JOURNAL OF APPLIED SCIENCE AND ENGINEERING MATERIALS

FARKLI SÜBSTİTÜENT GRUPLARI TAŞIYAN *E*-KALKON TÜREVLERİNİN SENTEZİ VE SÜBSTİTÜENTLERİN SPEKTROSKOPİK ÖZELLİKLERİ ÜZERİNDEKİ ETKİLERİNİN DEĞERLENDİRİLMESİ

SYNTHESIS OF *E*-CHALCONE DERIVATIVES BEARING DIFFERENT SUBSTITUENT GROUPS AND EVALUATION OF SUBSTITUENT EFFECTS ON THEIR SPECTROSCOPIC PROPERTIES

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Özet

Kalkonlar, enolat kimyasının çeşitli örneklerinin bulunduğu, basit ve önemli yapılar arasındadır. Bu bileşikler yaygın olarak Claisen-Schmidt kondenzasyonu, bunun yanı sıra Wittig reaksiyonu ve de çeşitli kenetlenme reaksiyonları gibi yöntemlerle sentezlenebilmektedir. Claisen-Schmidt kondenzasyonu, kolaylıkla uygulanabilmesi, yüksek verim sağlayabilmesi ve sentezde metot çeşitliliği sunması nedeniyle sıkça kullanılmaktadır. Kalkon bileşikleri, 1,3-doymamış sistemleri sayesinde çeşitli katılma reaksiyonlarını gerçekleştirebilirler ve basit çıkış kimyasallarıyla istenen özelliklere sahip çeşitli fonksiyonel gruplarla modifiye edilebilirler. Bu avantajları nedeniyle Kalkonlar geniş bir kullanım alanına sahiptir. Sensör çalışmaları, kimyasal problar ve floresans boyaların yanı sıra anti-kanser, anti-HIV ve antibakteriyel gibi çeşitli farmakolojik özellikleri nedeniyle tıbbi açıdan da araştırılan bileşiklerdir.

Bu çalışmada, çıkış bileşiği olarak kullanılmak üzere, uygun fonksiyonel gruba sahip farklı Kalkon bileşikleri sentezlenerek yapı karakterizasyonları gerçekleştirilmiş ve optik özellikleri incelenmiştir. Sentezlerin ardından gerçekleştirilen yapı analizleri çalışmaları, sentezlenen yapıları sadece kanıtlanmakla kalmamış aynı zamanda, ilgili iskelet üzerinde bulunan fonksiyonel grupların etkilerini de NMR ve optik özellikler açısından irdelemiş ve desteklemiştir.

Anahtar Kelimeler: *E*-Kalkon, Claisen-Schmidt kondenzasyonu, Fonksiyonel Grup Etkisi.

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 unstaurated structures, Chalcone derivatives can be used in different addition reactions. Also, 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. In addition to, sensor applications, chemical probes, and fluorescence dyes, chalcone derivates attract considerable attention in medical applications because of their diverse pharmacological activities such as anticancer, anti-HIV, and antibacterial activities.

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.

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. Chaclones are the molecules which two aromatic rings bonded with *α,β*- unsaturated ketone structure (Patil et al., 2009).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 (Gomes et al., 2017). 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) (Gomes et al., 2017). 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 completely planar geometry of the molecule (Rammohan et al., 2020). Chalcone derivatives are plant secondary metabolites with diverse biological activities which play an important role in organic chemistry and specially in natural product chemistry. Also they been used in different application areas such as pharmaceutical industry due to their choleretic, anti-ulcer, antimicrobial, anti-HIV, anticancer, and central nervous system (CNS) activities. (Patil et al., 2009; Gomes et al., 2017; Mahapatra et al., 2015; Rammohan et al., 2020; Goyal et al., 2021; Królicka et al., 2022; Liu et al., 2022; Sahu et al., 2022) Metochalcone (a choleretic drug) and sofalcone (an anti-ulcer and mucoprotective agent) are well known examples of chalcone dervatives uesd in clinical applications. (Sahu et al., 2022; Zhuang et al. 2017; Higuchi et al. 2010) (Figure 1b). Addition to 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. (Mellado et al 2023; Ghorpade et al. 2025; Khan et al. 2024; Liu et al. 2025; Bhagya et al. 2025) (Figure 1c).

