d**) 2-nitrophenyl, and (**e**) 3-nitrophenyl at I_0= 89.36 kW/m^2.

Sample	$\alpha\left(cm^1\right)$	ΔT_{PV}	$n_2(\times 10^{11}\, m^2/W)$
(**a**) Ar = phenyl	0.065	0.170	–6.08
(**b**) 2-Chlorophenyl	0.020	0.268	–9.89
(**c**) Ar = Furyl	0.047	0.471	–17.35
(**d**) 2-Nitrophenyl	0.193	0.767	–29.21
(**e**) 3-Nitrophenyl	0.041	0.470	–17.39

electron-withdrawing effects of substituting compounds. The NLO property of compound **d** is better than that of compound **e**; due to the nitro position in ortho, which causes molecule formation in more resonance forms, the electron-withdrawing effect of the furyl substitute in compound **c** is more than that of the 2-chloro ones in compound **b** because of oxygen as a more electronegative hetero atom in the furyl ring. It is certain that the electron traction effect of 2-chlorophenyl is stronger than that of the phenyl substitute in compound **a** due to the electronegativity of the chlorine atom.

2.3. Biological studies

The in vitro antibacterial and antifungal activities of the synthesized compounds in CH_3Cl against *Escherichia coli* and *Aspergillus fumigatus* are shown in Tables 2 and 3. The minimal inhibition concentrations (MICs) for enrofloxacin as the reference antibacterial drug and amphotericin were 36 µg/µL and 28 µg/µL in similar test conditions, respectively.

Table 2. In vitro antibacterial activity of synthetic compounds against *E. coli*.

Compound	**3a**	**3b**	**3c**	**3d**	**3e**
MIC (µg/µL)	10	15	0	10	0
MBC (µg/µL)	20	30	0	10	0

As shown in Table 2, these compounds showed higher antibacterial effects in comparison with enrofloxacin and amphotericin, which are normally used for treating such infections. The best results belonged to **3d** and **3a**, which showed higher activity against *E. coli*. A zero value indicates that there were no biological activities.

Table 3. In vitro antifungal activity of synthetic compounds against *Aspergillus fumigatus*.

Compound	**3a**	**3b**	**3c**	**3d**	**3e**
MIC (µg/µL)	0	0	10	0	20
MFC (µg/µL)	0	0	20	0	40

As shown in the Table 3, these compounds showed higher antibacterial effects in comparison with enrofloxacin and amphotericin, which are normally used for treating such infections. The best results antifungal belonged to **3c**, which showed higher activity against *Aspergillus fumigatus*.

2.4. Conclusions

In short, a new series of S-(5-aryl-1,3,4-oxadiazol-2-yl) 2-chloroethanethioate derivatives (**3a**–**3e**) was prepared by reaction of 1,3,4-oxadiazole-5-thione (**2a**–**2e**) moieties with chloroactylcholoride in the presence of potassium carbonate solved in dimethyl-formamide at room temperature. The new compounds were characterized by UV-Vis spectra and nonlinear optical measurements were performed by Z-scan technique. The closed aperture curves profile reveals a self-defocusing effect by negative nonlinear refractive coefficient. However, the pattern is typically observed by thermal lens effect. By comparison of curves, it was concluded that:

$$\Delta T_{PV}\, compound\, \mathbf{d} > \Delta T_{PV}\, compound\, \mathbf{e} > \Delta T_{PV}\, compound\, \mathbf{c} > \Delta T_{PV} compound\, \mathbf{b} > \Delta T_{PV} compound\, \mathbf{a}$$

The NLO of compounds **a**–**e** was increased directly by electron-withdrawing effects of substitutes. Because of existing donor-accepter groups, delocalized π-electrons, etc., they become appropriate candidates for photonic devices and biooptical applications. The antimicrobial results obtained from the novel oxadiazoles derivatives (**3a**–**3e**) revealed that, among the synthesized compounds, **3a** and **3d** and **3c** showed better antibacterial and antifungal activities, respectively.

3. Experimental

3.1. General

The melting points of all compounds were recorded on a Philip Harris C4954718 apparatus without calibration. FTIR spectra were determined on a Thermo Nicolet 670 Nexus spectrometer with KBr pellets. ^{1}H NMR (300 MHz) and ^{13}C NMR (75 MHz) measurements were recorded on a Bruker 300 MHz spectrometer in $CDCl_3$ using TMS as the internal reference. Thin-layer chromatography (TLC) analysis was carried out on silica gel plates. All chemicals were purchased from Merck (Tehran, Iran) and used as received by standard procedures. All of the instruments, chemicals, and solvents were dried according to standard methods. Freshly distilled solvents were used throughout the experiments and anhydrous solvents were dried according to the method of Perrin and Armarego.[31] Microanalyses were performed on a Leco Analyzer 932.

