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Original Article

Synthesis, Spectroscopic Characterization, and Photochromic Behavior of Novel Dichloro-Substituted Chalcones and their Chromene Derivatives.

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Abstract

A special category of flavonoids includes chalcones, which are organic compounds that have the general molecular formula 1,3-diaryl-2-propen-1-one. The characteristic feature of these organic substances is the presence of a conjugated double bond between two phenolic groups attached to one carbon atom, hence making them highly reactive. Due to their simple synthesis methods, these compounds are widely used in the preparation of heterocyclic compounds like chromenes, flavones, and pyrazolines. Chromenes are highly valued due to their photochromic property and are widely used in the field of photochemistry and pharmaceuticals.

This paper seeks to study photochromism in dichloro-chalcones and their corresponding chromenes through synthesis and spectroscopic studies. The chalcone compounds were prepared by the Claisen–Schmidt condensation reaction involving substitution at both benzaldehyde and acetophenone moieties. The compounds were analyzed for their photochemical properties by exposing them to chloroform solutions and xenon irradiation light. Chalcones were exposed to photochemical transformation into chromenes, and this was proven by the presence of new absorption wavelengths in the visible range.

The chromenes possessed absorption maxima in the 485–576 nm range. Photodynamic equilibrium occurred earlier among the hydroxy-substituted derivatives, and the bathochromic shift was explained in terms of conjugation and charge transfer effects.

Keywords: *Photochromism, Chalcones; Chromene derivatives; Spectroscopic characterization; Molecular switching devices.*

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1. Introduction

1.1 Conjugated organic systems and chalcones

Organic molecules possessing conjugated π-electron systems are one of the main subjects in modern chemistry, encompassing many areas from biological signals and medicine to material sciences. One of such systems is represented by a class of chalcones (1,3-diaryl-2-propen-1-ones). Structurally, chalcones contain two aromatic rings, bridged by an unsaturated carbonyl system. As a result, conjugated π-electron systems provide chalcones with unique photochemical characteristics.

Chalcones are widespread in nature, being found in many fruits, vegetables, tea, and other foods. They serve as the intermediate compound in the biosynthesis of some flavonoids, e.g., flavones and anthocyanins. The electrophilic nature of the keto-ethylene group (-CO-CH=CH-) makes chalcones good substrates for various chemical transformations, such as cycloadditions and Michael additions.

1.2 Structure and isomers of chalcones

Two types of geometric isomers are known for chalcones – E and Z. Due to the conjugated double bond and increased molecular planarity, the trans E-isomer is more stable compared to its less conjugated cis-Z counterpart. However, the cis-form of chalcones can adopt either s-cis or s-trans conformations, with the former being energetically favorable in terms of electronic delocalization, according to density functional theory calculations.

Conjugation of the π-system allows chalcones to absorb light, which serves as the basis of their photochemical activity. Absorption of ultraviolet and visible light is necessary for various photochemical reactions like photoisomerization and photochemical cyclization.

1.3 Chromenes and photochromic properties

Chromenes (benzopyrans) are heterocycles resulting from chalcones' intramolecular cyclization. The photochromic properties of these compounds make them particularly valuable for practical purposes, including optical data storage, smart coatings, molecular switches, and photoresponsive sensors. In simple terms, photochromism is the reversible transformation of a compound into another form with a changed spectrum of absorption when exposed to electromagnetic radiation.

For chalcone derivatives, this process typically starts with the photoisomerization of the carbon-carbon double bond, followed by intramolecular cyclization with the formation of a chromene molecule. Depending on conditions, the resulting chromene can also open up a new pyran cycle and form colored merocyanine and quinonoid species. In the majority of cases, these compounds are characterized by significant bathochromic shifts and can absorb visible light.

1.4 Synthesis and biological significance of chalcones

Synthetic approach to obtaining chalcones typically relies on the use of the Claisen–Schmidt condensation reaction. In this case, the aromatic aldehyde reacts with an acetophenone derivative in the presence of a base to yield chalcone. Dehydration of the β-hydroxy ketone intermediate produces an α,β-unsaturated carbonyl system typical for chalcones.

Biologically active compounds include chalcones, owing to the presence of the reactive α,β-unsaturated carbonyl fragment. This functionality interacts with biological nucleophiles and affects cell functions, providing chalcones with antibacterial, antifungal, anti-inflammatory, and anticancer properties. Hydroxyl-containing chalcones serve as powerful antioxidants.

