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Research Article

Synthesis of *E*-Chalcone Derivatives Bearing Different Substituent Groups and Evaluation of Substituent Effects on Their Spectroscopic Properties

Görkem Özcan¹Bleda Can Sadıkoğulları¹ Emre Can Uysal¹ Ayse Daut Özdemir^{1*} ¹Department of Chemistry, Faculty of Science and Letters, Istanbul Technical University, Istanbul, Türkiye**Abstract**

Chalcones are simple yet significant structures containing various examples of enolate chemistry. Nowadays, these compounds can be synthesized in various ways, such as through methods like Claisen-Schmidt condensation. This method is commonly used due to its ease of application, high efficiency, and method diversity in synthesis. Due to their unsaturated structures, chalcone derivatives can be used in different addition reactions. They can be modified with various functional groups to achieve desired properties using simple starting reactants. These advantages make chalcone derivatives suitable for a wide range of applications. Beyond their wide range of medicinal applications – driven by notable pharmacological properties such as anticancer, anti-HIV, and antibacterial activities – chalcone derivatives have also attracted considerable attention in materials chemistry as both optical sensors and heterocyclic intermediates for chemosensor applications. In this study, various chalcone derivatives bearing suitable functional groups were synthesized, and their structures were characterized while their optical properties were systematically investigated. The structural analyses performed following the syntheses not only confirmed the structures of the synthesized compounds but also examined and substantiated the effects of the functional groups present on the corresponding molecular scaffold in terms of NMR and optical properties.

Keywords: *E*-Chalcone, Claisen–Schmidt Condensation, Functional Group Effect.

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1. Introduction

The synthesis of novel compounds for industrial and scientific applications represents one of the continuously evolving areas of organic chemistry. In this context, the synthesis of novel chalcone derivatives has attracted considerable attention owing to their diverse physicochemical and biological properties. Chalcones are the molecules in which two aromatic rings bonded with α,β -unsaturated ketone structure [1]. They are polyphenolic compounds with colors ranging from yellow to orange, abundantly found in plants, and serving as the biogenetic precursors of flavonoids and isoflavonoids [2]. Stereochemically, they can exist as *E* and *Z* isomers. The *Z*- configuration is generally unstable because of strong steric hindrance between the carbonyl group and the aromatic ring, whereas the *E*- isomer is thermodynamically more stable (Figure 1a) [2]. Considering the *s*-cis and *s*-trans conformers of the molecule, the most stable chalcone isomer identified experimentally is the *trans*-(*s*-cis)-chalcone. This enhanced stability is attributed to the complete planar geometry of the molecule [3]. Chalcone derivatives are planted with secondary metabolites with diverse biological activities which play an important role in organic chemistry and specially in natural product chemistry. They have been used in different application areas such as the pharmaceutical industry due to their choleretic, anti-ulcer, antimicrobial, anti-HIV, anticancer, and central nervous system (CNS) activities [1-8]. Metochalcone (a choleretic drug) and sofalcone (an anti-ulcer and mucoprotective agent) are well-known examples of chalcone derivatives used in clinical applications [8-10] (Figure 1b). Besides these applications they can be served as chemical probes, fluorescent dyes, and sensors which chalcone derivatives functionalized with electron-withdrawing and/or electron-donating groups on their aromatic rings exhibit fluorescent properties [11-15] (Figure 1c).

