

# A STUDY ON THE OPTICAL PROPERTIES 4-CHLORO-7-METHACRYLOYLOXY COUMARIN COMPOUND IN DIFFERENT CONCENTRATIONS AND SOLVENTS

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## Abstract

*In present work, some optical parameters such as maximum peak position, absorption band edge, direct allowed band gap, refractive index, etc. were determined for 4-chloro-7-methacryloyloxy coumarin (CMAOC) compound in various solvents and concentrations. The absorbance spectra of CMAOC were dominant at the near ultraviolet region. A positive relation was observed between absorbance and concentration. The highest mass extinction coefficient ( $\alpha_{mass}$ ) was obtained for the concentration of 0.007 mM, which was  $131.226 \text{ Lg}^{-1}\text{cm}^{-1}$ . The optical band gap values were found to be 3.457, 3.465 and 3.476 eV for THF, DMSO and DMF solvents, respectively. The experimental refractive index values were compared with those of Moss, Ravindra, Herve-Vandamme, Reddy and Kumar-Singh relations. The experimental refractive index was increased from 2.156 to 2.326 by increasing concentration. It can be say from these results that 4-chloro-7-methacryloyloxy coumarin compound has a semiconductor feature and may be used in some optoelectronic devices such as diode, photo-diode, sensor, etc.*

**Keywords:** Absorption band edge, coumarin compound, optical parameters, optical band gap, refractive index.

## 1. Introduction

As is well known, the majority of organic compounds are heterocyclic compounds. These compounds contain at least one or more heteroatoms in their chemical structure, forming different classes of compounds such as pyridine, pyrrole, furan, thiophene and have a wide range of applications [1]. An important member of this class of compounds containing oxygen atom is coumarins [2]. Coumarins consist of fusion of benzene and pyran rings and are in the lactone class [3]. Many coumarin derivatives with different functional groups are found naturally in the roots, stems and leaves and can be used directly by extraction of these compounds from their sources [4]. On the other hand, many coumarin derivatives can also be synthesized in a laboratory environment using different synthetic methods [5]. In addition to the biological properties of coumarins [6-10], their photophysical and spectroscopic properties [11] are also important. Thanks to these features, they have a wide range of usage in fields such as organic light-emitting diodes [12], electroluminescence [13], fluorescence materials [14], nonlinear optical materials [15], photoalignment of liquid crystalline molecules [16], charge-transfer agents [17], two photon absorption materials [18], organic-inorganic hybrid materials [19] and laser dyes [20].

In addition to these properties of coumarins, their optical properties are remarkable. Because, there are significant pi-conjugate bond systems in the structures of these compounds. The fact that coumarins have this structure gives them a very important advantage [11,21]. In this case, coumarins are included in the class of important candidate compounds for optically sensitive

materials. Generally, the optical characterization of coumarin compounds are accomplished by receiving ultraviolet absorption spectra [22]. In this context, some reported studies can be seen in the literature [11,23-25]. For example, Rabahi and coworkers reported the synthesis and optical characterization with UV-vis and fluorescence spectroscopies of some coumarin derivatives [23]. Besides, the different optical properties of some coumarin derivatives were analyzed by our research group and the results were reported. According to the obtained results, the studied coumarins exhibited semiconductor behavior and might be used optoelectronic devices such as diode and sensor [24,25]. As is just exemplified, various properties of some coumarin compounds have been reported. But, the studies investigating the optical parameters of 4-chloro-7-methacryloyloxy coumarin compound in different solvents and concentrations have not found as a result of our literature research. Herewith, we have described the optoelectronic parameters such as absorbance, transmittance, mass extinction coefficient, absorption band edge, optical band gap, refractive index using various methods for 4-chloro-7-methacryloyloxy coumarin compound different solvents and concentrations. A semiconductor behavior is observed for 4-chloro-7-methacryloyloxy coumarin. This behavior makes it an important candidate for many optoelectronic devices such as diode, photo-diode and sensor, etc.

