

Research Article

A Way to Improve Luminescent Efficiency of Bis-Chalcone Derivatives

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Chalcone related compounds have been reported as a poor luminescence molecule due to the quenching processes from the intramolecular torsional motions and *cis-trans* isomerization in the α,β -unsaturated ketone moiety. Despite this limitation, we found a way to improve the luminescent efficiency of our bis-chalcone derivative. In this project, two series of bis-chalcone compounds have been synthesized through Claisen-Schmidt condensation by reacting terephthalaldehyde or 2,5-dimethoxyterephthalaldehyde with the respective R-acetophenone [where R = H (**1a** and **2a**) and *ortho*-hydroxy (**1b** and **2b**)] in 1:2 mole ratio. The presence of a methoxy (OMe) substituent on the central phenyl ring of bis-chalcone has weakened the C=C bond at the α,β -unsaturated ketone moiety of **2a** and **2b**. Interestingly, the OMe group has improved the emission efficiency of the bis-chalcone; that is, the quantum yield of **1a** in DCM solution was not able to be determined due to poor luminescence, but the quantum yield of **2a** in DCM solution was improved to 0.57. In addition, compound **2a** also shows solvatochromism effect where the λ_{\max} emission shifted from 499 nm in nonpolar solvents (benzene) to 523 nm in polar solvents (acetonitrile). This work provides another way to improve the emission efficiency of chalcone related compounds apart from using the complexation method which has been reported before.

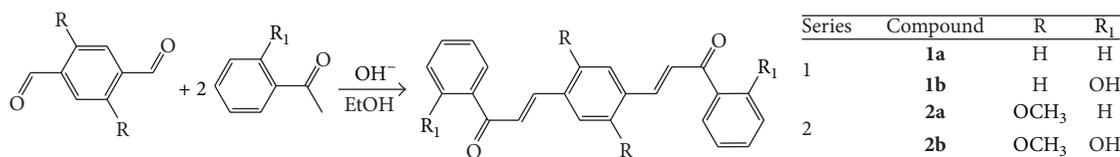
1. Introduction

Chalcone and its derivatives have been known to exhibit antimicrobial, antitumor, anti-HIV, antimalarial, anti-inflammatory, and anticarcinogenic activities [1–6]. The α,β -unsaturated ketone (C=C-C=O) moiety in the chalcone structure plays an important role in chalcone derivative compounds' biological activities. Selvi and coworkers (2012) found that most of these biological effects are related to the ability to create an electrophilic site that is then able to act as a binding site for biological targets [7].

Apart from the biological activities, the photophysical properties of chalcone derivatives also attracted considerable attention from both chemists and physicists. For example, chalcone derivatives have been reported in relation to nonlinear optics (NLO), photorefractive polymers, holographic recording materials, and fluorescent probes for the sensing

of metal ions [8–14]. However, one of the main limitations in the photophysical properties of chalcones is their weak luminescent efficiency, which is due to quenching processes from the intramolecular torsional motions and *cis-trans* isomerization in the α,β -unsaturated ketone moiety [15, 16].

Unlike the biological properties, the photophysical properties of chalcone derivatives such as 2'-hydroxychalcones have still not been fully understood. This could be the reason for limited use of chalcone derivatives and complexes in current green technology devices. D'Aléo and his coworkers (2012) found that the boron complexes with 2'-hydroxychalcone derivatives can increase the fluorescent efficiency from 0.02 to 0.79 by substituting different electron donor groups at the 2'-hydroxychalcone moiety [17]. Herein, we report two series of bis-chalcone derivatives (Scheme 1) in which the emission efficiency also showed dramatic improvement after addition of the methoxy (OMe) moiety on the central

SCHEME 1: Synthesis of bis-chalcones **1a–2b**.

phenyl ring. This work provides another way to improve the emission efficiency of chalcone related compounds apart from using the complexation method as reported by D'Aléo et al. (2012). Compounds **1a** and **1b** were reported by a number of researchers; however, the compounds were mainly used as a starting material for other compounds' synthesis and also were studied concerning their biological activities [18–25], yet, the photophysical properties of **1a** and **1b** were not the main concern in these papers.