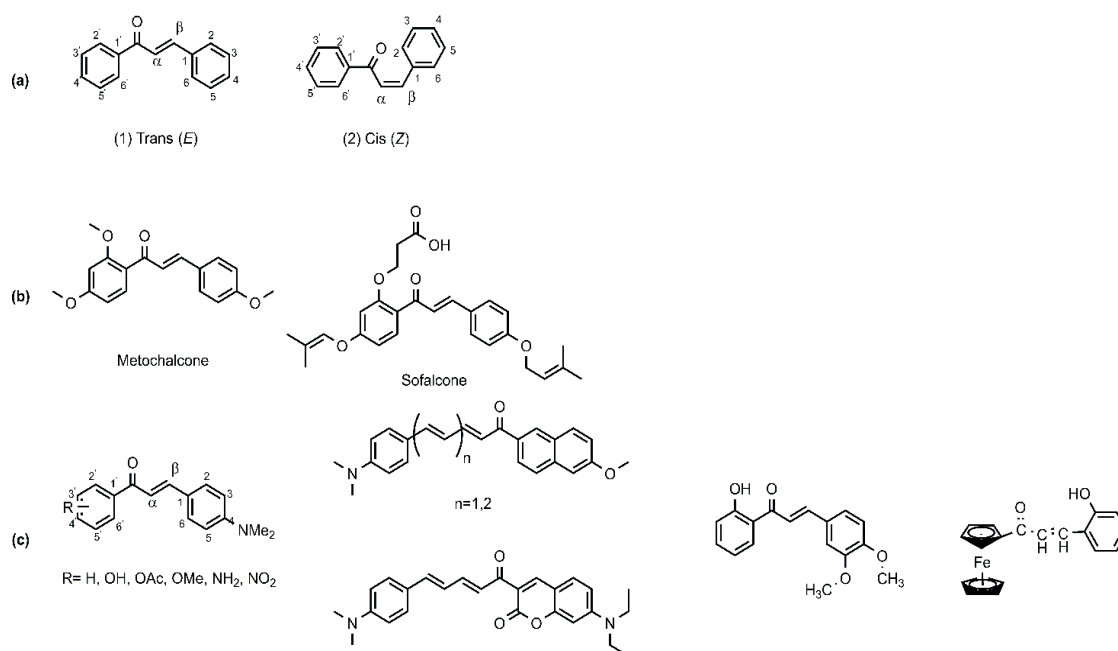


Figure 1. *E*- and *Z*- isomers of chalcones (a), Chemical structures of approved and clinically evaluated chalcones (b), Literature examples of chalcone derivatives (c) [11-15].

Additionally, chalcone derivatives used in chemical probes, fluorescence dyes, chemosensors [3,16,17] as well as intermediates in the production of sweeteners and as components of perfumes [18].

Because of their relatively simple synthesis and ease of structural modification, chalcones are widely used as versatile intermediates in the synthesis of more complex molecules [19]. For the synthesis of chalcone molecules, cross-aldol condensation which is known as Claisen-Schmidt condensation, is commonly used (Figure 2).

Also, Figure 3 presents examples of the Wittig reaction and coupling reactions, which are among the alternative methods currently employed for the synthesis of chalcones. Within the scope of this study, a series of *E*-chalcone derivatives were synthesized, and their structural characterization is presented in detail. The structural characterization of the synthesized compounds was discussed with particular emphasis on the effects of the substituent groups. Moreover, the optical properties of the synthesized dyes were investigated through absorption and emission studies. The chalcone derivatives synthesized in this study are listed in Table 1.