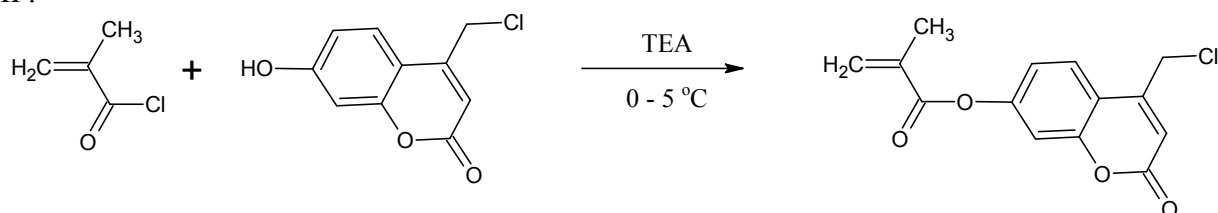
## 2. Experimental

### 2.1. Instrumental Techniques

A Bruker 300 Mhz Ultrashield TM instrument was used to record the nuclear magnetic resonance spectrum ( $^1\text{H-NMR}$ ). The infrared characterization was obtained with a Perkin Elmer Spectrum 100 FTIR spectrometer. The UV measurements of the CMAOC solutions for different solvents and molarities were recorded with a Shimadzu model UV-1800 spectrophotometer using a cylindrical bathtub (Hellma QS-100) in the wavelength 1100-190 nm at room temperature. Therefore, a series of solutions at different molarities were prepared. These concentrations were set at 28.65, 0.358, 0.054, 0.013 and 0.007 mM using 12 mL of DMSO. A digital vortex mixer (Four E's Scientific CO., Ltd.) were used to prepare these solutions.

### 2.2. Materials

Triethyl amine (TEA), methacryloyl chloride, magnesium sulfate ( $\text{MgSO}_4$ ), sodium hydroxide ( $\text{NaOH}$ ), tetrahydrofuran (THF), N,N-dimethylformamide (DMF), dimethyl sulfoxide (DMSO), chloroform were purchased from Sigma-Aldrich. In addition, the following proses was used to synthesize 4-chloro-7-methacryloyloxy (CMAOC) coumarin compound. For this, 4-chloromethyl-7-hydroxy coumarin (5.6039 g) as starting reactive, triethyl amine (2.6917 g) and 75 ml THF were dissolved in a reaction balloon and stirred with external ice water cooling to 0-5 °C temperature range. After that, methacryloyl chloride (2.783 g) in 25 ml THF was added dropwise to this solution and the mixture was stirred with a magnetic stirrer about 10 hours. Then, the reaction mixture was filtered and THF was evaporated with a rotary evaporator. The raw organic products were collected into chloroform phase and extracted several times with dilute  $\text{NaOH}$  solution (3%, w/v). The extracted phase was dried over anhydrous  $\text{MgSO}_4$  overnight, filtered and chloroform was evaporated. The white product 4-chloro-7-methacryloyloxy coumarin (Scheme 1) was recrystallized in THF.



Scheme 1. Synthesis of CMAOC coumarin compound

### 3. Results and Discussions

FTIR spectrum of CMAOC is shown in Figure 1. In this figure, the most characteristic absorption bands are observed for aromatic and aliphatic C-H stretchings ( $3122\text{-}3027\text{ cm}^{-1}$  and  $2989\text{-}2860\text{ cm}^{-1}$ ), carbonyl stretchings of methacrylate and coumarin ring ( $1727\text{ cm}^{-1}$  and  $1702\text{ cm}^{-1}$ ), vinylic and aromatic C=C stretching vibrations ( $1632\text{ cm}^{-1}$  and  $1613\text{ cm}^{-1}$ ). Figure 2 also shows the  $^1\text{H-NMR}$  spectrum of CMAOC. The signals between  $7.9\text{ ppm} - 7.2\text{ ppm}$  are attributed to the aromatic =CH- protons. The aliphatic =CH- proton in coumarin ring is observed at  $6.6\text{ ppm}$ . The  $6.3$  and  $5.9\text{ ppm}$  signals are characteristic for vinylic =CH<sub>2</sub> protons. The signals for -CH<sub>2</sub>Cl protons and -CH<sub>3</sub> protons next to vinyl group are seen at the resonances  $5.0\text{ ppm}$  and  $2.0\text{ ppm}$ , respectively. Other two resonances  $3.3\text{ ppm}$  and  $2.5\text{ ppm}$  are also reasoned from DMSO solvent.