2. Materials and Methods

2.1. Characterizations. Melting point was measured using open capillary in Stuart MP3 melting point apparatus. UV-Vis spectra were recorded in dichloromethane (DCM) or dimethyl sulfoxide (DMSO) solution with a Jasco V-630 ultraviolet spectrophotometer. Infrared spectra were recorded on KBr discs using Thermo Scientific Nicolet iS10 Fourier-Transform Infrared Spectrophotometer in the range of 4000–370 cm^{-1} at room temperature. The ^1H and ^{13}C -NMR spectra were recorded on a JEOL 500 MHz-NMR spectrometer using $\text{DMSO}-d_6$ and CDCl_3 which were set at 2.50 and 7.25 ppm as standard reference of the solvent. Elemental analyses were performed using a Thermo Scientific FLASH 2000 HT Elemental Analyser operated by Eager 300 software.

X-ray measurements for compound **1b** were performed on a Bruker SMART diffractometer equipped with a graphite monochromated Mo K α ($\lambda = 0.71073$) radiation source and a CCD detector. Frame integration was performed using the program SAINT [28]. The structure was resolved using a direct method provided by the program package SHELXTL-97 and refined using full matrix least square against F2 for all data [29]. All nonhydrogen atoms were refined anisotropically. All hydrogen atoms were introduced at idealized positions and were allowed to refine isotropically.

2.2. Quantum Yield and Lifetime Measurements of Compound 2a. The fluorescence quantum yield of **2a** in DCM solution was measured using a calibrated integrating sphere (inner diameter: 150 mm) from Edinburgh Instruments FLSP920 spectrometer. The lifetime of this compound was recorded in FLS980 spectrometer using time-correlated single-photon counting (TCSPC) method. The DCM solution of compound **2a** was excited with pulsed laser diode at the wavelength of 376 nm in the lifetime measurement.

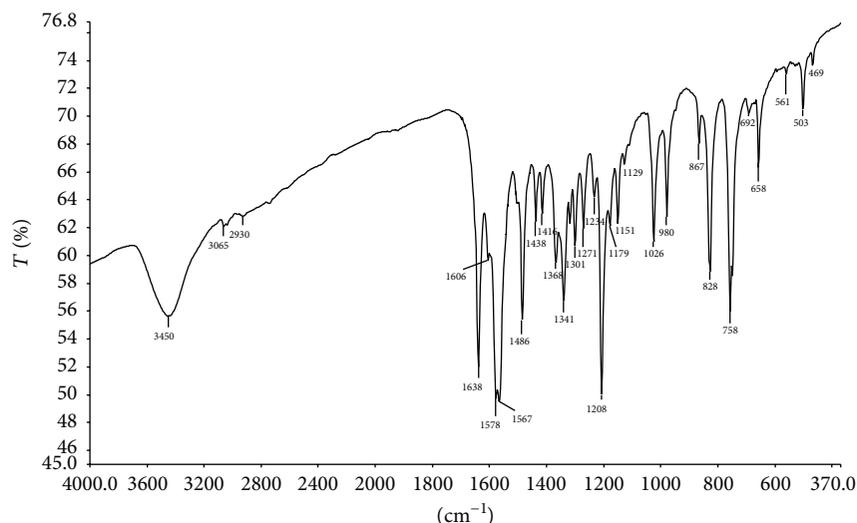
2.3. General Preparation Procedures of Bis-Chalcones 1a–2b. 10 mL of 10% KOH was added to a 125 mL conical flask that contained a solution of R-acetophenone (2 eq. mol) [where R = H (**1a** and **2a**) and *ortho*-OH (**1b** and **2b**)] in 20 mL

ethanol. The mixture was stirred for 5 min and added to 10 mL ethanolic solution of terephthaldehyde for the synthesis of bis-chalcones **1a** and **1b** (0.67 g, 5 mmol) or 2,5-dimethoxyterephthaldehyde for the synthesis of bis-chalcones **2a** and **2b** (0.20 g, 1 mmol). The mixture was stirred vigorously for 24 h at room temperature. Then, HCl (3.0 M) was added dropwise until the mixture solution was neutralized. The precipitated solid was filtered and then washed with ethanol and hot water and dried under reduced pressure. The solid was purified *via* recrystallization from the slow diffusion of hexane into the DCM solution.