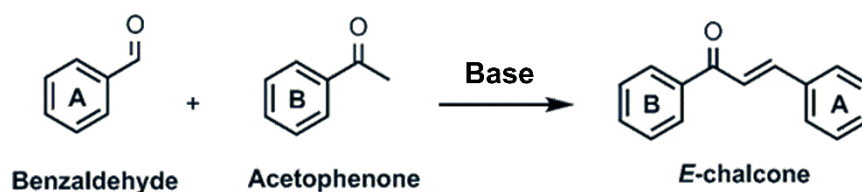


Figure 2. Synthesis of *E*-chalcone

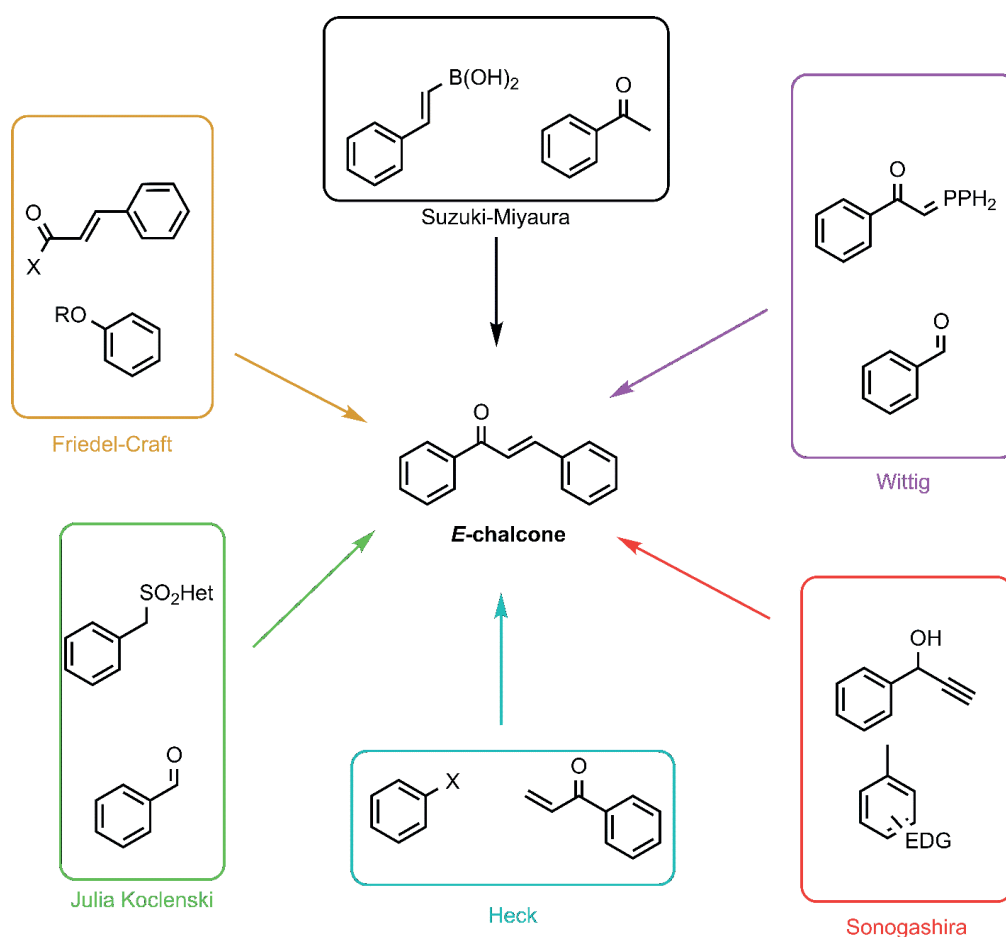


Figure 3. Various synthetic routes for the synthesis of *E*-chalcone [3]

2. Materials and Methods

All chemicals used for the syntheses (benzaldehyde, 2-hydroxybenzaldehyde, pyrene-1-carbaldehyde, acetophenone, 4-hydroxyacetophenone, sodium hydroxide, and hydrochloric acid) were purchased from Sigma-Merck and used without further purification. All solvents employed throughout this study (ethyl acetate, hexane, ethanol, dimethyl sulfoxide, and acetonitrile) were distilled when supplied in technical grade; otherwise, they were used as received.

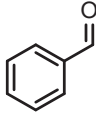
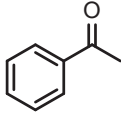
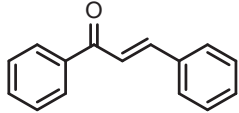
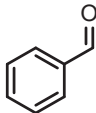
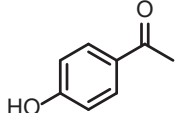
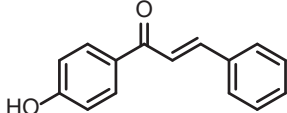
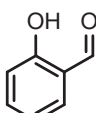
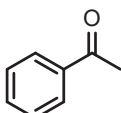
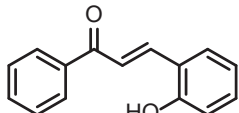
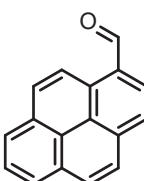
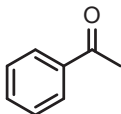
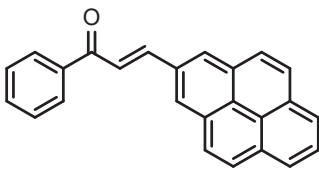
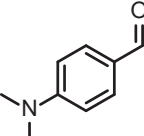
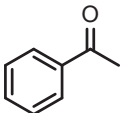
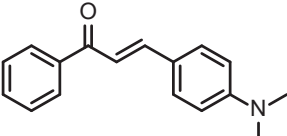
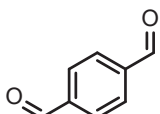
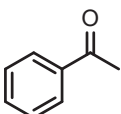
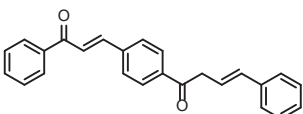


Structural characterization was performed using a Varian nuclear magnetic resonance (NMR) spectrometer, and all spectra were processed using MestReNova software. The chemical shifts of protons and carbons (500 MHz for ^1H NMR and 125 MHz for ^{13}C NMR) are reported in parts per million (ppm) relative to tetramethylsilane (TMS) as the internal standard. UV-Vis absorption spectra were recorded over the 190–1100 nm range using a T80+ UV-Vis spectrophotometer with 10 mm path-length quartz cuvettes. Fluorescence measurements were carried out at room temperature using an Agilent Cary Eclipse fluorescence spectrophotometer with a 10 mm path-length quartz cuvette.