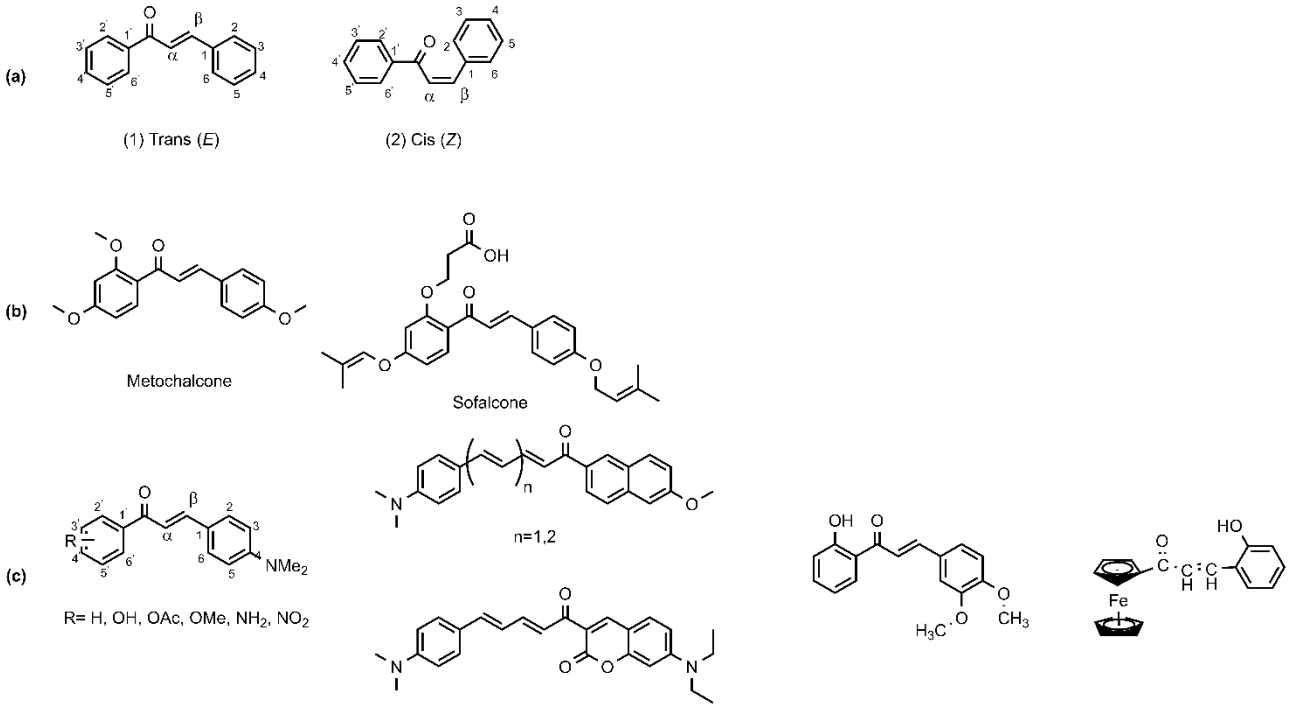


Figure 1. (a) *E*- and *Z*- isomers of chalcones. **(b)** Chemical structures of approved and clinically evaluated chalcones. **(c)** Literature examples of chalcone derivatives (Mellado et al 2023; Ghorpade et al. 2025; Khan et al. 2024; Liu et al. 2025; Bhagya et al. 2025).

Addition to these applications, chalcone derivatives used in chemical probes, fluorescence dyes, chemosensors (Rammohan et al. 2020; Rurack et al. 2000; Gupta et al. 2020) as well as as intermediates in the production of sweeteners and as components of perfumes (Manchanayakage. 2016).

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 (Farooq and Ngaini, 2019). For the synthesis of chalcone molecules, cross aldol condensation which is known as Claisen-Schmidt condensation, is commonly used (Figure 2).

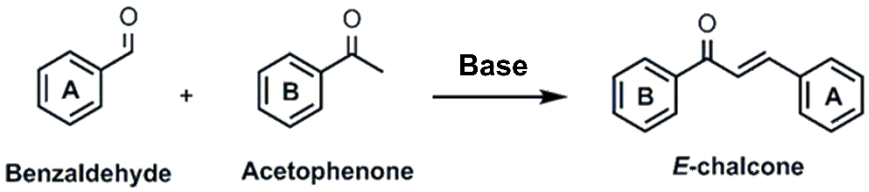


Figure 2. Synthesis of *E*-chalcone.

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.