3.2. Synthesis procedures

The main route for the synthesis of 5-substituted-1,3,4-oxadiazole-2-thiols/thiones (**2a**–**2e**) involves an initial reaction between an acylhydrazide (**1a**–**1e**) (0.1 mol) and carbon disulfide (15.2 g, 0.2 mol) in basic alcohol solution (50 mL) followed by acidification of the reaction mixture. The precipitate was filtered, washed thoroughly with cold water, and recrystallized from ethanol to give 1,3,4-oxadiazole-5-thiones (**2a**–**2e**) (yield: 60%–70%). The following compounds were prepared under analogous procedures.[32–34] Thiol-thione tautomerism is known for compounds **2a**–**2e** and one of the forms usually predominates (Figure 4).[35]

3.2.1. Synthesis of S-(-5-aryl-1,3,4 oxadiazole-2-yl)2-chloroethanethioate (3a–3e)

First 1,3,4-oxadiazole-5-thione (0.01 mol) was dissolved in N,N-dimethylformamide (10 mL), and then potassium carbonate (0.3 to 0.5 g) was added to the mixture and stirred for 30 min. Then chloro acetyl chloride (0.01 mol) was added dropwise at 0–5 °C and the mixture was stirred for 3 h at room temperature. The formation of the intermediate was confirmed by observing the TLC using ethyl-acetate : hexane as the mobile phase. After completion of the reaction the mixture was poured into cold water to obtain the product (Figure 4).

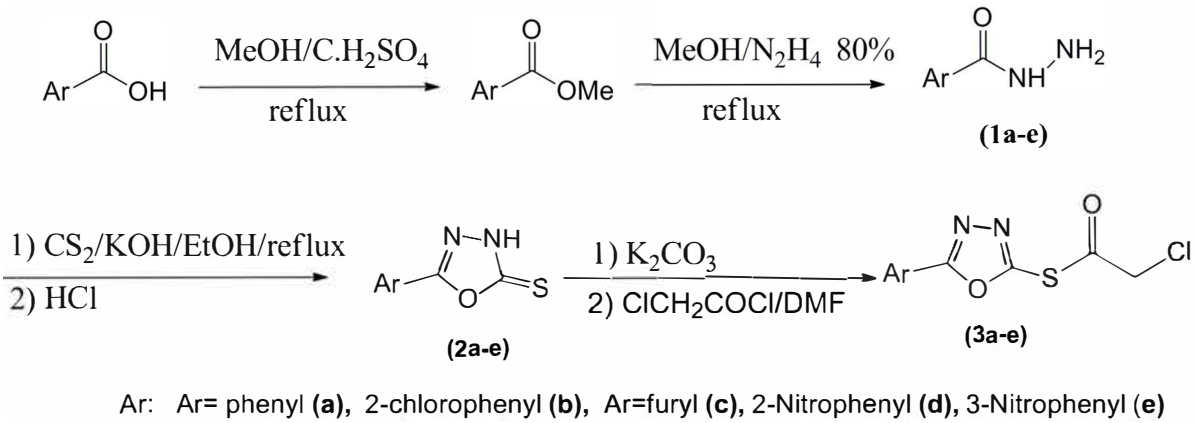

Figure 4. Synthesis of S-(5-aryl-1,3,4-oxadiazol-2-yl) 2-chloroethanethioate derivatives.

3.2.2. Synthesis of S-(5-phenyl-1,3,4-oxadiazol-2-yl) 2-chloroethanethioate (3a)

Yield: 0.192 g, 75%, Lit: mp 160–162 °C; IR (KBr), ?/cm^{-1}: 3440, 2930, 1759, 1622, 1490, 1316, 1051, 968, 753, 689; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 4.84 (2H, s, CH_2), 7.50–7.66 (3H, m, ArH), 7.94–8.02 (2H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 172.50, 162.67, 158.81, 133.43, 132.65, 130.33, 128.24, 127.50, 125.49, 43.44. Calcd: C, 47.16; H, 2.77; N, 11.00; S, 12.59%; Found: C, 47.14; H, 2.75; N, 10.98; S, 12.58%.

3.2.3. Synthesis of S-(5-(2-chlorophenyl)-1,3,4-oxadiazol-2-yl)2-chloroethanethioate (3b)

Yield: 0.22 g, 76%, Lit: mp 149–151 °C; IR (KBr), ?/cm^{-1}: 3444, 2949, 1764, 1615, 1591, 1317, 1032, 932, 729, 684; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 4.84 (2H, s, CH_2), 7.44–7.50 (1H, m, ArH), 7.54–7.63 (2H, m, ArH), 7.95–7.97 (1H, d, J = 7.8 Hz, ArH); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 171.56, 162.63, 156.60, 133.72, 132.90, 132.81, 132.00, 129.92, 128.48, 43.30. Calcd: C, 41.54; H, 2.09; N, 9.69; S, 11.09%; Found: C, 41.53; H, 2.07; N, 9.71; S, 11.08%.