2.1 Experimental Setup and Procedures

The α,β -unsaturated ketone compounds were synthesized via the Claisen-Schmidt condensation reaction. Equimolar amounts of a substituted acetophenone and an aromatic aldehyde derivative (1.25 mmol of the aromatic aldehyde derivative) were dissolved in ethanol and added dropwise at room temperature to a 10% aqueous sodium hydroxide solution (EtOH/H₂O, 4:5 v/v). The reaction mixture was stirred for 16 h, and the reaction progress was monitored by TLC. Upon completion, the mixture was neutralized with 4 N HCl in an ice bath. The reaction mixture was then stored in a refrigerator (+4 °C) overnight, allowing the product to precipitate. The precipitated solid was collected by filtration, washed with cold ethanol, and dried. When necessary, the crude products were further purified by recrystallization from ethanol [20-23].

**Table 1.** Synthesized *E*-chalcone derivatives

Aromatic Aldehyde Derivative	Substituted Acetophenone Compound	Synthesized Chalcone Derivative	Compound Name (Yield)
			Chalcone-3 (90%)
Benzaldehyde	Acetophenone	<i>E</i> -chalcone	
			Chalcone-5 (95%)
Benzaldehyde	4-hydroxyacetophenone	4'-hydroxy- <i>E</i> -chalcone	
			Chalcone-6 (97%)
2-hydroxybenzaldehyde	Acetophenone	2-hydroxy- <i>E</i> -chalcone	
			Chalcone-7 (84%)
Pyrene-1-carboxaldehyde	Acetophenone	(<i>E</i>)-1-phenyl-3-(pyren-2-yl)prop-2-en-1-one	
			Chalcone-9 (92%)
4-dimethylaminobenzaldehyde	Acetophenone	4-dimethylamino- <i>E</i> -chalcone	
			Chalcone-12 (86%)
terephthalaldehyde	Acetophenone	bis- <i>E</i> -chalcone	

Chalcone-3 (^1H NMR, 500 MHz, CDCl_3): 8.04 (d, 2H); δ = 7.83 (d, 1H); δ = 7.67 (d, 2H); δ = 7.60 (t, 1H); δ = 7.53 (m, 3H); δ = 7.44 (m, 3H). **Chalcone-3** (^{13}C NMR, 126 MHz, CDCl_3): 190.60, 144.87, 138.23, 134.90, 132.80, 130.56, 128.98, 128.64, 128.52, 128.46, 122.12.

Chalcone-5 (^1H NMR, 500 MHz, CDCl_3): 8.02 (d, 2H); δ = 7.83 (d, 1H); δ = 7.67 (d, 2H); δ = 7.60 (t, 1H); δ = 7.53 (m, 3H); δ = 7.44 (m, 3H). **Chalcone-5** (^{13}C NMR, 126 MHz, CDCl_3): 188.95, 160.10, 144.27, 135.02, 131.18, 131.17, 130.42, 128.95, 128.40, 121.84, 115.49.

Chalcone-6 (^1H NMR, 500 MHz, CDCl_3): 10.29 (s, 1H), 8.09 (d, 2H), 8.05 (d, 1H), 7.89 – 7.81 (m, 2H), 7.64 (t, 1H), 7.56 (t, 2H), 7.27 (t, 1H), 6.94 (d, 1H), 6.87 (t, 1H). **Chalcone-6** (^{13}C NMR, 126 MHz, CDCl_3): 189.98, 157.74, 140.03, 133.35, 132.53, 129.23, 129.11, 128.80, 121.83, 121.40, 119.87, 116.70.





Chalcone-7 (^1H NMR, 500 MHz, CDCl_3): 9.02 (d, 1H), 8.58 (d, 1H), 8.45 (d, 1H), 8.26 – 8.04 (m, 9H), 7.85 (d, 1H), 7.68 – 7.61 (m, 1H), 7.58 (d, 2H). **Chalcone-7** (^{13}C NMR, 126 MHz, CDCl_3): 190.23, 141.44, 138.42, 132.92, 132.86, 131.27, 130.68, 130.32, 128.72, 128.69, 128.65, 128.61, 127.31, 126.30, 126.06, 125.92, 125.01, 124.92, 124.55, 124.14, 123.83, 122.53.

Chalcone-9 (^1H NMR, 500 MHz, CDCl_3): 8.07 – 7.97 (m, 2H), 7.81 (d, 1H), 7.62 – 7.46 (m, 5H), 7.35 (d, 1H), 6.70 (d, 2H), 3.05 (s, 6H). **Chalcone-9** (^{13}C NMR, 126 MHz, CDCl_3): 190.71, 152.06, 145.89, 139.08, 132.16, 130.44, 128.47, 128.32, 116.89, 111.83, 40.14.