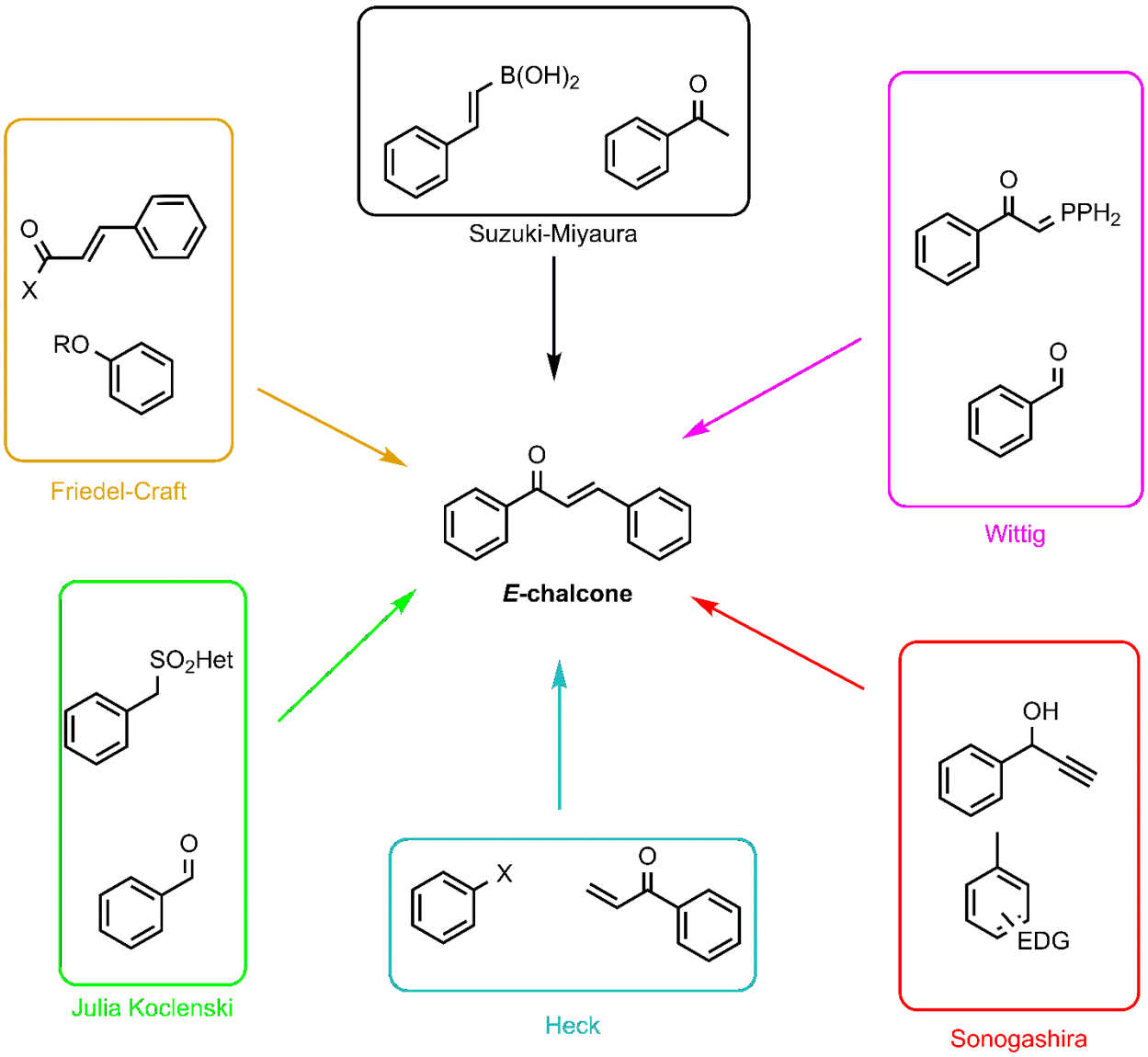


Figure 3. Various synthetic routes for the synthesis of *E*-chalcone. (Rammohan et al. 2020)

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. In addition, 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.

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,

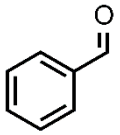
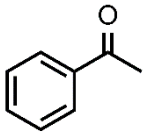
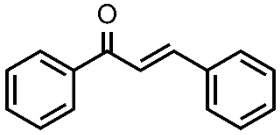
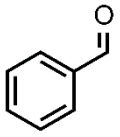
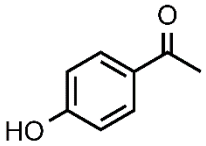
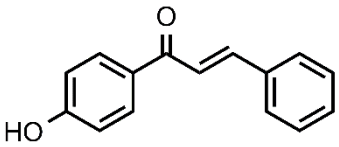
hexane, ethanol, dimethyl sulfoxide, and acetonitrile) were distilled when supplied in technical grade; otherwise, they were used as received.

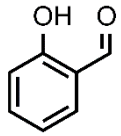
Structural characterization was performed using a Varian nuclear magnetic resonance (NMR) spectrometer, and all spectra were processed using MestReNova software. The chemical shifts of protons and carbons (500 MHz for ^{1}H NMR and 125 MHz for ^{13}C NMR) are reported in parts per million (ppm) relative 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. Synthesis of α,β-Unsaturated Ketone Compounds

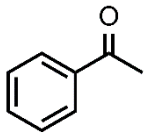
The *α,β*-unsaturated ketone compounds were synthesized *via* the Claisen–Schmidt condensation reaction. Equimolar amounts of a substituted acetophenone and an aromatic aldehyde derivative (1.25 mmol of the aromatic aldehyde derivative) were dissolved in ethanol and added dropwise at room temperature to a 10% aqueous sodium hydroxide solution ($EtOH/H_2O$, 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 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 (Kagatikar et al. 2023; Pereira et al. 2023; Silva et al., 2013; Nurkenov et al. 2019).