3-[4-(3-Oxo-3-phenylpropenyl)-phenyl]-1-phenyl-propenone, 1a [18–24, 30]. Yellow solid. Yield: 1.58 g, 93%. M.P.: 193.7–195.4°C. IR (KBr, cm^{-1}): 1655 ($\nu\text{C=O}$), 1606 ($\nu\text{C=C}$), 1579 and 1445 ($\nu\text{C=C}$, aromatic ring). ^1H -NMR (500 MHz, CDCl_3) δ : 8.02 (d, 4H, $J = 8$ Hz, Ar-H), 7.80 (d, 2H, $J = 16$ Hz, $\text{CH}=\text{CH}_\beta$), 7.68 (s, 4H, Ar-H), 7.59 (t, 2H, $J = 6$ Hz, Ar-H), 7.57 (d, 2H, $J = 16$ Hz, $\text{CH}_\alpha=\text{CH}$), 7.51 (t, 4H, $J = 7$ Hz, Ar-H), 6.96 (t, 2H, $J = 7$ Hz, Ar-H). ^{13}C -NMR (125 MHz, CDCl_3) δ : 189.9, 143.5, 138.0, 136.8, 133.0, 129.0, 128.7, 128.5 and 123.0. UV-Vis (DCM) ($\lambda_{\text{max}}/\text{nm}$): 270 and 348. Anal. Cal. for $\text{C}_{24}\text{H}_{18}\text{O}_2$: C, 85.18; H, 5.36; Found: C, 85.23; H, 5.37.

1-(2-Hydroxyphenyl)-3-[4-[3-(2-hydroxyphenyl)-3-oxo-propenyl]-phenyl]-propenone, 1b [25, 30]. Yellow solid. Yield: 1.45 g, 80%. M.P.: 235.4–236.9°C. IR (KBr, cm^{-1}): 3450 (νOH), 1638 ($\nu\text{C=O}$), 1606 ($\nu\text{C=C}$), 1567 and 1486 ($\nu\text{C=C}$, aromatic ring). ^1H -NMR (500 MHz, CDCl_3) δ : 12.74 (s, 2H, OH), 7.93 (d, 2H, $J = 8$ Hz, Ar-H), 7.92 (d, 2H, $J = 15$ Hz, $\text{CH}=\text{CH}_\beta$), 7.73 (s, 4H, Ar-H), 7.71 (d, 2H, $J = 15$ Hz, $\text{CH}_\alpha=\text{CH}$), 7.51 (t, 2H, $J = 6$ Hz, Ar-H), 7.04 (d, 2H, $J = 8$ Hz, Ar-H), 6.97 (t, 2H, $J = 7$ Hz, Ar-H). ^{13}C -NMR (125 MHz, $\text{DMSO}-d_6$) δ : 193.5, 143.5, 161.8, 143.7, 136.7, 136.4, 130.9, 129.7, 123.0, 120.8, 119.2 and 117.8. UV-Vis (DCM) ($\lambda_{\text{max}}/\text{nm}$): 285 and 383. Anal. Cal. for $\text{C}_{24}\text{H}_{18}\text{O}_4$: C, 77.82; H, 4.90; Found: C, 77.85; H, 4.73.

3-[4-(3-Oxo-3-phenylpropenyl)-phenyl]-2,5-dimethoxyphenyl-propenone, 2a. Yellowish green solid. Yield: 0.36 g, 80%. M.P.: 242.1–243.8°C. IR (KBr, cm^{-1}): 1639 ($\nu\text{C=O}$), 1577 ($\nu\text{C=C}$), 1564 & 1484 ($\nu\text{C=C}$, aromatic ring), 1180 (νOCH_3). ^1H -NMR (500 MHz, $\text{DMSO}-d_6$) δ : 8.06 (d, 2H, $J = 16$ Hz, $\text{CH}=\text{CH}_\beta$), 8.03 (d, 4H, $J = 7$ Hz, Ar-H), 7.72 (s, 2H, Ar-H), 7.67 (d, 2H, $J = 16$ Hz, $\text{CH}_\alpha=\text{CH}$), 7.60 (t, 4H, $J = 8$ Hz, Ar-H), 7.53 (t, 2H, $J = 8$ Hz, Ar-H), 7.16 (d, 2H, $J = 8$ Hz, Ar-H), 3.96 (s, 6H, OCH_3). ^{13}C -NMR (125 MHz, $\text{DMSO}-d_6$) δ : 189.9, 152.8, 138.1, 133.3, 129.4, 129.0, 127.1, 123.7, 111.7, 110.6 and 57.3. UV-Vis (DCM) ($\lambda_{\text{max}}/\text{nm}$): 268, 333 and 421. Anal. Cal. for $\text{C}_{26}\text{H}_{22}\text{O}_4$: C, 78.37; H, 5.57; Found: C, 77.75; H, 5.54.