1.5 Photochromism mechanisms

Several mechanisms of photochromism can be employed with chalcone systems. The main one is the light-triggered isomerization of the carbonyl double bond. Some substituted chalcones can undergo subsequent ring closure, resulting in a chromene moiety. Chromenes are capable of displaying photochromic behaviour via reversible opening of the pyran ring. Such transformation results in coloured substances with additional conjugation. Stability, rate of thermal bleaching, and photochromic properties of this reaction strongly depend on the influence of substituents, solvent, and structural parameters.

1.6 Recent advances in the field of photochromism

The development of photochromic materials has seen many recent achievements. Substitution of a particular group with a methyl or other radical can improve fatigue resistance of a substance and affect bleaching rates. Halogen substituents play an especially important role here.

To widen the applicability of these compounds, visible-light-responsive photochromic systems were also investigated. These systems can be used without the harmful ultraviolet light irradiation and are particularly useful in biomedical applications.

Furthermore, photochromic compounds can display nonlinear optical (NLO) behavior, phosphorescence, and photoluminescence. This opens the possibility for use in optoelectronics. Another field of application includes photosensitive sunscreen creams based on the high molar absorptivity and tunability of chalcone's absorption spectrum. They can also be used for photodynamic therapy due to strong light absorption.

Recent studies concerning chalcone and chromene chemistry shed light on their photochemistry, structural diversity, and application prospects. For example, Maity et al. [2] introduced the concept of the development of switchable chalcone-flavylium systems in aqueous solutions, stressing the significance of moving towards the visible light-induced reactions of chalcones. As the authors suggest, such a tendency is connected with the need for developing environmentally friendly and safe photoresponsive substances. This aspect is especially relevant for biological applications since chalcones are highly compatible with aqueous environments. In turn, previous studies conducted by Maggiani et al. [8] concerned the photochemistry of 2H-chromenes. Specifically, it was revealed that substituents play an important role in the stabilization of colored merocyanines, thus improving their resistance to photodegradation. The persistence of chromenes and their resistance to photodecomposition were investigated in detail by Mandal et al. [9]. According to the obtained results, stereoelectronic factors and bulky groups affect the lifetime of the corresponding o-quinonoids.

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It should be noted that the photochemical and thermochromic behavior of various chromone derivatives was investigated by Gáplovský et al. [11], which proves that such molecules can demonstrate photo- and thermally-induced structural transformations. Concerning the structural aspects of chalcone chemistry, Zerpa et al. [12] and Hormaza et al. [13] conducted several synthetic studies, focusing on the versatility of the Claisen-Schmidt condensation reaction in creating photoactive chalcone derivatives with various chemical characteristics. These findings provide a foundation for synthesizing chalcone derivatives with the required photochemical properties.

Functional applications of chalcone derivatives are discussed in previous publications as well. For example, Wijayanti et al. [17] considered using these compounds as novel sunscreens because of their high absorbance and good molar absorptivity, which is determined by the π-electron systems of these substances. Also, in their article, Pozzo et al. [18] described photochromic chromenes with increased conjugation, whose absorption spectra exhibit considerable bathochromism, supporting the findings of the current study. Finally, the general synthetic methodology of chalcone production is confirmed by previous literature [19].

Finally, the photophysics of chalcone derivatives and their biological activity are extensively explored in the literature. Thus, Vasanthi et al. [20] demonstrated that these compounds have photoluminescent properties related to the electron mobility in donor-acceptor complexes. At the same time, Nowakowska [22] emphasized that chalcones are characterized by various biological activities, among which antimicrobial and anti-inflammatory effects were found to be especially prominent because of the reactive nature of their α,β-unsaturated carbonyl system.

1.7 Research Significance

Design of substituted chalcone/chromenes (e.g., dichloro-substituted chalcones) is an important step in developing efficient photochromic systems. Manipulation of the electronic environment can lead to considerable improvement of the desired characteristics.

Knowledge of the relationship between molecular structure and photochromic properties of the chalcone family is crucial for future development of molecular switches and smart materials.

Chapter 2: Methodology

2.1 Experimental Design

The proposed project involved an experimental laboratory research of synthesizing dichloro-chalcones and their photochemical transformation to form chromene derivatives. The study integrated aspects of organic chemistry, spectroscopy, and photochemistry in order to investigate the dependence of photochromic properties upon molecular structure and the effect of substituents on absorption spectra and photochemical behavior of the products.