3.2.4. Synthesis S-(5-(furan-2-yl)-1,3,4-oxadiazol-2-yl) 2-chloroethanethioate (3c)

Yield: 0.191 g, 79%, Lit: mp 105–108 °C; IR (KBr), ?/cm^{-1}: 3145, 2942, 1769, 1654, 1466, 1319, 1251, 1071, 945, 589; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 4.80 (2H, s, CH_2), 6.67 (1H, s, furyl), 7.27 (1H, s, furyl), 7.73 (1H, s, furyl); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 171.25, 149.05, 146.39, 116.79, 113.97, 113.79, 43.35. Calcd: C, 39.28; H, 2.06; N, 11.45; S, 13.10%; Found: C, 39.27; H, 2.07; N, 11.43; S, 13.9%.

3.2.5. Synthesis S-(5-(2-nitrophenyl)-1,3,4-oxadiazol-2-yl) 2-chloroethanethioate (3d)

Yield: 0.23 g, 76%, Lit: mp 119–121 °C; IR (KBr), ?/cm^{-1}: 3440, 2949, 1771, 1657, 1536, 1337, 1304, 1253, 1042, 748, 720, 566; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 4.77 (2H, s, CH_2), 7.85–7.91 (3H, m, ArH), 8.14–8.17 (1H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 171.68, 162.48, 159.19, 134.70, 133.08, 132.98, 132.71, 132.63, 132.53, 132.18, 43.30. Calcd: C, 40.08; H, 2.02; N, 14.02; S, 10.70%; Found: C, 40.07; H, 2.03; N, 14.01; S, 10.71%.

3.2.6. Synthesis S-(5-(3-nitrophenyl)-1,3,4-oxadiazol-2-yl) 2-chloroethanethioate (3e)

Yield: 0.21 g, 74%, Lit: mp 134–138 °C; IR (KBr), ?/cm^{-1}: 3332, 2945, 2875, 1723, 1537, 1470, 1354, 1286, 1196, 1028, 718; ^{1}H NMR ($CDCl_3$, 300 MHz) δ (ppm): 4.77 (2H, s, CH_2), 7.77 (1H, t, J = 7.8 Hz, ArH), 8.30 (1H, d, J = 7.5 Hz, ArH), 8.45 (1H, d, J = 8.1, ArH), 8.82 (1H, s, ArH); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 178.23, 167.94, 162.94, 133.32, 133.26, 133.16, 124.94, 44.16. Calcd: C, 40.08; H, 2.02; N, 14.02; S, 10.70%; Found: 40.06; H, 2.03; N, 14.00; S, 10.71%.

3.3. Optical studies

In order to evaluate the sign and the magnitude of nonlinear coefficients simultaneously, the Z-scan technique was used, which was developed by Sheik-Bahae et al.[30] A schematic diagram of the Z-scan setup is shown in Figure 5. As the sample moves towards the focusing point of the lens ($f = 10\ cm$) in the light in the propagating direction (arbitrary z-axis) from –z to +z, the normalized transmittance at far field varies. A photodiode behind an aperture records the data of normalized transmittance. A power meter measures the incident power of the laser beam.

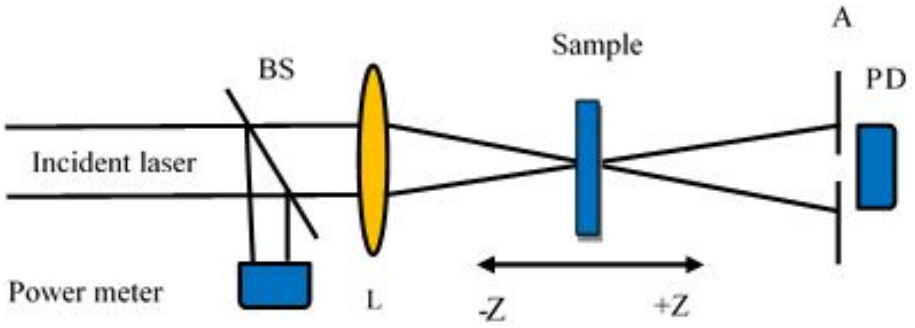

Figure 5. A schematic diagram of Z-scan setup including BS (beam splitter), L (lens), A (aperture), and PD (photodiode).

During the Z-scan measurement, a CW laser at a wavelength of 532 nm was used. All of the samples were liquid, poured into a cell with 6 mm thickness. Since the Rayleigh length ($z_0 = k\omega_0^2/2$) is less than the cell thickness, the thin sample approximation is valid and ω_0 is the beam waist at the focal point ($z = 0$).[30]

3.4. Biological studies

3.4.1. Bacterial strain

The antibacterial and antifungal activities of the compounds were assayed according to our previously published method.[36] The mentioned activities were tested against *E. coli* and *Aspergillus fumigatus*.

3.4.2. Statistical analysis

All statistical analyses were performed with SPSS 11.0 for Windows (SPSS Inc., Chicago, IL, USA).

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