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An Efficient Synthesis of Novel Type Chalcones Containing 8-hydroxyquinoline Under Green Conditions

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Abstract
In this study, new type of chalcones containing 8-hydroxyquinoline as an alternative to natural and synthetic chalcones have synthesized for the first time using microwave irradiation with environmentally benign approach. Firstly, (E)-1-(4-((8-hydroxyquinolin-5-yl)diazenyl)phenyl)ethanone (**3**) were synthesized by diazotization of p-aminoacetophenone. Secondly, most important step of synthesis included the Claisen-Schmidt condensation of chalcones using the microwave method. The structures of all compounds were characterized by means of FT-IR, NMR and elemental analysis.

Keywords: Azo coupling, Chalcone, Diazotization, 8-Hydroxyquinoline

Yeşil Şartlar Altında 8-Hidroksikinolin İçeren Yeni Tip Kalkonların Etkili Bir Sentezi

Öz
Bu çalışmada, çevreci bir yaklaşımla mikrodalga ışıması kullanılarak doğal ve sentetik kalkonlara alternatif olarak 8-hidroksikinolin içeren yeni tip kalkonlar ilk kez sentezlendi. İlk olarak, (E)-1-(4-((8-hidroksikinolin-5-yl)diazenil)fenil)ethanon (**3**) p-aminoasetofenonun diazotizasyonu tarafından sentezlendi. İkincil olarak sentezin en önemli adımını, mikrodalga yöntemi kullanılarak kalkonların Claisen-Schmidt kondenzasyonunu içermektedir. Tüm bileşiklerin yapıları FT-IR, NMR ve elemental analiz yöntemleriyle karakterize edildi.

Anahtar Kelimeler: Azo kenetlenme, Diazotizasyon, Kalkon, 8-Hidroksikinolin

1. Introduction

The chalcone moieties occur widely in natural and synthetic product (Arslan et al., 2016). They are significant intermediates in organic synthesis. They serve as starting materials for the synthesis of variety of heterocyclic compounds and as substrates for new enantioselective synthetic methods. Chalcones plays also an essential role in the synthesis of many pharmaceuticals (Hua et al., 2014; Abdullah and Salman, 2011). Due to a wide range of biological activities such as diabetes (Hsieh et al., 2012), anticancer (Modzelewska et al., 2006), antimicrobial (Yaylı et al., 2006), antibacterial (Kazuhiro et al., 2011), antifungal (Lopez et al., 2001) and prevent cardiovascular diseases (Furman et al., 2001), the synthesis of chalcone and analogues has received considerable interest. Because of its conjugated system, chalcones with proper electron-pulling and electron-pushing functional groups on the benzene ring(s) can be fluorescent, making them potential chemical probes for mechanistic investigations and imaging/diagnosis. Especially, the dimethylamino group is a widely used substituent in fluorescent probes and has also been introduced into fluorescent chalcone compounds (Zhuang et al., 2017). Aromatic and heteroaromatic azo compounds constitute the most important class of synthetic dyes (Zhao et al., 2011; Hunger 2003). Azo compounds are also involved in a number of biological reactions such as inhibition of DNA, RNA and protein synthesis, carcinogenesis and nitrogen fixation (Ginni et al., 2011; Song et al., 2012). One of the most important compounds

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among the N-heterocycles is the quinoline core, and have found broad application in both the pharmaceutical and agrochemical industries (Szala et al., 2017). It has been discovered in a number of natural products. Due to their a wide range of biological activities, quinoline and analoques have attracted considerable attention. These conjugated systems have been widely employed in components for molecular electronic devices (Szala et al., 2017). 8-hydroxyquinoline is one of the most important derivatives of quinoline because of its chelator properties for significant metal ions (Saylam et al., 2014). Azo compounds based on 8-hydroxyquinoline derivatives play a central role as chelating agents for a large number of metal ions (Saylam et al., 2014). In addition, 8-hydroxyquinoline and its analoques display many different biological activities such as antiparasitic, antibacterial, cytotoxic and antineoplastic, antimycobacterial and antiinflammatory activities (Tosun et al., 2015). In this paper, we have synthesized for the first time a one-pot green synthesis for new type chalcone containing 8-hydroxyquinoline using Claisen Schmidt condensation with a simple friendly benign approach, by the microwave method.

2. Material and Methods

^{1}H and ^{13}C spectra were recorded on a Bruker Ascend 400 (100)-MHz spectrometers and chemical shifts were reported (λ) relative to Me_4Si as internal standard. The elemental analyses were performed on a Costech ESC 4010 instrument. Melting points were determined by using a Barnstead electrothermal 9200 series digital apparatus. The IR spectra were determined using a Perkin Elmer 1600 and JASCO 6600 Fourier Transform-Infrared (FT/IR-ATR) spectrophotometer.