Chalcone-12 (^1H NMR, 500 MHz, CDCl_3): 8.08 – 8.00 (m, 4H), 7.82 (d, 2H), 7.70 (s, 4H), 7.64 – 7.51 (m, 8H). **Chalcone-12** (^{13}C NMR, 126 MHz, CDCl_3): 190.28, 143.58, 138.04, 136.88, 133.00, 128.99, 128.72, 128.55, 123.03

2.1.1. UV-Vis and Fluorescence Spectroscopic Analysis

The stock solutions of Chalcone-3 (1 mM), Chalcone-5 (1 mM), and Chalcone-7 (5 mM) were prepared in acetonitrile, whereas the stock solution of Chalcone-6 (1 mM) was prepared in dimethyl sulfoxide (DMSO). UV-Vis absorption measurements were performed over the concentration range of 0–80 μM , and the corresponding calibration curves were constructed. Also, owing to the presence of the pyrene moiety, the fluorescence emission spectrum of Chalcone-7 was recorded over the concentration range of 0–20 μM , and the corresponding fluorescence calibration curve was established.

3. Results

The functional groups present in a molecular structure, and their positions are among the key factors influencing the properties of the final product. In this study, we confirmed the structures of the synthesised *E*-chalcone derivatives through structural characterisation and investigated the effects of the functional groups on their properties.

The structural characterization of all synthesized compounds was carried out using ^1H NMR and ^{13}C NMR spectroscopy. Examination of the ^1H NMR spectra presented in Figure 4 revealed a coupling constant of approximately 15 Hz for the $\text{O}=\text{C}-\text{CH}=\text{CH}_2$ moiety. This large coupling constant clearly indicates the trans (*E*-) configuration of the double bond. Furthermore, the proton attached to the $\text{O}=\text{C}-\text{CH}$ fragment resonates at 7 ppm or higher, which can be attributed to the reduced shielding of this proton resulting from the extensive conjugation within the *E*-chalcone framework. Analysis of the chemical shifts corresponding to the $\text{O}=\text{C}(\text{R})-\text{CH}=\text{CH}_2-\text{R}$ moiety showed that, for each derivative, the proton attached to the β -carbon is less shielded than the proton attached to the α -carbon. This observation can be explained by the partial positive charge developed at the β -carbon due to the α,β -unsaturated carbonyl system. Based on the ^1H NMR analyses, the effects of different substituents can be evaluated by taking Chalcone-3 as the reference compound. Accordingly, Table 2 was prepared to summarize the chemical shifts of the α - and β -protons.