Table 1. Synthesized *E*-chalcone Derivatives.

Aromatic Aldehyde Derivative	Substituted Acetophenone Compound	Synthesized Chalcone Derivative
 **Benzaldehyde** **106.12 g/mol** **1.040 g/mL**	 **Acetophenone** **120.15 g/mol** **1.030 g/mL**	**Chalcone-3**  **(*E*)-Chalcone** **208.26 g/mol** **Yield: 90%**
 **Benzaldehyde** **106.12 g/mol** **1.040 g/mL**	 **4'-hydroxyacetophenone** **136.15 g/mol**	**Chalcone-5**  **4'-hydroxy-(*E*)-Chalcone** **224.26 g/mol** **Yield: 95%**

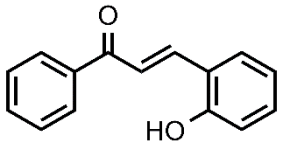


2'-hydroxybenzaldehyde
122.12 g/mol
1.170 g/mL

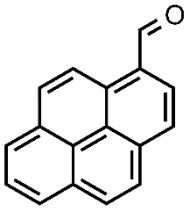


Acetophenone
120.15 g/mol
1.030 g/mL

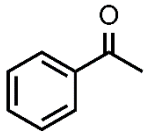
Chalcone-6



2'-hydroxy-(*E*)-Chalcone
224.26 g/mol
Yield: 97%

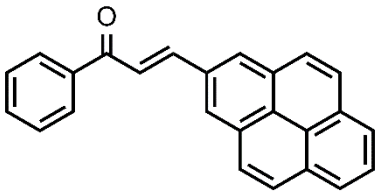


pyrene-1-carboxyaldehyde
230.27 g/mol

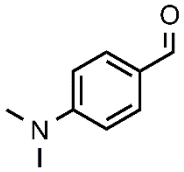


Acetophenone
120.15 g/mol
1.030 g/mL

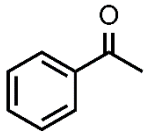
Chalcone-7



(*E*)-1-phenyl-3-(pyrene-2-yl)prop-2-en-1-on
332.40 g/mol
Yield: 84%

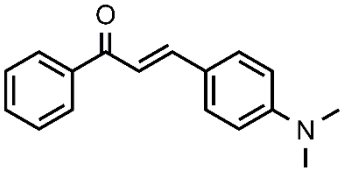


4-(dimethylamino)benzaldehyde
149.19 g/mol

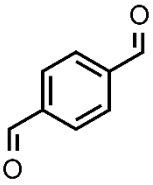


Acetophenone
120.15 g/mol
1.030 g/mL

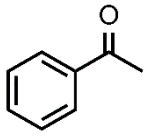
Chalcone-9



4'-dimethylamino-(*E*)-Chalcone
251.33 g/mol
Yield: 92%

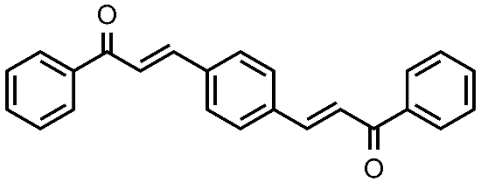


terephthaldheyde
134.13 g/mol



Acetophenone
120.15 g/mol
1.030 g/mL

Chalcone-12



bis-(*E*)-Chalcone
338.41g/mol
Yield: 86%

Chalcone-3 (^{1}H 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 (^{1}H 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, 1.02.

Chalcone-6 (^{1}H 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 (^{1}H 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 (^{1}H 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 (^{1}H 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. In addition, 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 also 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, in addition to confirming the structures of the synthesized *E*-chalcone derivatives through structural characterization, the effects of the functional groups on their properties were also investigated in this context.

The structural characterization of all synthesized compounds was carried out using ^{1}H NMR and ^{13}C NMR spectroscopy.

Examination of the ^{1}H NMR spectra presented in **Figure 4** revealed a coupling constant of approximately 15 Hz for the O=C–CH=CH_2 moiety. This large coupling constant clearly indicates the trans (*E*-) configuration of the double bond. Furthermore, the proton attached to the O=C–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. In addition, analysis of the chemical shifts corresponding to the O=C(R)–CH=CH_2–R moiety showed that, for each *E*-chalcone 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 ^{1}H 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.