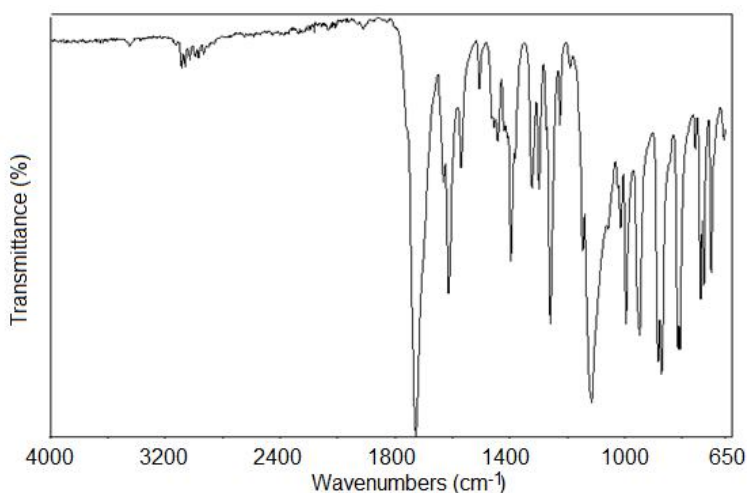


Figure 1. FTIR spectrum of 4-chloro-7-methacryloyloxy coumarin.

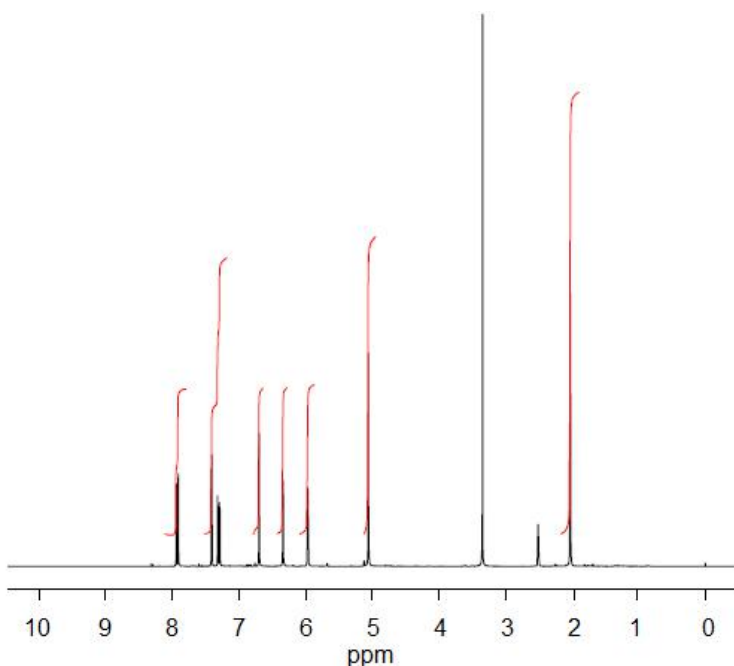


Figure 2.  $^1\text{H-NMR}$  spectrum of 4-chloro-7-methacryloyloxy coumarin.

Figure 3 shows the absorbance spectra of the CMAOC compound in various solvents of THF, DMF, DMSO and also in various concentrations of  $28.65$ ,  $0.358$ ,  $0.054$ ,  $0.013$  and  $0.007\text{ mM}$ , respectively.

As seen in this figure, the CMAOC exhibits two dominant absorbance peaks at 282 nm and 314 nm for THF, DMF and DMSO solvents. The absorbance of the CMAOC is not observed at higher molarities than 0.358 mM. Obtained results show that the absorbance spectra of the CMAOC coumarin compound are dominant at the near ultraviolet (NUV) region. In addition, a positive relation presents between absorbance and concentrations.