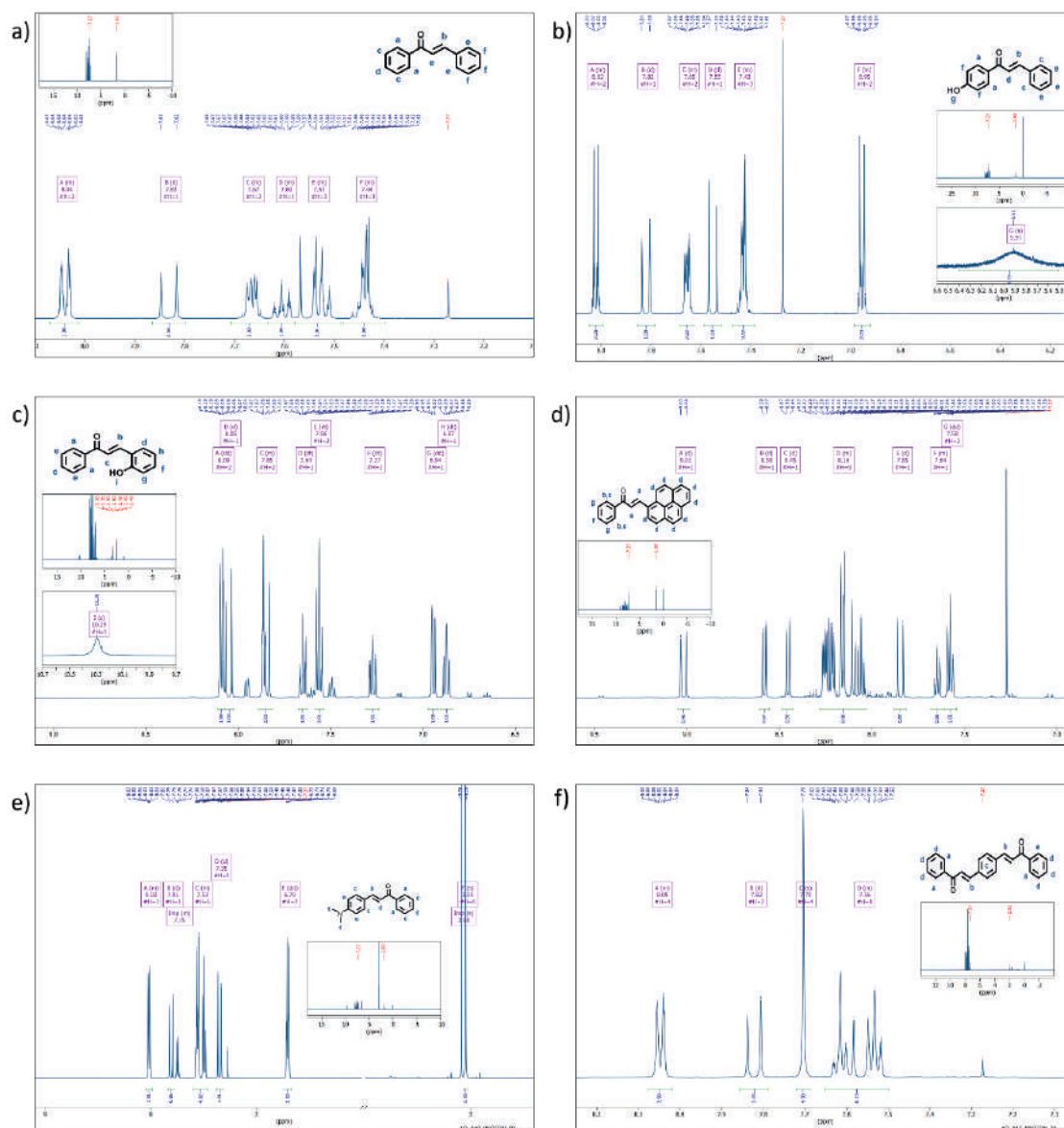
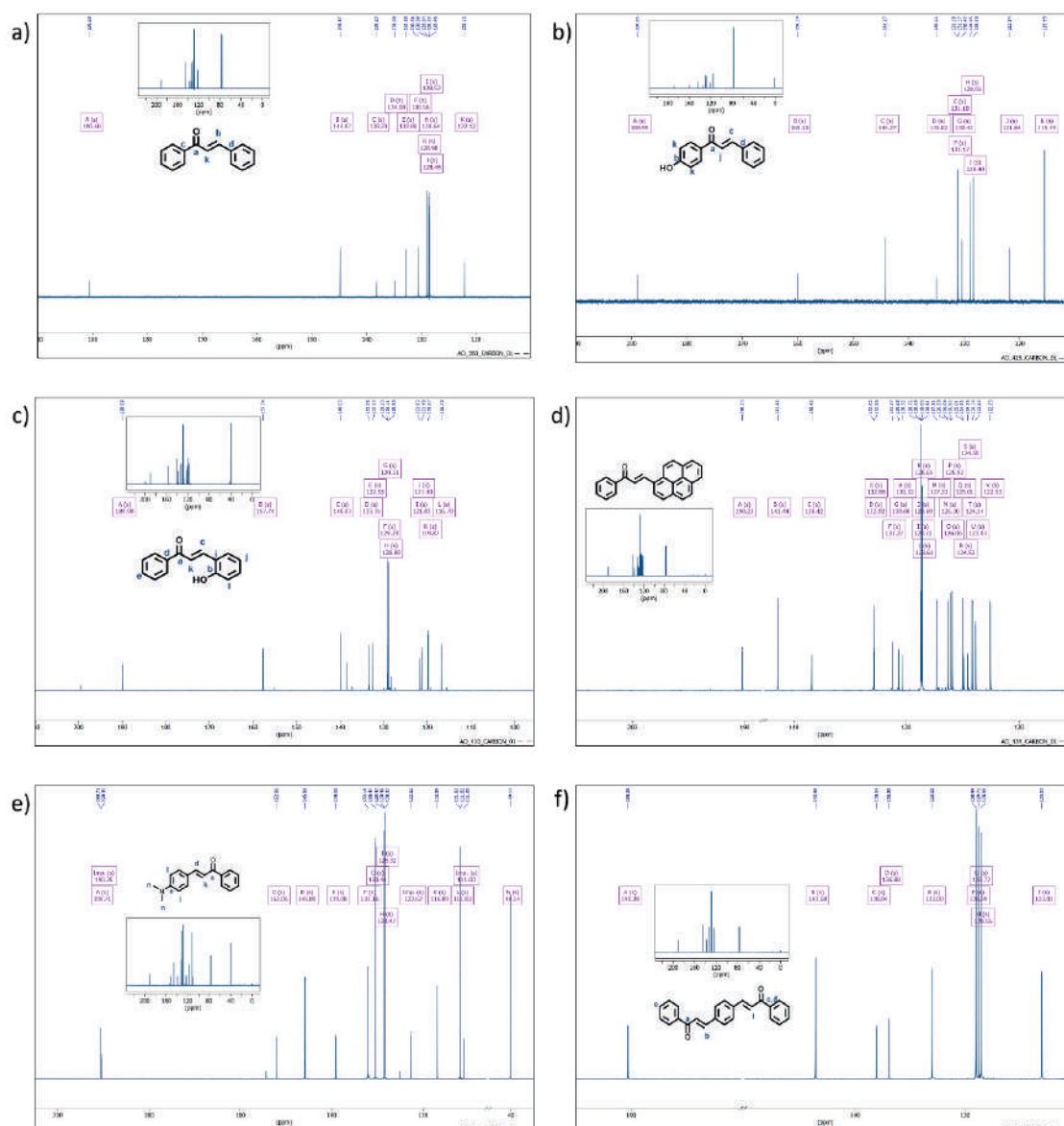