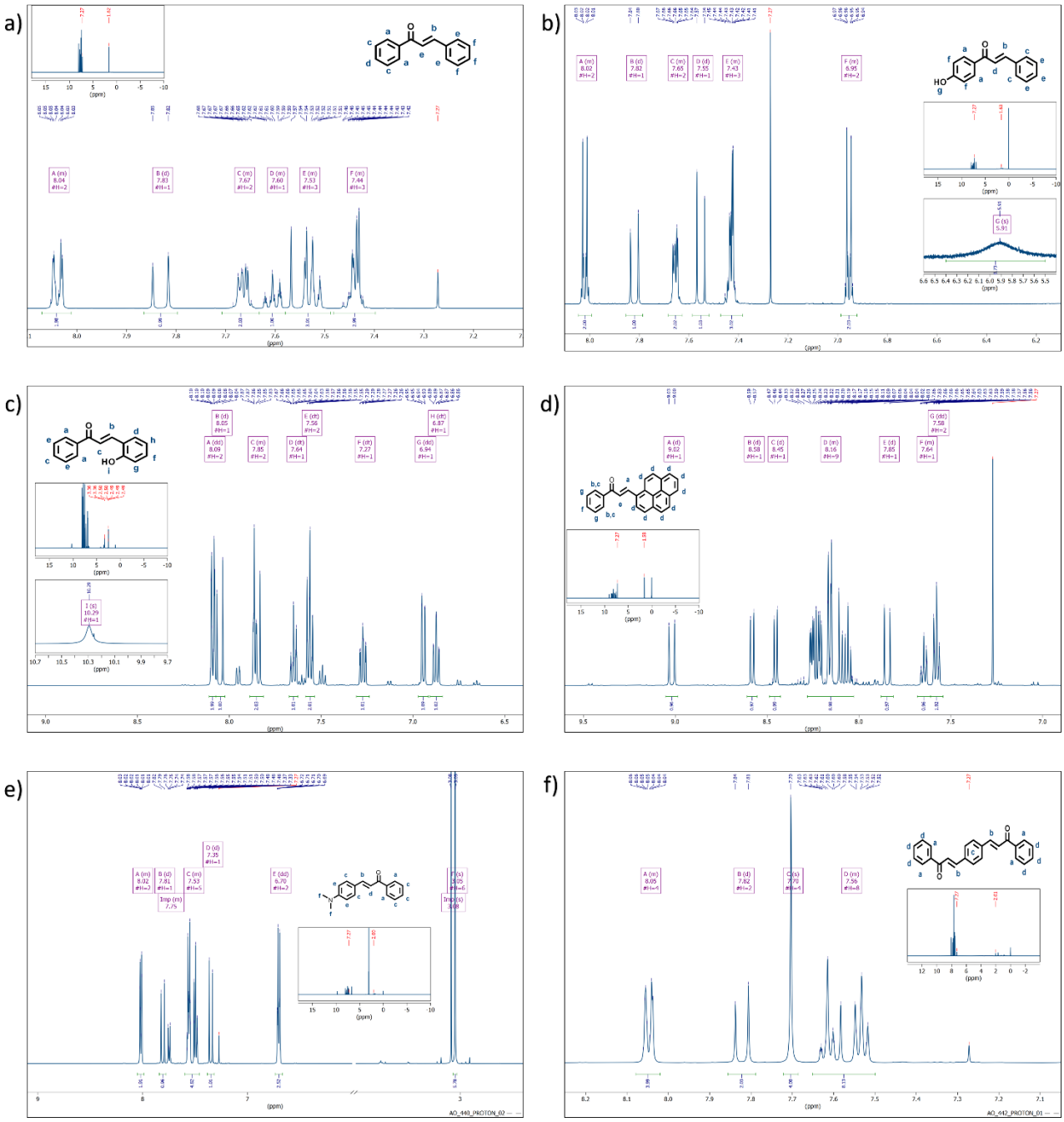
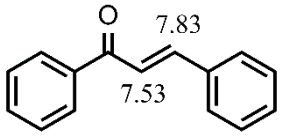
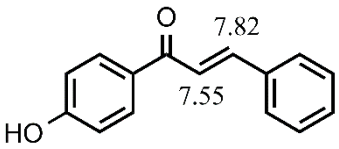
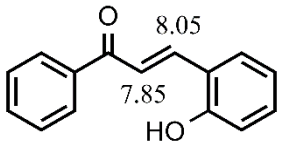
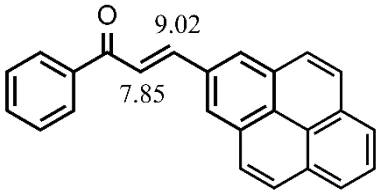
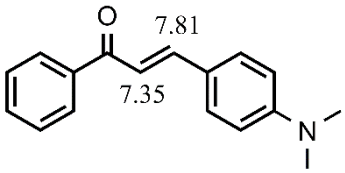
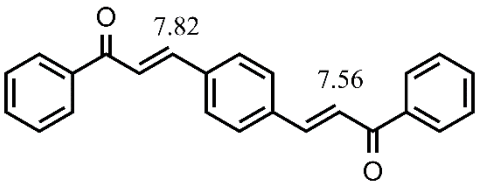