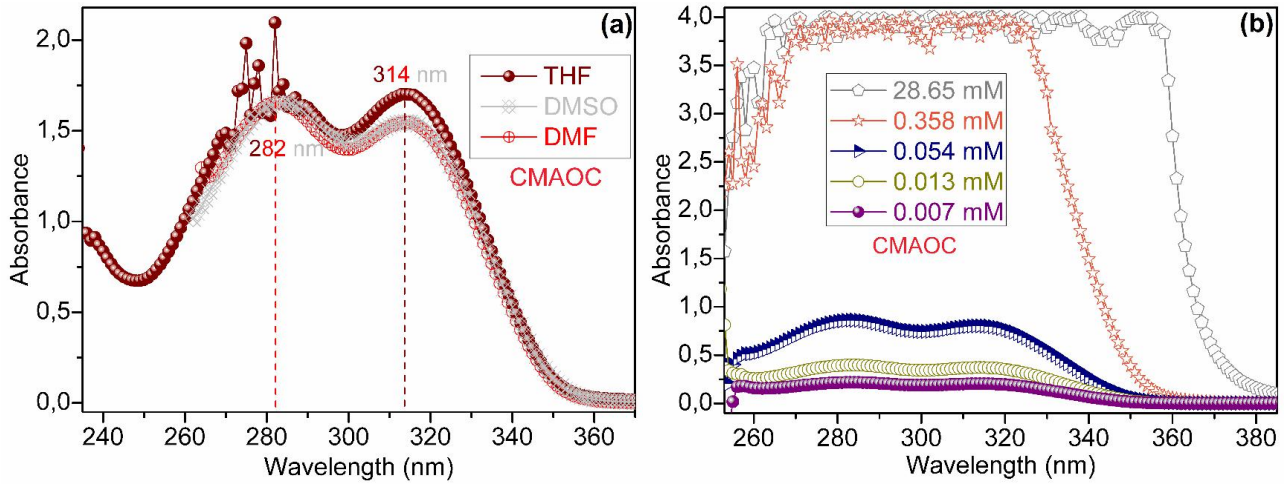


Figure 3. The absorbance spectra for (a) THF, DMF, DMSO solvents and (b) 28.65, 0.358, 0.054, 0.013, 0.007 mM of CMAOC

The mass extinction coefficient ( $\alpha_{mass}$ ) is investigated for optoelectronic applications and is given by [26]

$$\alpha_{mass} = \frac{Abs}{CIM_A} \tag{1}$$

where C is the molar concentration, Abs is the absorbance, l is length of optical path of the used cylindrical bathtub and  $M_A$  is the molecular mass which is 278.603 g/mol for CMAOC. The  $\alpha_{mass}$  values of the CMAOC for different solvents and molarities are calculated using Eq. (1). The plots of the  $\alpha_{mass}$  vs. wavelength ( $\lambda$ ) of the CMAOC compound for the solvents of THF, DMF, DMSO and for the concentrations of 28.65, 0.358, 0.054, 0.013, 0.007 mM are illustrated in Figure 4(a,b), respectively. The  $\alpha_{mass}$  values of the CMAOC for present solvents are similar with each other. On the other hand, as is illustrated in Figure 4b, the  $\alpha_{mass}$  of the CMAOC increases with decreasing molarities. This result shows an inverse relationship between  $\alpha_{mass}$  and molarity. The highest  $\alpha_{mass}$  value of the CMAOC is obtained for the concentration of 0.007 mM, which is  $131.226 \text{ Lg}^{-1}\text{cm}^{-1}$ .

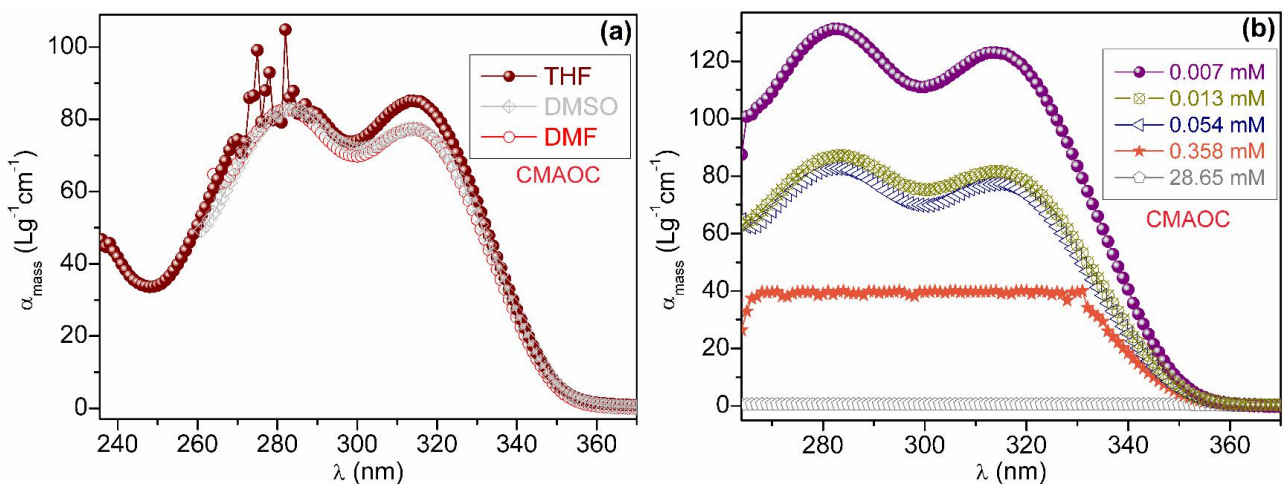


Figure 4. The mass extinction coefficient ( $\alpha_{\text{mass}}$ ) plot vs. wavelength ( $\lambda$ ) for (a) THF, DMF, DMSO solvents and (b) 28.65, 0.358, 0.054, 0.013, 0.007 mM of CMAOC