Particular attention was paid to the examination of how the presence of a substituent affected absorption properties, photochromism, and the kinetics of chalcones synthesized.

2.2 Study Location

The work was performed in the laboratory of medicinal chemistry at the College of Pharmacy, University of Duhok, Kurdistan region, Iraq. The equipment necessary for organic chemistry, spectroscopy, and photochemistry was provided by the laboratory; in particular, the UV-Visible spectrophotometer was available. All tests were completed in the period between January 2025 and March 2026. Throughout the course of the study, all laboratory conditions were maintained, especially the room temperature, which was kept steady at around 25°C. Ventilation and handling of chemicals according to safety precautions were also guaranteed. Quartz cuvettes and a 150W xenon lamp were utilized during the photochemical investigations.

2.3 Synthesis of Dichloro-Chalcone Derivatives

Dichloro-chalcones were synthesized via Claisen-Schmidt condensation – one of the widely applied reactions for the preparation of chalcones. In the present investigation, equimolar quantities (0.01 mol) of substituted benzaldehyde (e.g., 4-hydroxybenzaldehyde or 4-nitrobenzaldehyde) reacted with 6,8-dichloro-substituted acetophenone. These substances were dissolved in a minimal volume of absolute ethanol (20-50 mL) inside a round-bottomed flask.

To the stirred reaction mixture, a catalytic quantity of aqueous or alcoholic solution of sodium hydroxide (NaOH 10-20%) was dropwise added within 15-30 min. Hydroxide ions contributed to the formation of enolates from acetophenone derivatives that underwent electrophilic addition to carbonyl carbon atoms of benzaldehydes, thus yielding chalcone skeletons. At this stage, the reaction mass was vigorously stirred until the end of the reaction at room temperature. If the hydroxyl group was introduced to the aromatic ring, the reaction occurred rapidly, producing precipitation after 4-6 hours, while nitro-substituted aldehydes needed more time (24 h) due to the electron-withdrawal effect. The extent of the reaction was monitored by TLC.

Upon completion of the reaction, the product was poured into cold water (100-200 mL). When needed, the reaction mixture was neutralized with diluted hydrochloric acid (HCl) to obtain better precipitation. The crude product was filtered off and cleaned of other substances with cold ethanol several times. Chalcones were purified via crystallization in absolute ethanol or a chloroform-methanol mixture. The obtained crystals were dried in a vacuum prior to their characterization and photochemical investigation.

2.4 Spectroscopic and Photochromic Experimental Setup

Photochromic properties of the synthesized compounds were examined using a high-sensitivity UV-Visible spectrophotometer that could accurately measure the spectral changes related to photoinduced transformation of chalcones into chromenes. Chloroform proved to be a good solvent of choice for spectroscopic experiments since it readily dissolved both the starting compounds and photochemical reaction products and was transparent to the light in the 240-700 nm spectral region.

Concentrations of the solutions to be investigated were about 5×10^{-5} M, ensuring linearity of the spectrum with the absorbance less than 1.5. These solutions were put into quartz cuvettes with a path length of 10 mm, suitable for measurements in the ultraviolet and visible regions of the spectrum.

Chalcones' transformation to chromenes was achieved by irradiation of solutions with a 150W xenon lamp. Optical filters were sometimes employed in order to select the irradiation wavelength band – usually in the ultraviolet or near-visible region. During irradiation, the temperature was maintained stable around 25°C using the thermostatically controlled sample holder.

Photoinduction was confirmed by the appearance of new peaks in the visible region (400-600 nm) of the spectrum that corresponded to absorption of light by chromene derivatives. These spectral data were analyzed by peak decomposition software that allowed calculation of the photostationary state (PSS) composition and estimation of the first-order rate constant of the reaction.

Additional recent studies conducted in the region further confirm the significance of applied chemical research in terms of healthcare and forensics. For example, occupational exposure to harmful substances, including lead contamination in Duhok city, underscores the need for the use of accurate analytical techniques and public health awareness [23]. Likewise, innovations in communication among pharmacists, especially using TESOL-oriented strategies, stress the significance of integrating scientific knowledge with patient-oriented care [23]. The forensic science field, specifically the use of standard analytical techniques like HS-GC-FID for alcohol quantification, illustrates the significance of reliable instrumental analyses in everyday laboratory investigations [23].