Figure 4. ^{1}H NMR spectra of Chalcone-3 **(a)**, Chalcone-5 **(b)**, Chalcone-6 **(c)**, Chalcone-7 **(d)**, Chalcone-9 **(e)** and Chalcone-12 **(f).**

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

Synthesized Derivative	Schematic representation and NMR chemical shifts of the α- and β-protons	Chemical Shift Analysis
Chalcone-3		Reference Compound
Chalcone-5		• Although the -OH group is an electron-donating substituent, no significant effect was observed due to: • The electron-withdrawing nature of the carbonyl group • Its distance from the carbon-carbon double bond
Chalcone-6		• Although the -OH group is an electron-donating substituent, the opposite effect was observed due to: • Intramolecular hydrogen-bond formation involving the β-proton • An additional downfield shift of approximately 0.5 ppm caused by the use of DMSO-d_6 as the NMR solvent.
Chalcone-7		• The high electron density of the pyrene moiety arises from its extensive π-electron delocalization, resulting in reduced shielding. • This effect is observed for the proton at the β-position.
Chalcone-9		• The $N(CH_3)_2$ group is an electron-donating substituent. • An increase in shielding is observed. • This effect is more pronounced at the α-position than at the β-position, which can be explained by electron delocalization within the *α,β*-unsaturated system.
Chalcone-12		• It does not contain any functional groups different from those of the reference compound.

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.

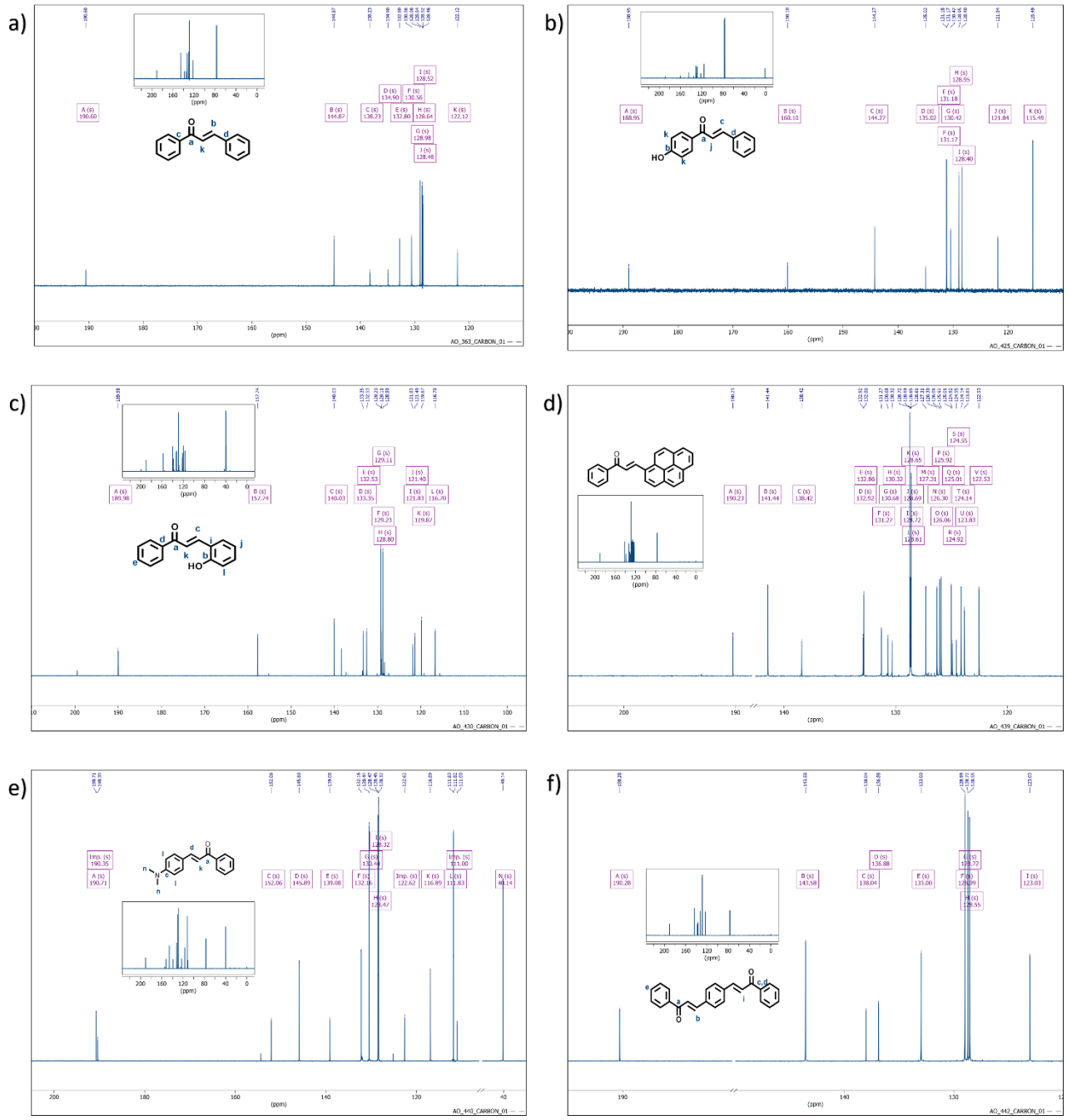


Figure 5. ^{13}C NMR spectra of Chalcone-3 **(a)**, Chalcone-5 **(b)**, Chalcone-6 **(c)**, Chalcone-7 **(d)**, Chalcone-9 **(e)** and Chalcone-12 **(f).**