The transmittance (T) spectra of the CMAOC for THF, DMF and DMSO solvents are shown in Figure 5a where the transmittance spectra are very close to each other for the investigated solvents. Transmittance values sharply increases between the wavelengths about 315 nm - 365 nm. After the wavelength of 365 nm, this increasing suddenly ends and maintains its fixed position. The T curves vs.  $\lambda$  of the CMAOC for 28.65, 0.358, 0.054, 0.013 and 0.007 mM concentrations are shown in Figure 5b. As can be seen from this figure, there is an inverse relationship between the transmittance and molarities. The highest T values are obtained for the concentration of 28.65 mM.

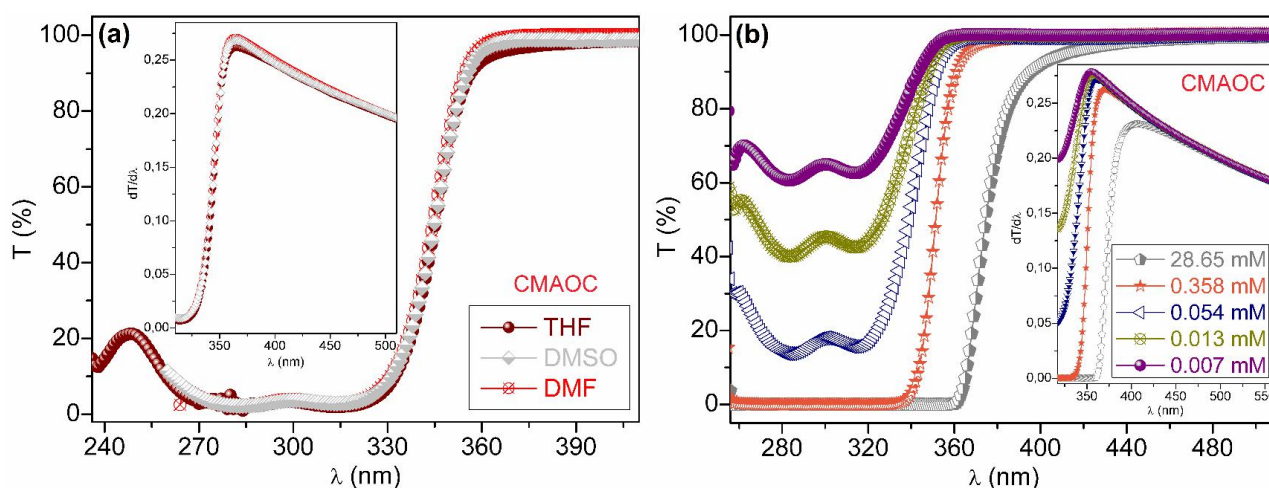


Figure 5. The transmittance (T) spectra for (a) THF, DMF, DMSO solvents and (b) 28.65, 0.358, 0.054, 0.013, 0.007 mM of CMAOC (insert: their  $dT/d\lambda$  plots vs.  $\lambda$ ).

The first derivation ( $dT/d\lambda$ ) vs.  $\lambda$  values have been plotted to determine the absorption band edge ( $E_{\text{Abs-be}}$ ) of CMAOC. The alterations are shown in Figure (5a,b). The  $E_{\text{Abs-be}}$  values for THF, DMF, DMSO solvents and for 28.65, 0.358, 0.054, 0.013, 0.007 mM concentrations are calculated using the maximum peak position ( $\lambda_{\text{max}}$ ) value which is determined from  $dT/d\lambda$  vs.  $\lambda$  plots in Fig 5. The  $\lambda_{\text{max}}$  and  $E_{\text{Abs-be}}$  values of CMAOC compound for present solvents have been given in Table 1. The  $E_{\text{Abs-be}}$  value of CMAOC for THF is determined to be 3.397 eV while that value for DMF is 3.416 eV. The  $E_{\text{Abs-be}}$  value of CMAOC is the lowest value for THF and also is the highest value for DMF. The  $E_{\text{Abs-be}}$  values corresponding to various concentrations are also seen in Table 1. It can be seen from this table that there is a negative relation between  $E_{\text{Abs-be}}$  values and concentrations. values. There is an increase in  $E_{\text{Abs-be}}$  values from 3.062 eV to 3.503 eV due to the change in concentration from 28.65 mM to 0.007 mM. Obtained results suggest that solvents and concentrations have an important role on the absorption band edge of CMAOC compound.