All microwave experiments were carried out in dedicated Anton Paar Monowave-300 reactor. (E)-1-(4-((8-hydroxyquinolin-5-yl)diazenyl)phenyl)ethanone (3) synthesized according to earlier literature (Mchugh et al., 2004; Makhloufa et al., 2017).

General methods for the synthesis of chalcones (5–11).

Dissolve 1 mmol of (E)-1-(4-((8-hydroxyquinolin-5 yl)diazenyl)phenyl)ethanone (3) and respective aldehydes 1 mmol 3 mL of absolute ethanol in G-10 process vial capped with teflon septum. To this, aqueous sodium hydroxide solution 3 mmol was added slowly and mixed in the rt at one minute. Then reaction mixture was microwave irradiated for about 2–6 min at 150 watts. The reactions were monitored through TLC. After completion of the reaction, cool the mixture in an ice-water bath until solids formation is complete. The crude product filtered. The solids was washed with cold water to remove the sodium hydroxide and recrystallized from $EtOH/H_2O$ (1:1) twice to give crystals.

(E)-1-(4-((E)-(8-hydroxyquinolin-5-yl)diazenyl)phenyl)-3-phenylprop-2-en-1-one (5).

Yield: 45 mg. (92 %); mp: 125-126 ^{0}C; IR (ATR), ν/cm^{-1}: 3285, 1675, 1510; ^{1}H NMR (400 MHz, $CDCl_3$): 9.32 (d, 1H, J = 8 Hz), 8.90 (d, 1H, J = 8 Hz), 7.99-8.10 (m, 6H), 7.88 (d, 1H, J = 15.4 Hz,), 7.69 (d, 1H, J = 8 Hz), 7.50 (d, 1H, J = 8.2 Hz), 7.11-7.41 (m, 5H), 4.85 (s,1H, OH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.9, 160.1, 158.2, 148.0, 144.2, 140.8, 139.3, 138.8, 137.5, 137.2, 129.4, 129.0, 128.5, 128.0, 124.7, 122.8, 122.9, 121.2, 120.1, 112.2; Anal. Calcd. for $C_{24}H_{17}N_3O_2$: C, 75.97; H, 4.52; N, 11.08. Found: C, 75.93; H, 4.49; N, 11.03

(E)-1-(4-((E)-(8-hydroxyquinolin-5-yl)diazenyl)phenyl)-3-o-tolylprop-2-en-1-one (6).

Yield: 48 mg. (90 %); mp: 112-113 ^{0}C; IR (ATR), ν/cm^{-1}: 3285, 1675, 1512; ^{1}H NMR (400 MHz, $CDCl_3$): 9.28 (d, 1H, J= 8.2 Hz),

8.94 (d, 1H, J= 8 Hz), 8.07-8.25 (m, 5H), 7.90 (d, 1H, J = 16 Hz,), 7.70 (d, 1H, J = 8 Hz), 7.48 (d, 1H, J = 8 Hz), 7.08-7.40 (m, 5H), 4.85 (s,1H, OH), 2.33 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.9, 160.0, 158.4, 148.2, 143.5, 140.0, 139.1, 136.8, 136.0, 135.4, 129.9, 129.3, 128.6, 127.9, 127.0, 124.1, 122.5, 121.1, 120.8, 120.3, 120.0, 110.8, 20.8; Anal. Calcd. for $C_{25}H_{19}N_3O_2$: C, 76.32; H, 4.87; N, 10.68. Found: C, 76.30; H, 4.83; N, 10.65

(E)-1-(4-((E)-(8-hydroxyquinolin-5-yl)diazenyl)phenyl)-3-m-tolylprop-2-en-1-one (7).

Yield: 46mg. (90 %); mp: 141-142 ^0C; IR (ATR), ν/cm^{-1}: 3285, 1676, 1512; ^1H NMR (400 MHz, $CDCl_3$): 9.30 (d, 1H, J = 8.2 Hz), 8.92 (d, 1H, J = 8 Hz), 7.98-8.10 (m, 6H), 7.85 (d, 1H, J = 16 Hz,), 7.70 (d, 1H, J = 8 Hz), 7.05-7.40 (m, 5H), 4.86 (s,1H, OH), 2.32 (s, 3H, CH_3); ^{13}C NMR (100 MHz, CDCl3): δ 189.9, 160.1, 158.4, 147.8, 144.1, 140.5, 139.4, 138.0, 137.8, 137.6, 137.1, 130.0, 129.7, 129.5, 128.1, 127.3, 124.2, 122.9, 122.1, 121.5, 119.8, 111.0, 21.1; Anal. Calcd. for $C_{25}H_{19}N_3O_2$: C, 76.32; H, 4.87; N, 10.68. Found: C, 76.30; H, 4.84; N, 10.65

(E)-1-(4-((E)-(8-hydroxyquinolin-5-yl)diazenyl)phenyl)-3-p-tolylprop-2-en-1-one (8).