FIGURE 1: IR spectrum of compound **1b**.

1-(2-Hydroxyphenyl)-3-[4-[3-(2-hydroxyphenyl)-3-oxo-propenyl]-2,5-dimethoxyphenyl]-propenone, **2b**. Orange solid. Yield: 0.36 g, 76%. M.P.: 252.6–253.4 °C. IR (KBr, cm^{-1}): 3449 (ν_{OH}), 1633 ($\nu_{\text{C=O}}$), 1578 ($\nu_{\text{C=C}}$), 1561 and 1489 ($\nu_{\text{C=C}}$, aromatic ring), 1197 (ν_{OCH_3}). $^1\text{H-NMR}$ (500 MHz, CDCl_3) δ : 12.74 (s, 2H, OH), 7.93 (d, 2H, $J = 8$ Hz, Ar-H), 7.92 (d, 2H, $J = 15$ Hz, $\text{CH}=\text{CH}_\beta$), 7.73 (s, 2H, Ar-H), 7.71 (d, 2H, $J = 15$ Hz, $\text{CH}_\alpha=\text{CH}$), 7.51 (t, 2H, $J = 6$ Hz, Ar-H), 7.04 (d, 2H, $J = 8$ Hz, Ar-H), 6.96 (t, 2H, $J = 6$ Hz, Ar-H). $^{13}\text{C-NMR}$ (125 MHz, CDCl_3) δ : 194.1, 163.6, 153.4, 140.1, 136.4, 129.7, 126.6, 122.4, 120.2, 118.8, 118.6, 112.2 and 56.3. UV-Vis (DCM) (λ_{max} /nm): 275, 350 and 445. Anal. Cal. for $\text{C}_{26}\text{H}_{22}\text{O}_6$: C, 72.55; H, 5.15; Found: C, 71.79; H, 4.95.

3. Results and Discussion

3.1. Spectroscopic Analyses. The formation of bis-chalcones can be represented by the presence of α,β -unsaturated ketone (C=C-C=O) bands at 1633–1665 cm^{-1} for C=O and 1577–1606 cm^{-1} for C=C in the IR spectra. The IR frequency of C=O group is lower than the typical value (ca. 1700 cm^{-1}) because of the conjugation effect along the C=C-C=O moiety. The presence of the electron donor and acceptor at the *para*- and *ortho*-positions of the wing phenyl rings shifted the energy of the carbonyl group and C=C aliphatic group. For example, for bis-chalcone **1a**, the C=O band was present at the frequency of 1655 cm^{-1} , but a lower frequency of C=O band (ca. 1638 cm^{-1}) was observed in bis-chalcone **1b**, which has *ortho*-OH as the substituent (Figure 1). In addition, the presence of methoxy (OMe) substituent on the central phenyl ring also shifts the IR frequency of C=C in bis-chalcones **2a** and **2b** (1583–1578 cm^{-1}) to a lower frequency compared to compounds **1a** and **1b** (1606 cm^{-1}). This is due to the electron donation from OCH_3 group to the C=C aliphatic moiety which consequently weakens the C=C bond.

The H_α and H_β protons of bis-chalcone appear as two doublets in the ranges of 7.6–8.2 ppm (H_α) and 7.5–7.8 ppm

(H_β) with the coupling constant (J) value of 15–16 Hz (Figure 2). The large J value of these two protons clearly indicates that compounds **1a–2b** are in the *trans*-conformation. For the $^{13}\text{C-NMR}$ spectrum, the carbonyl carbon (C=O) of the bis-chalcones usually appears at the region of 189.9 to 194.1 ppm. The presence of 2'-hydroxyl group at the wing phenyl rings shifted the carbon resonance of C=O from 190 ppm for compound **2a** downfield by 4 ppm at 194 ppm for compound **2b**. This is due to the formation of hydrogen bond in between the 2'-hydroxyl and oxygen in the carbonyl groups.