Table 1. Some optical parameters for different solvents and molarities

Solvents	$\lambda_{\text{max}}$ (nm)	$E_{\text{Abs-be}}$ (eV)	$E_{\text{gd}}$ (eV)
THF	365	3,397	3,457
DMSO	364	3,407	3,465
DMF	363	3,416	3,476
Molarities (mM)	$\lambda_{\text{max}}$ (nm)	$E_{\text{Abs-be}}$ (eV)	$E_{\text{gd}}$ (eV)
28.65	405	3,062	3,120
0.358	370	3,351	3,422

0.054	361	3,435	3,501
0.013	358	3,464	3,526
0.007	354	3,503	3,558

$\lambda_{max}$  : maximum peak position,  $E_{Abs-be}$  : absorption band edge,  $E_{gd}$  : direct allowed band gap

Optical band gap ( $E_g$ ) is a fundamental optical parameter and the  $E_g$  of optical transitions can be obtained using Tauc relation [27];

$$(\alpha h\nu) = A(h\nu - E_g)^n \tag{2}$$

where  $h\nu$  is photon energy,  $\alpha$  is absorption coefficient,  $A$  is a constant and  $n$  is a parameter, which measure type of band gaps. For CMAOC compound, the type of band gaps [28] is the direct allowed band gap ( $E_{gd}$ ). According this, we have plotted the  $(\alpha h\nu)^2$  plots vs  $E$  for the solvents of THF, DMF, DMSO and also for the concentrations of 28.65, 0.358, 0.054, 0.013 and 0.007 mM. These plots are illustrated in Figure 6(a,b), respectively. From the linear regions of the Figure 6(a,b), the optical band gap ( $E_{gd}$ ) values are calculated and listed in Table 1 for all solvents and concentrations. The optical band gap values have been found to be 3.457 eV, 3.465 eV and 3.476 eV for THF, DMSO and DMF solvents, respectively. The  $E_{gd}$  value determined in case of DMF solvent is the highest level. As seen in Table 1, the  $E_{gd}$  value of the CMAOC for the concentration of 28.65 mM is the lowest (3.120 eV) value whereas that for 0.007 mM is the highest value (3.558 eV). Obtained these results show that the lowest direct allowed band gap of CMAOC is obtained with THF solvent and also this parameter decreases depend on the increasing concentration.