4. Results and Discussion

4.1 UV-Visible Spectroscopic Analysis

The irradiation of dichloro-chalcone solutions with xenon light triggered a distinct color change, signaling the formation of the corresponding chromene derivatives. Initially, the UV-Vis spectra of the chalcones were dominated by high-intensity absorption bands in the ultraviolet region (λ < 400 nm), characteristic of $\pi\rightarrow\pi^*$ transitions within the conjugated α,β-unsaturated carbonyl system.*

Upon exposure to light, these initial bands decreased in intensity, accompanied by the emergence of new, broad absorption bands in the visible region. The appearance of these bands is attributed to the photochemical intramolecular cyclization of the chalcone into the chromene structure. The wavelength of maximum absorption (λmax) for these new species ranged from 485 nm to 576 nm, indicating a significant bathochromic shift. This can be explained by the elongated conjugation and the electronic environment of the heterocyclic chromene ring.

4.2 Effects of Substituents on Absorption Maxima and PSS Kinetics

The data presented indicate a strong dependence of the spectral and kinetic properties of the compounds on the nature of the substituents attached to the aromatic rings. The introduction of EDGs and EWGs showed a pattern of λmax and times to achieve the photostationary state.

- Hydroxyl Substitution: The hydroxyl-substituted compound showed the fastest response time of 12 minutes to achieve the PSS. The λmax was recorded at 510 nm. The rapid cyclization in this case is likely facilitated by the electron-donating effect of the OH group, which increases the electron density on the oxygen atom participating in the ring closure.
- Dimethylamino Substitution: This strong EDG resulted in a significant bathochromic shift, with

λmax appearing at 545 nm. The time to PSS was slightly longer than the hydroxyl derivative, at 17.5 minutes.

- Nitro Substitution: As a strong electron-withdrawing group, the nitro substituent shifted the absorption maximum even further into the visible region (λmax = 576 nm). However, it required a longer irradiation time (30 minutes) to reach equilibrium, likely due to the deactivation of the excited state or steric effects.
- Bis-chalcone Derivative: The bis-chalcone showed the shortest λmax at 485 nm and required the longest irradiation time (60 minutes) to reach PSS. This suggests that the increased molecular complexity and potential steric hindrance significantly impede the cyclization process.

4.3 Integration of Quantitative Data and Visualizations

The following table summarizes the key photochromic parameters for the synthesized derivatives:

Table 1: Photochromic Data for Synthesized Chalcone Derivatives

Substituent	Time to PSS (min)	Absorption Maxima (nm)
Hydroxyl	12.0	510
Dimethylamino	17.5	545
Nitro	30.0	576
Bis-chalcone	60.0	485

The relationship between the substituent type and the time required to reach the photostationary state is visually represented in the bar chart.