3.2. X-Ray Crystallography of Bis-Chalcone 1b. The molecular structure of bis-chalcone **1b** was previously reported by Gaur and Mishra in 2013 [25]. The yellow crystal of bis-chalcone **1b** was grown in DCM solution *via* slow diffusion in hexane under ambient conditions. The molecular structure of bis-chalcone **1b** is shown in Figure 3 and the main crystallographic parameters are presented in Table 1. Bis-chalcone **1b** crystallized into monoclinic crystal system with the space group of $P2_1/n$. The bond length of C6–C7 is about 1.325 Å, which is close to the typical C=C bond (1.34 Å). The single bonds of C4–C5, C5–C6, and C7–C8 are in the range of 1.459–1.467 Å, which is relatively shorter than the typical single bond (1.54 Å). This is due to the fact of the conjugated system present along the molecule [31]. On the other hand, supporting the discussion of the $^{13}\text{C-NMR}$ above, the molecular structure of bis-chalcone **1b** shows the presence of intramolecular hydrogen bond in between phenolic hydrogen and the oxygen atom at C=O .

3.3. Photophysical Studies on Bis-Chalcone Compounds. The photophysical data of bis-chalcones **1a–2b** are shown in Table 2, and the absorption spectra of bis-chalcones **1a** and **2a** in DCM are shown in Figure 4.

Two absorption bands located at 268–278 nm and 333–383 nm were observed in bis-chalcones **1a–2b**; however, for bis-chalcones **2a–2b**, an additional absorption band was observed at about 421 nm, which corresponded to

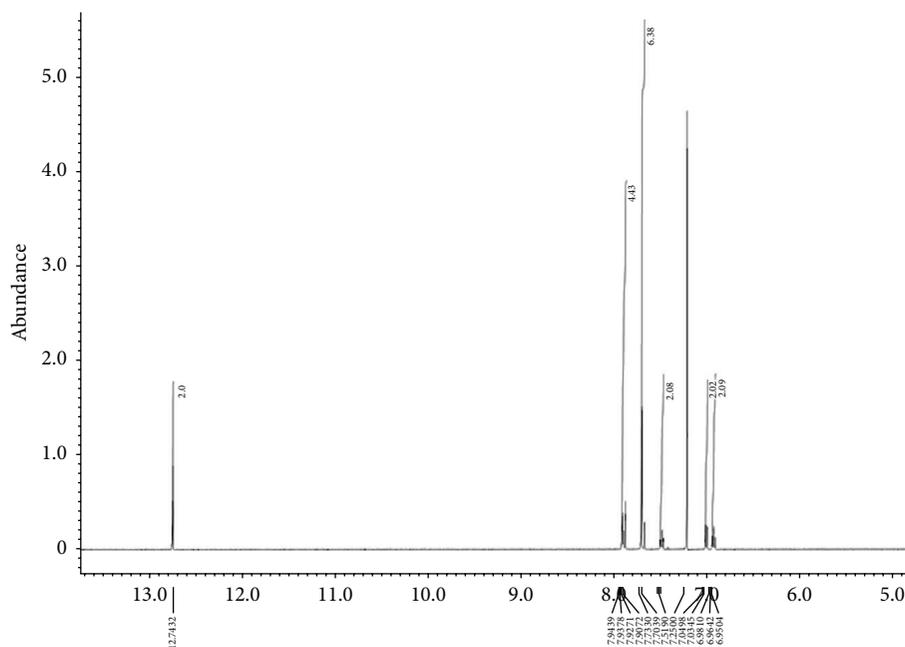


FIGURE 2: ^1H NMR spectrum of compound **1b**.

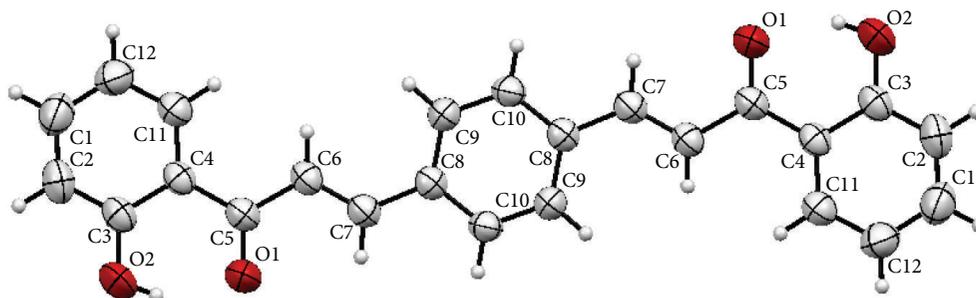


FIGURE 3: Molecular structure of bis-chalcone **1b** with the thermal ellipsoids plotted at 50% probability.