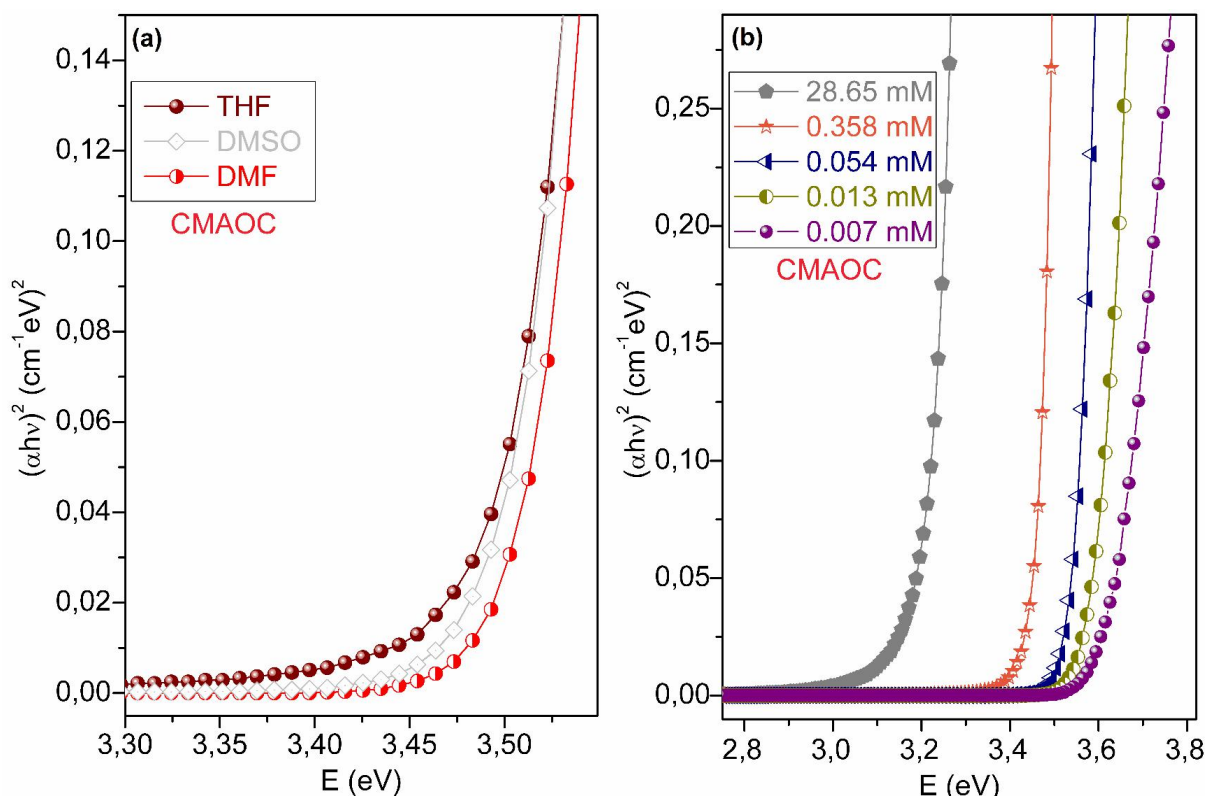


Figure 6. The  $(\alpha h\nu)^2$  plot vs. photon energy ( $E$ ) for (a) THF, DMF, DMSO solvents and (b) 28.65, 0.358, 0.054, 0.013, 0.007 mM of CMAOC

The refractive index or index of refraction ( $n$ ) is a fundamental optical parameter for optoelectronic applications. The  $n$  values can be obtained by [26,29],



$$n = \left\{ \left[ \frac{4R}{(R-1)^2} - k^2 \right]^{1/2} - \frac{R+1}{R-1} \right\} \quad (3)$$

The refractive index values of CMAOC compound for the solvents of THF, DMF, DMSO and the concentrations of 28.65, 0.358, 0.054, 0.013 and 0.007 mM are calculated using Eq. (3) from the experimental data. The obtained results are also listed in Table 2. As is seen in this table, the lowest experimental refractive index value of CMAOC compound is determined in case of DMF solvent to be 2.187. Additionally, the refractive index values show an alteration depending on the concentration. An increasing on the refractive index was observed from 2.156 to 2.326 by increasing in the concentration.

Table 3. The refractive index parameters obtained from various relations

Solvents	Exp	M	Ra	H-V	Re	K-S
THF	2,21 6	2,290	1,941	2,203	2,657	2,257
DMSO	2,205	2,288	1,936	2,201	2,655	2,256
DMF	2,187	2,287	1,929	2,199	2,653	2,253
Molarities (mM)	Exp	M	Ra	H-V	Re	K-S
28.65	2,326	2,349	2,150	2,293	2,734	2,333
0.358	2,194	2,295	1,962	2,212	2,664	2,265
0.054	2,182	2,282	1,913	2,192	2,647	2,248
0.013	2,161	2,278	1,898	2,186	2,642	2,243
0.007	2,156	2,273	1,878	2,178	2,635	2,236

*Exp:experimental, M:Moss, Ra:Ravindra, H-V:Herve-Vandamme, Re:Reddy, K-S:Kumar-Singh*

The optical band gap and refractive index relationship may be explained by various methods as Moss, Ravindra, Herve-Vandamme, Reddy and Kumar-Singh [30]. These methods are represented by the following equations:

Moss relation is given by:

$$n^4 E_g = 95 eV, \quad (4)$$

Ravindra relation is given by:

$$n = 4.084 - 0.62 E_g \quad (5)$$

Herve-Vandamme relation is given by:

$$n^2 = 1 + \left( \frac{A}{E_g + B} \right)^2 \quad (6)$$

where A is the hydrogen ionization energy 13.6 eV and B = 3.47 eV is a constant.