Figure 4. ¹H NMR spectra of Chalcone-3 (a), Chalcone-5 (b), Chalcone-6 (c), Chalcone-7 (d), Chalcone-9 (e) and Chalcone-12 (f)

**Table 2.** Chemical shifts of the α - and β -protons as a functional groups

Synthesized Derivative	Schematic representation and NMR chemical shifts (in ppm) of the α - and β -protons	Chemical Shift Analysis
Chalcone-3		Reference Compound
Chalcone-5		Although the hydroxyl function is an electron-donating group, no significant effect was observed due to the electron-withdrawing nature of the carbonyl group and its distance from the α , β -unsaturated bond.
Chalcone-6		While the hydroxyl function is an electron-donating group, the opposite effect was observed due to intramolecular hydrogen-bond formation involving the β -proton.
Chalcone-7		The extensive π -electron delocalization within the pyrene moiety increases <i>E</i> -chalcones' electron density, leading to reduced shielding of the β -position proton.
Chalcone-9		The electron-donating nature of the $N(CH_3)_2$ group leads to increased shielding, particularly at the α -position compared to the β -position, due to electron delocalization throughout the α , β -unsaturated system.
Chalcone-12		Chalcone-12 does not contain any additional functional groups.

Similarly, examination of the ^{13}C NMR spectra (Figure 5) revealed the absence of a signal at approximately 200 ppm, indicating that no aldehyde functionality remained in the final products. Furthermore, comparison with the starting ketones showed a noticeable shift in the resonance of the carbonyl carbon. This shift can be attributed to the fact that the synthesized *E*-chalcone derivatives are phenylstyryl ketone analogues, possessing greater electron density and a higher degree of π -electron delocalization than the corresponding starting ketones.



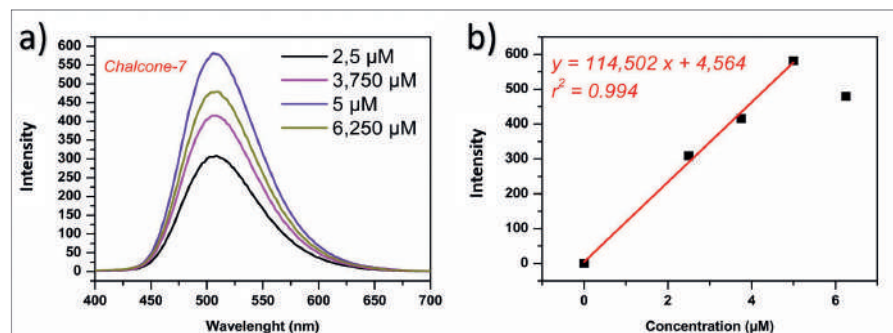


Figure 6. Fluorescence emission spectrum of Chalcone-7 (a), and the corresponding calibration curve (b)