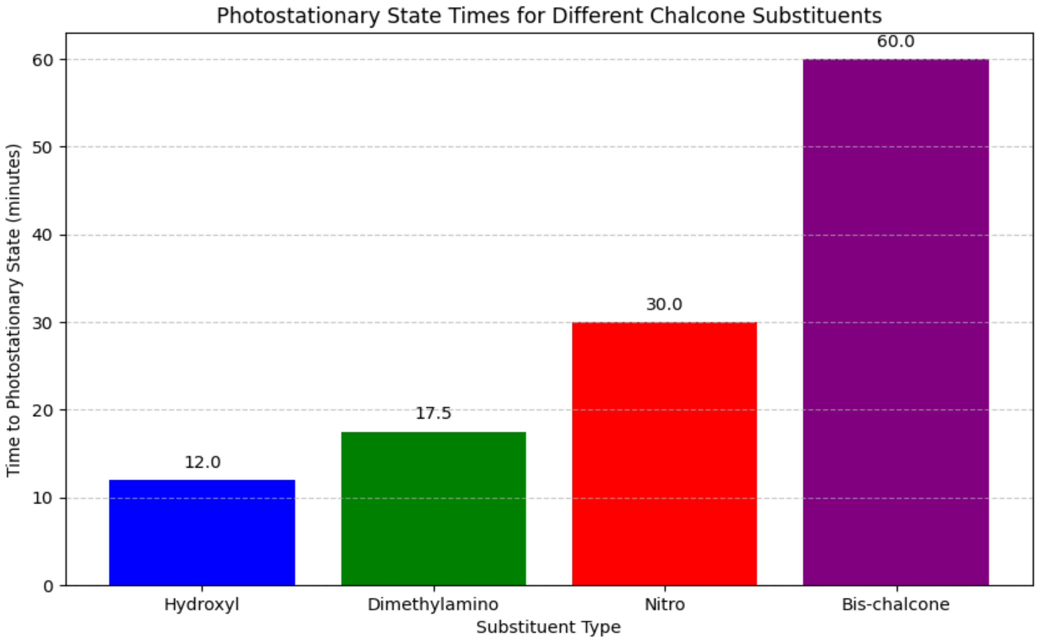

Figure 1: Comparison of the time required to reach the photostationary state (PSS) for different substituted dichloro-chalcones.

Additionally, the relative distribution of irradiation times across the series is shown in the pie chart:

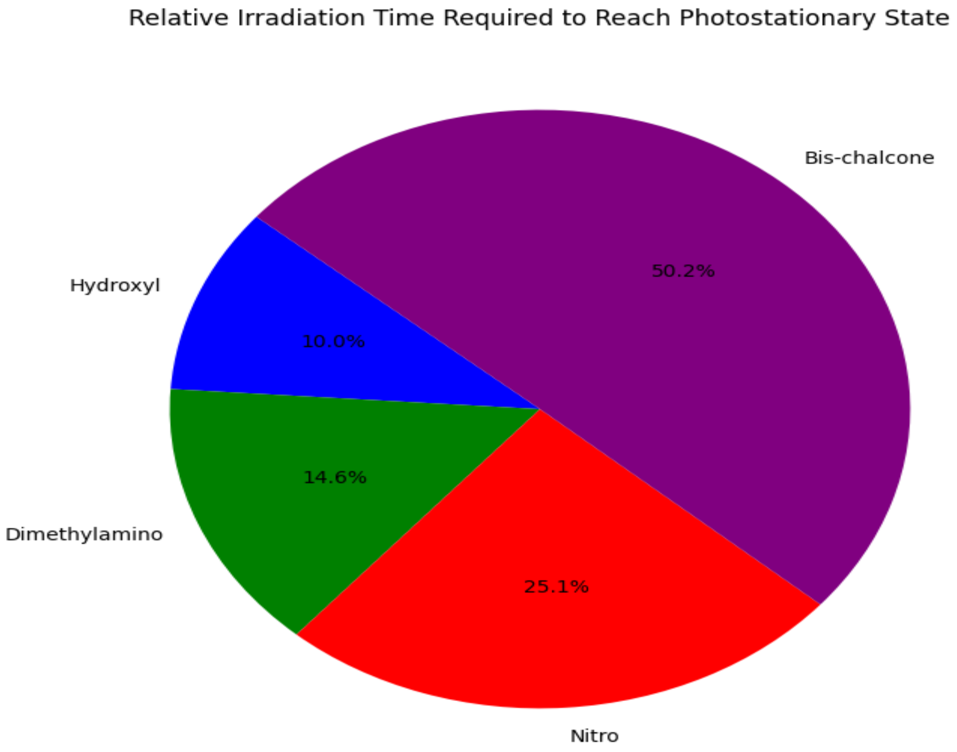

Figure 2: Relative distribution of PSS times among the studied derivatives.

The quantitative analysis clearly shows that electron-donating groups accelerate the cyclization process and provide moderate bathochromic shifts, while electron-withdrawing groups like the nitro group provide the most significant shifts towards longer wavelengths but at the cost of slower kinetics. The bis-chalcone derivative represents a more stable but less reactive system in terms of photochromic conversion.

5. Comparative Analysis

The findings of this study, particularly the work of Nirozh Azad Chalabi, align with and expand upon existing literature in organic photochemistry. The observation of bathochromic shifts upon cyclization is consistent with the general behavior of chromene systems reported by Metelitsa et al. (1997) and Pozzo et al. (1994) [5, 18]. Specifically, the λmax values observed (485–576 nm) are comparable to those reported for other 2H-chromenes with π-donor substituents, which often fall in the 430–680 nm range depending on the degree of annulation [5].