intramolecular charge transfer (ICT) from an ethylenic π electron to a carbonyl π electron [31, 32]. The absorption band at the range of 268 to 278 nm corresponded to the $\pi \rightarrow \pi^*$ transition in the aromatic ring. The absorption of this transition normally appears at around 200–250 nm; however, due to the conjugation effect of the carbonyl group and the carbon-carbon double bond system with the aromatic ring, the $\pi \rightarrow \pi^*$ transition was shifted to a longer wavelength. The presence of *ortho*-hydroxyl group at the wing phenyl rings has shifted the $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions in compounds **1b** and **2b** to a lower energy region (a longer wavelength). This is because the electron donating nature of the hydroxyl group has weakened the bond order of C=C and C=O groups. Surprisingly, the $n \rightarrow \pi^*$ transition ($\epsilon = \text{ca. } 22,000\text{--}26,000 \text{ mol}^{-1} \text{ cm}^{-1} \text{ dm}^3$) is much stronger than the $\pi \rightarrow \pi^*$ transition ($\text{ca. } 9,700\text{--}15,000 \text{ mol}^{-1} \text{ cm}^{-1} \text{ dm}^3$) in all the reported bis-chalcone compounds. This phenomenon can be explained by the presence of extended conjugation C=C-C=O chromophoric system in the bis-chalcone structure, which resulted in greater delocalization of π electrons along

the molecule [31]. Similar phenomena of absorption spectra were also found in the papers reported by Si and his coworkers in 2011 [13] and Tay et al. [33]. In the bis-chalcones reported by Si et al., they claimed that the increase of the absorption band 259 nm and the decrease of the band at 341 nm resulted from the $[2\pi + 2\pi]$ cycloaddition of the double bond in the chalcone unit to give a cyclobutane ring [13].

The emission spectra of bis-chalcones **1a-1b** and **2a-2b** are shown in Figure 5. The emissions λ_{max} of bis-chalcones **2a** and **2b** were shifted to a lower energy after the addition of the OMe group on the central phenyl ring. In general, bis-chalcone compounds **1a**, **1b**, and **2b** exhibited weak emission as seen in the low signal versus noise emission spectra. Due to this reason, we could not obtain the quantum yields and lifetimes of these compounds even in less polar solvents, that is, DCM. The quenching process in chalcones is normally due to intramolecular torsional motions as well as the *cis-trans* isomerization of the α,β -unsaturated ketone moiety [15, 16]. However, for compounds **1b** and **2b**, quenching could also

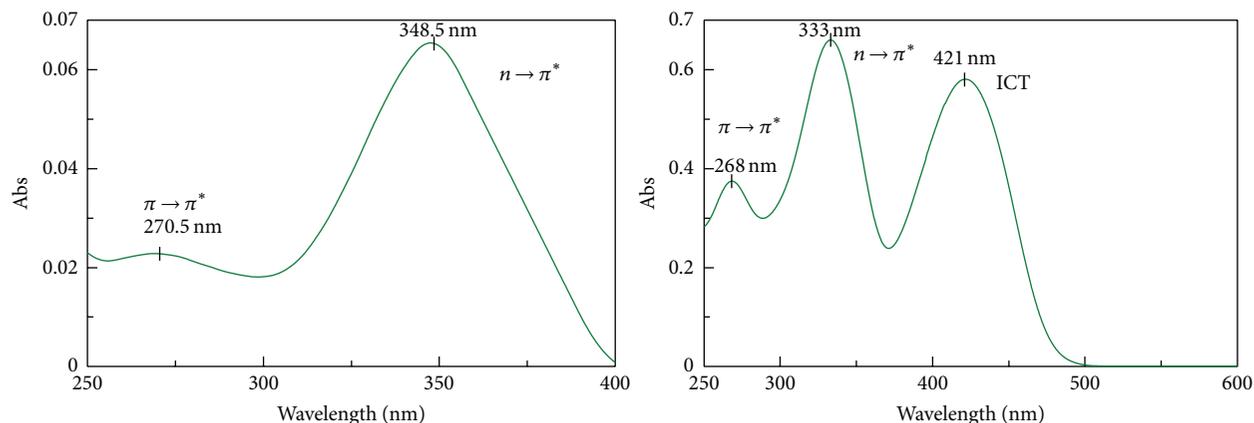


FIGURE 4: Absorption spectrum of bis-chalcones **1a** (top) and **2a** (bottom) in DCM.

TABLE 1: Summary of crystal data and structure refinement parameters of bis-chalcone **1b**.