The UV–Vis absorption spectra and the corresponding calibration curves of Chalcone-3, Chalcone-5, Chalcone-6, and Chalcone-7 are presented in **Figure 7**. Examination of the UV–Vis absorption spectra clearly demonstrates the electron-donating effect of the hydroxyl group through a red (bathochromic) shift in the maximum absorption wavelength. Furthermore, comparison of Chalcone-5 and Chalcone-6 shows that positioning the hydroxyl group on the aldehyde-derived aromatic ring increases the number of possible resonance structures within the conjugated system. As a result, a noticeable broadening of the absorption band is observed in the UV–Vis spectra. In addition, the UV–Vis absorption spectrum of Chalcone-6 exhibits a distinct double-peaked profile. Unlike the other derivatives, the absorption of Chalcone-7 is red-shifted to approximately 400 nm, which can be attributed to the presence of the pyrene moiety. Comparison with the absorption spectrum of pyrene itself clearly demonstrates the optical changes induced by the *α,β*-unsaturated conjugated system.

In addition to the UV–Vis absorption study, the fluorescence emission spectrum of Chalcone-7 was recorded because of its pyrene functionality, and the corresponding calibration curve is presented in **Figure 6**.

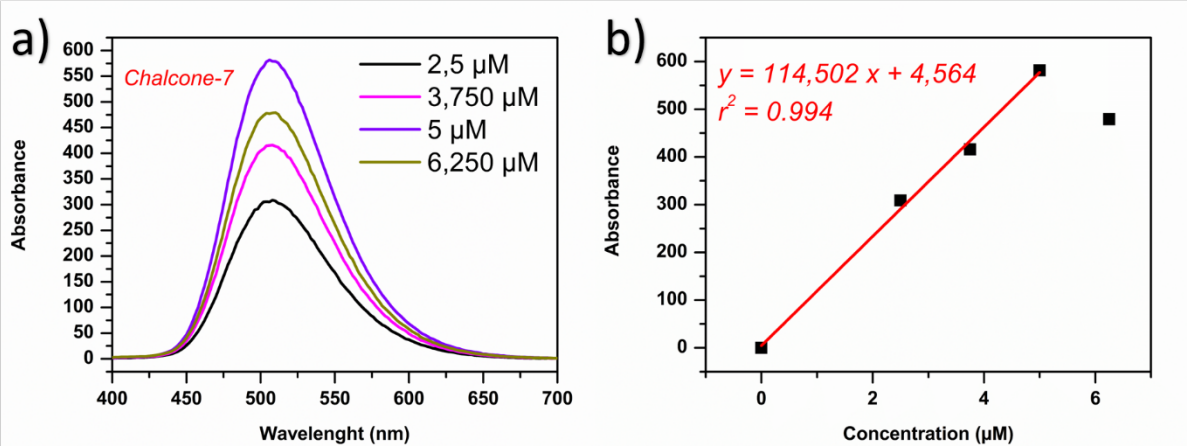


Figure 6. Fluorescence emission spectrum of Chalcone-7 **(a)** and the corresponding calibration curve **(b).**