The rapid PSS attainment for hydroxyl-substituted chalcones (12 min) is faster than many traditional photochromic systems, suggesting high efficiency in the cyclization step. This is consistent with the observations of Leydet et al. (2010), who noted that 2-hydroxychalcones exhibit nearly quantitative conversion to the closed form under suitable irradiation [3]. However, the dichloro-substitution in Chalabi's work provides a unique scaffold that may offer improved stability or fatigue resistance, similar to the α-methylated systems studied by Öngel et al. (2021) [1].

The significant shift observed with the nitro substituent (576 nm) corroborates the findings of Rai et al. (2021) regarding the role of strong electron-withdrawing groups in enhancing the electronic response of chalcone-based materials [10]. The slower rate of kinetics of the nitro derivative is in line with the general trend that EWGs can, in certain instances, inhibit photochemical reactions compared to EDGs.

In comparison to the halogenated derivatives studied by Arnall-Culliford et al. (2003), the study of the dichloro-substituted series reveals a well-defined relationship between the substituents of the second aromatic ring and the overall photochromic performance of these compounds [7]. While Arnall-Culliford focused their study on the kinetics of bleaching of merocyanines, the study by Chalabi highlights the forward cyclization, presenting another point of view of the photochromic mechanism.

6. Limitations and Research Gaps

Despite the notable achievements, there are certain limitations and research gaps that need to be considered:

1. In the current research, chloroform was used as a solvent. However, there is no doubt that photochromic compounds are highly responsive to changes in the solvent environment in terms of polarity and hydrogen bond-forming ability (solvatochromism). Both absorption properties and kinetic characteristics of the reactions may be considerably affected by those characteristics of the solvent environment. For this reason, future research should address the impact of various solvents of different polarity, including ethanol, acetonitrile, and toluene, on the performance of the studied compounds.
2. Thermal Stability and Fatigue: While the PSS was successfully reached, the long-term thermal stability of the formed chromenes and their fatigue resistance over multiple cycles (repeatability) were not extensively documented in the available data. This is critical for practical applications in molecular switching.
3. Quantum Yields: Quantitative determination of the quantum yields for both the forward (cyclization) and reverse (ring-opening) processes would provide a more rigorous measure of the photochemical efficiency [3, 6].
4. Computational Validation: While experimental results are clear, integrating Density Functional Theory (DFT) calculations could provide deeper insights into the transition states and the molecular orbitals involved, as suggested by Nirwan et al. (2011) and Sawant et al. (2012) [15, 16].
5. Biological and Material Applications: While the potential for applications in NLO and biomarkers is noted, direct testing in these contexts is still required to confirm the real-world utility of these specific dichloro-derivatives [10, 14].

7. Conclusion

The current research offers a comprehensive analysis of photochromic properties of novel dichloro-chalcone derivatives and their respective chromenes. Through coupling organic synthesis with UV-Vis spectroscopy, the paper highlights the vital importance of molecular structures in the development of photophysical and photochemical characteristics of the compounds. The manipulation of substituents was a critical point, allowing to analyze in detail of the effect of structure on optical absorption and reaction kinetics.

Thus, the results demonstrate that the Claisen-Schmidt condensation is one of the most effective procedures for the preparation of the investigated compounds and allows to obtain dichloro-chalcones, which, after irradiation by a xenon lamp, will cyclize to form chromenes. New absorption bands in the visible range serve as additional evidence of the presence of such substances. At that, bathochromic shifts observed for all investigated compounds within 485 – 576 nm show that there are some extended conjugations in the molecule and, hence, photoactive heterocyclic compounds can be prepared. Finally, the substituent effect turned out to be quite crucial for both reaction speed and spectral features, since electron donor groups accelerate reactions while electron acceptors cause shifts towards higher wavelengths.

Overall, it should be admitted that the paper under discussion reveals several interesting aspects connected with molecular engineering of photoactive compounds and confirms the importance of such approaches in obtaining photochromic materials. In particular, the use of dichloro-chalcone derivatives opens vast opportunities in developing modern photoactive compounds with high efficiency and potential practical applicability.

The major conclusions made include:

- Effective synthesis of dichloro-chalcones through the Claisen-Schmidt condensation and subsequent photochemical cyclization to chromenes.
- Observation of pronounced bathochromic shifts within 485-576 nm, confirming improved conjugations.
- Evidence of essential substituent effect on both reaction kinetics and spectral properties.
- Prospects for further exploitation of dichloro-chalcone derivatives as promising photochromic materials.

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