Compound	1b
Empirical formula	C ₂₄ H ₁₈ O ₄
Formula weight	370.38
Temperature (K)	296(2)
Crystal system	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>n</i>
<i>a</i> (Å)	4.9841(10)
<i>b</i> (Å)	27.0759(7)
<i>c</i> (Å)	6.9770(2)
α (°)	90.00
β (°)	101.37(2)
γ (°)	90.00
Volume (Å ³)	923.05(4)
<i>Z</i>	2
Density (calculated) (mg/m ³)	1.333
Absorption coefficient (mm ⁻¹)	0.090
Θ range for data collection (°)	3.07 to 25.87
Reflections collected	2111
Independent reflections	1546
Data/restraints/parameters	2111/0/129
Final <i>R</i> indices [<i>I</i> > 2 σ (<i>I</i>)]	<i>R</i> 1 = 0.0394 <i>wR</i> 2 = 0.1164
<i>R</i> indices (all data)	<i>R</i> 1 = 0.0565 <i>wR</i> 2 = 0.1326

be due to the intramolecular hydrogen bond, which can be observed from the molecular structure of **1b** in Figure 3.

Interestingly, bis-chalcone **2a** was found to be highly emissive at 505 nm with a quantum yield of 0.57 as well as lifetimes of 2.44 and 4.04 ns in DCM solution. Apart from DCM, this compound also shows intense emission in polar solvents, that is, acetonitrile. The quantum yield in acetonitrile was 0.40 using Coumarin 307 as the reference [26, 27]. The nanosecond lifetime indicates that emission originates from

the singlet excited state. Comparing bis-chalcone **1a**, the emission efficiency of bis-chalcone **2a** improves when there is an OMe group on the central phenyl ring. Chalcone and related compounds are generally known as poor emitters. The introduction of an OMe group on the central phenyl ring in compound **2a** is considered another method to improve the emission efficiency of chalcone compounds apart from the method reported by D'Aléo and his coworkers (2012) [17].

3.4. Solvatochromism Study of Bis-Chalcone 2a. The solvatochromism study of bis-chalcone **2a** was performed by dissolving the compound in the following solvents: benzene, tetrahydrofuran (THF), DCM, and acetonitrile (MeCN). The emission λ_{\max} changed from 499 nm (blue) in benzene to 523 nm (yellowish green) in MeCN (Figure 6), which indicates that the excited molecule of bis-chalcone **2a** has intermolecular interaction with the polar solvent molecules. It is believed that intramolecular charge transfer occurred from the OMe group on the central phenyl ring towards both sides of the phenyl rings.

4. Conclusions

A method to improve the emission efficiency of bis-chalcone has been found. By adding a methoxy group at the central phenyl ring of bis-chalcone, the quantum yield of bis-chalcone **2a** was improved to 0.57 in DCM solution, and it was still emissive ($\Phi_F = 0.40$) in acetonitrile using Coumarin 307 as the reference. A solvatochromism study of bis-chalcone **2a** was performed and the emission λ_{\max} increased from 499 nm in benzene to 523 nm in MeCN.

Competing Interests

The authors declare that they have no competing interests.