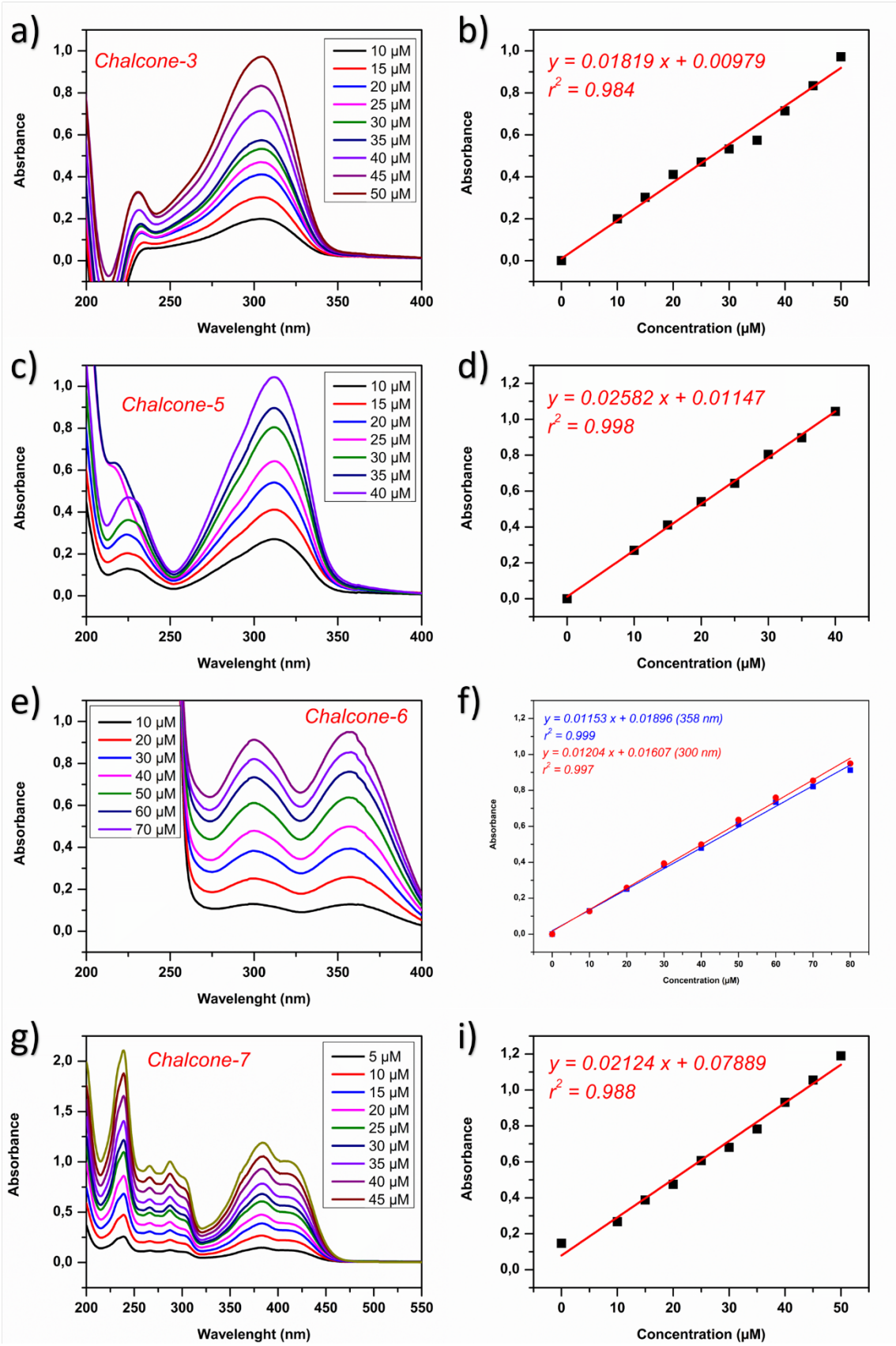


Figure 7. UV–Vis absorption spectrum of Chalcone-3 **(a)** and the corresponding calibration curve **(b);** UV–Vis absorption spectrum of Chalcone-5 **(c)** and the corresponding calibration curve **(d);** UV–Vis absorption spectrum of Chalcone-6 **(e)** and the corresponding calibration curve **(f);**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

4. Discussion

E-Chalcone derivatives, which are characterized by their high product yields and low synthetic cost, are important synthetic intermediates because they allow facile modulation of the optical and electronic properties of the final products. Considering all the studies conducted within the scope of this study, the targeted *E*-chalcone derivatives were successfully synthesized. Structural characterization not only confirmed the identities of the synthesized compounds but also enabled investigation of the effects of the functional groups attached to the chalcone scaffold on both the NMR characteristics and the optical properties of the molecules. The primary motivation for this investigation is that the present thesis constitutes a fundamental component of a broader research project. The chalcone derivatives were successfully synthesized from substituted benzaldehyde and acetophenone derivatives via the Claisen–Schmidt condensation reaction.

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DECLARATIONS

SECTION	*STATEMENT / DISCLOSURE*
Author Contributions	**Görkem Özcan:** Data curation, Formal analysis, Investigation, Methodology, Writing – original draft. **Bleda Can Sadikogullari:** Data curation, Formal analysis, Investigation, Methodology, Writing – original draft. **Emre Can Uysal:** Data curation, Formal analysis, Investigation, Methodology, Writing – original draft. **Ayse Daut Ozdemir:** Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Supervision, Validation, Writing – review and editing. All authors have read and agreed to the published version of the manuscript.
Conflict of Interest	The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.
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Declaration of Generative AI	During the preparation of this work, a generative AI tool was used to improve the grammar, clarity, and terminological consistency of English texts written by the authors. The generative AI tool did not generate any experimental data, design experiments, or independently produce scientific conclusions. All experimental data, calculations, interpretations, and scientific conclusions presented in this work were reviewed, verified, and approved in their final form by the authors.
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