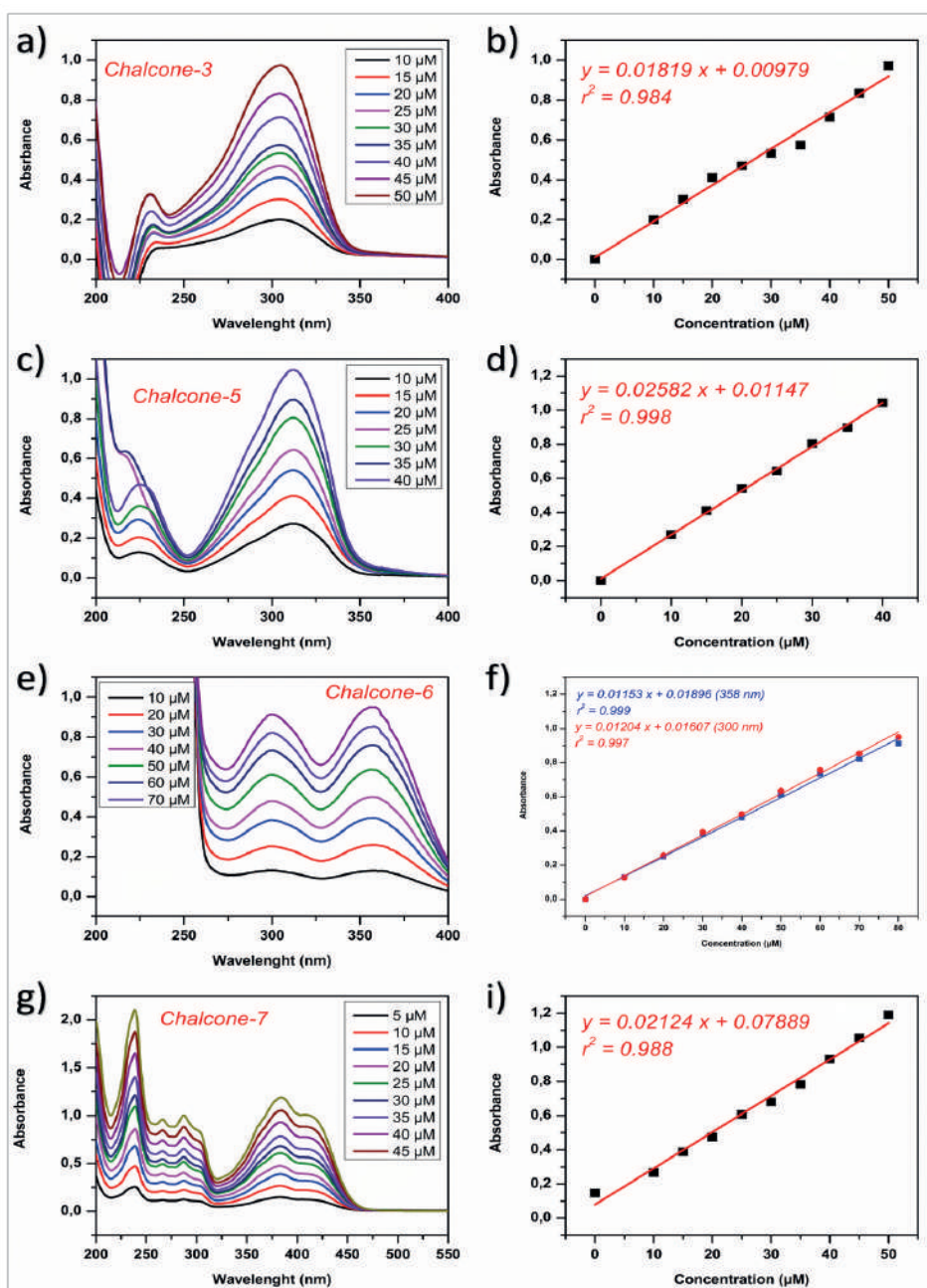


Figure 7. UV-Vis absorption spectrum of Chalcone-3 (a) and the corresponding calibration curve (b), UV-Vis absorption spectrum of Chalcone-5 (c), and the corresponding calibration curve (d), UV-Vis absorption spectrum of Chalcone-6 (e), and the corresponding calibration curve (f), UV-Vis absorption spectrum of Chalcone-7 (g), and the corresponding calibration curve (h).



4. Conclusions

Herein a series of substituted *E*-chalcone derivatives were successfully synthesized via Claisen-Schmidt condensation with high yields (up to 97%) under basic conditions in line with literature [20-23], and the resulting structures were confirmed by spectroscopic characterization. In this study, The Claisen-Schmidt reaction was performed in the synthesis of targeted chalcones, in which the reaction pathway proceeds through an aldol addition followed by dehydration step, whereby the base-catalyzed enolate of the methyl ketone attacks the carbonyl carbon of the benzaldehyde, and subsequent elimination of water furnishes the conjugated α,β -unsaturated ketone framework with the thermodynamically favored *E*- geometry [9]. Given that chalcones possess remarkable versatility as synthetic intermediates – specifically because of their α,β -unsaturated ketone core serving as an electrophilic Michael acceptor – that can be utilized in the construction of heterocyclic scaffolds, [24, 25] a comprehensive identification of substituent effects will provide valuable insights into their electronic tuning and chemical behavior. In the study, these effects were discussed over synthesized six different chalcones demonstrating that the optical and electronic properties of *E*-chalcones can be precisely fine-tuned by strategic functional group positioning and electronic modification. These structurally characterized derivatives provide a robust foundational library for the ongoing development of further heterocyclic scaffolds that can be utilized in various fields, such as fluorescent probes and chemosensors.

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