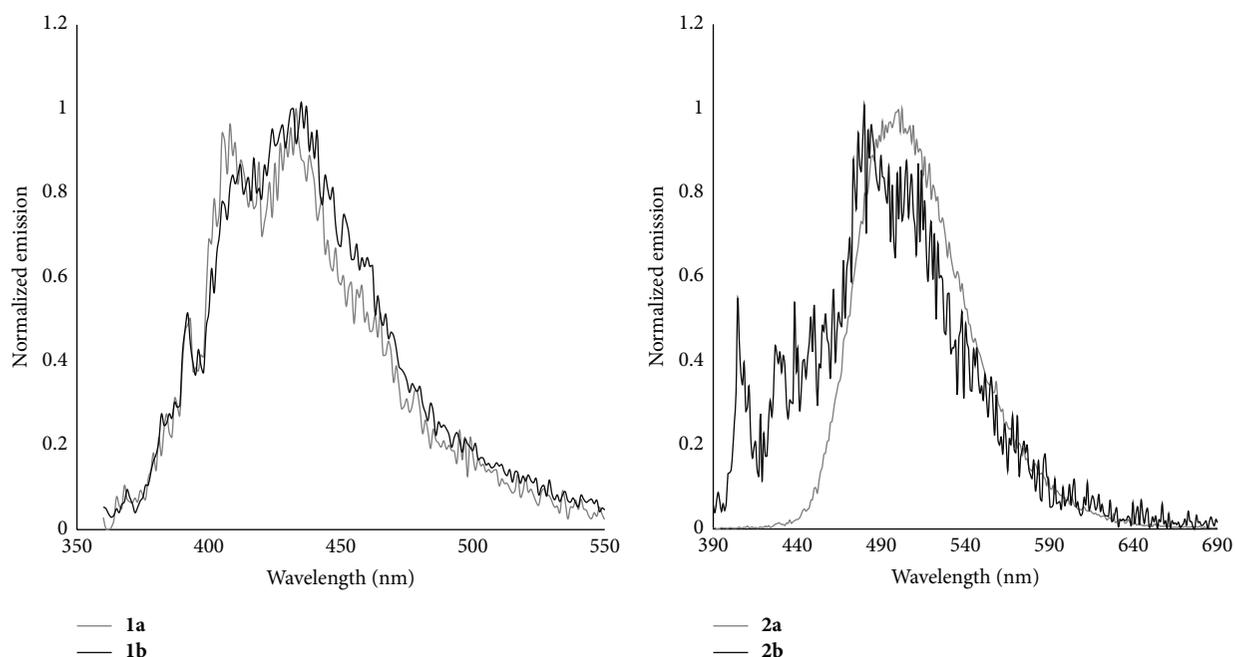
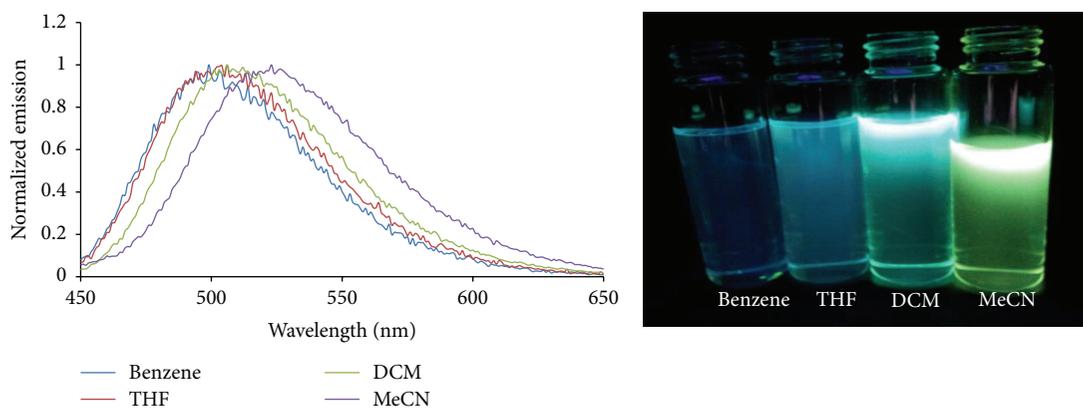
Acknowledgments

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TABLE 2: Photophysical data for compounds **1a** to **2b** in solution state at room temperature.

Compound	$\lambda_{\text{abs}}^{\text{a}}$ /nm ($\epsilon/10^4 \text{ M}^{-1} \text{ cm}^{-1}$)	$\lambda_{\text{em}}/\text{nm}$	Φ_{F}	$\tau_{\text{F}}/\text{ns}$	Stokes shift ^e / cm^{-1}
1a	270 (0.970)	~430	— ^b	— ^b	5500
	348 (2.228)				
1b	278 (0.979)	~432	— ^b	— ^b	3000
	383 (2.369)				
2a	268 (1.519)	505	0.57 ^c	2.44 (15%)	10200
	333 (2.662)		0.40 ^d	4.04 (85%)	
	421 (2.384)				
2b	275 (1.385)	— ^b	— ^b	— ^b	—
	350 (2.164)				
	445 (2.576)				

^aAll the UV data was recorded in DCM solution. ^bNot able to be determined due to very poor emission. ^cFluorescence quantum yield which was measured in DCM solution using an integrating sphere. ^dFluorescence quantum yield which was measured in acetonitrile solution using Coumarin 307 as a reference [26, 27]. ^eStokes shift was calculated based on $\lambda_{\text{max abs}} - \lambda_{\text{max em}}$.

FIGURE 5: Emission spectra of compounds **1a-1b** (top) and **2a-2b** (bottom) in DMSO.FIGURE 6: Solvatochromism study of bis-chalcone **2a**.

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