Reddy relation is given by:

$$n^4 (E_g - 0.365) = 154. \quad (7)$$

Kumar-Singh relation is given by

$$n = \frac{3.3668}{(E_g)^{0.82284}} \quad (8)$$

The refractive index curves derived from these equations for CMAOC compound in the solvents of THF, DMF, DMSO and also the concentrations of 28.65, 0.358, 0.054, 0.013, 0.007 mM are illustrated in Figure 7(a,b). The obtained results for these methods have been listed in Table 2. The highest refractive index value is obtained as 2.657 in case of THF solvent and using Reddy equation whereas the lowest value is 1.929 for DMF solvent and Ravindra equation. In general, an increase in refractive index values is observed depending on the concentration increasing for all methods.

The highest refractive index value among these methods is obtained in Reddy equation with a value of 2,734.

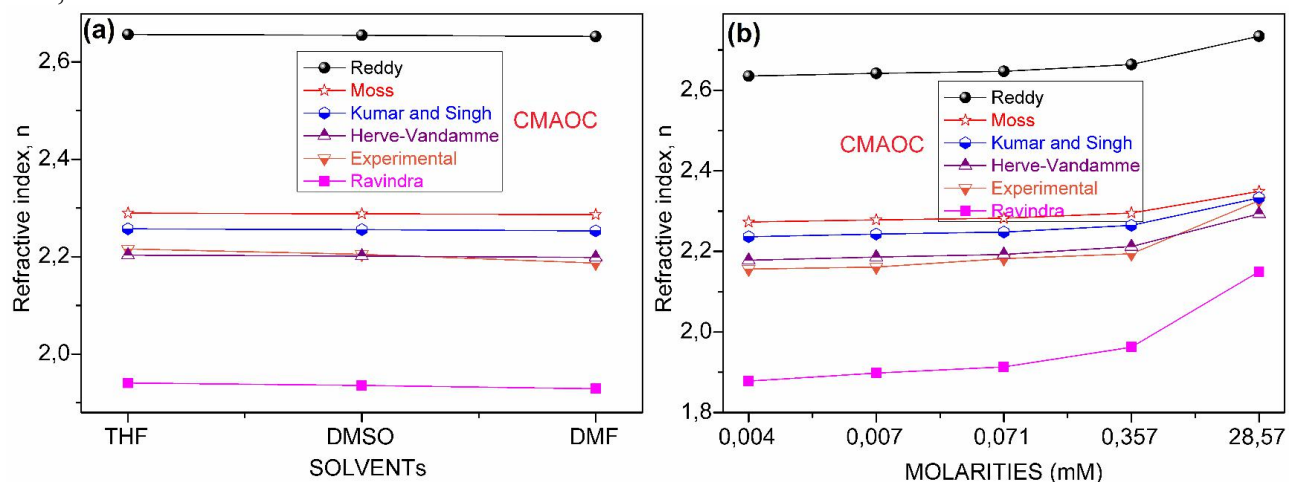


Figure 7. The refractive index plots obtained from various relations for (a) THF, DMF, DMSO solvents and (b) 28.65, 0.358, 0.054, 0.013, 0.007 mM of CMAOC

## Conclusion

A coumarin derivative compound, 4-chloro-7-methacryloyloxy coumarin (CMAOC), was synthesized in order to test the concentration and solvent effect on its optical properties. The absorbance spectra of CMAOC were dominant at the near ultraviolet region. A positive relation was observed between absorbance and concentration. The highest mass extinction coefficient ( $\alpha_{\text{mass}}$ ) was obtained for the concentration of 0.007 mM, which was  $131.226 \text{ Lg}^{-1}\text{cm}^{-1}$ . An inverse relation was observed between the highest mass extinction coefficient ( $\alpha_{\text{mass}}$ ) and molarity. There was an increase in the absorption band edge ( $E_{\text{Abs-be}}$ ) values from 3.062 eV to 3.503 eV due to the change in concentration from 28.65 mM to 0.007 mM. The optical band gap values were found to be 3.457, 3.465 and 3.476 eV for THF, DMSO and DMF solvents, respectively. The highest refractive index value was obtained as 2.657 in case of THF solvent and using Reddy equation whereas the lowest value was 1.929 for DMF solvent and Ravindra equation. 4-chloro-7-methacryloyloxy coumarin compound showed a semiconductor feature and might be used in some optoelectronic devices such as diode, photo-diode, sensor, etc.



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