Yield: 45 mg. (90 %); mp: 121-122 ^0C; IR (ATR), ν/cm^{-1}: 3285, 1675, 1514; ^1H NMR (400 MHz, $CDCl_3$): 9.33 (d, 1H, J = 8 Hz), 8.91 (d, 1H, J = 8 Hz), 8.00-8.25 (m, 6H), 7.82 (d, 1H, J = 16 Hz,), 7.68 (d, 1H, J = 8 Hz), 7.06-7.45 (m, 5H), 4.85 (s,1H, OH), 2.32 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.9, 159.8, 158.0, 147.7, 144.0, 140.2, 139.5, 139.3, 137.8, 137.1, 134.2, 130.3, 129.1, 128.4, 122.5, 121.7, 121.3, 121.0, 120.6, 111.8, 20.8; Anal. Calcd. for $C_{25}H_{19}N_3O_2$: C, 76.32; H, 4.87; N, 10.68. Found: C, 76.36; H, 4.91; N, 10.61

(E)-1-(4-((E)-(8-hydroxyquinolin-5-yl)diazenyl)phenyl)-3-(2-methoxyphenyl)prop-2-en-1-one (9).

Yield: 52 mg. (95 %); mp: 132-133 ^0C; IR (ATR), ν/cm^{-1}: 3286, 1676, 1510; ^1H NMR (400 MHz, $CDCl_3$): 9.30 (d, 1H, J = 8 Hz), 8.91 (d, 1H, J = 8 Hz), 8.01-8.30 (m, 6H), 7.85 (d, 1H, J = 15.4 Hz,), 7.69 (d, 1H, J = 8 Hz), 7.02-7.48 (m, 5H), 4.85 (s,1H, OH), 3.92 (s, 3H, OCH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.9, 160.0, 159.3, 158.0, 147.8, 143.5, 140.7, 140.2, 138.8, 137.1, 131.5, 128.6, 128.2, 127.8, 124.1, 123.3, 122.0, 121.5, 121.2, 120.4, 112.8, 112.1, 55.3; Anal. Calcd. for $C_{25}H_{19}N_3O_3$: C, 73.34; H, 4.68; N, 10.26. Found: C, 73.35; H, 4.70; N, 10.30.

(E)-1-(4-((E)-(8-hydroxyquinolin-5-yl)diazenyl)phenyl)-3-(3-methoxyphenyl)prop-2-en-1-one (10).

Yield: 48 mg. (90 %); mp: 110-111 ^0C; IR (ATR), ν/cm^{-1}: 3285, 1675, 1510; ^1H NMR (400 MHz, $CDCl_3$): 9.32 (d, 1H, J = 8.2 Hz), 8.94 (d, 1H, J = 8 Hz), 8.08-8.30 (m, 6H), 7.88 (d, 1H, J = 16 Hz,), 7.70 (d, 1H, J = 8.2 Hz), 7.10-7.49 (m, 5H), 4.85 (s,1H, OH), 3.92 (s, 3H, OCH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.9, 162.7, 160.3, 158.2, 147.8, 143.5, 141.1, 140.9, 138.9, 138.4, 136.6, 129.2, 128.5, 123.0, 122.7, 122.1, 121.3, 121.0, 120.3, 115.8, 114.3, 11.8; Anal. Calcd. for $C_{25}H_{19}N_3O_3$: C, 73.34; H, 4.68; N, 10.26. Found: C, 73.38; H, 4.70; N, 10.29.

(E)-3-(4-bromophenyl)-1-(4-((E)-(8-hydroxyquinolin-5-yl)diazenyl)phenyl)prop-2-en-1-one (11).

Yield: 55 mg. (94 %); mp: 136-137 ^0C; IR (ATR), ν/cm^{-1}: 3285, 1670, 1512; ^1H NMR (400 MHz, $CDCl_3$): 9.30 (d, 1H, J = 8 Hz), 8.95 (d, 1H, J = 8 Hz), 7.98-8.30 (m, 6H), 7.85 (d, 1H, J = 15.4 Hz,), 7.75 (d, 1H, J = 8 Hz), 7.02-7.45 (m, 5H), 4.86 (s,1H, OH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.9, 160.1, 158.3, 147.8, 144.3, 139.9, 139.4, 138.4,

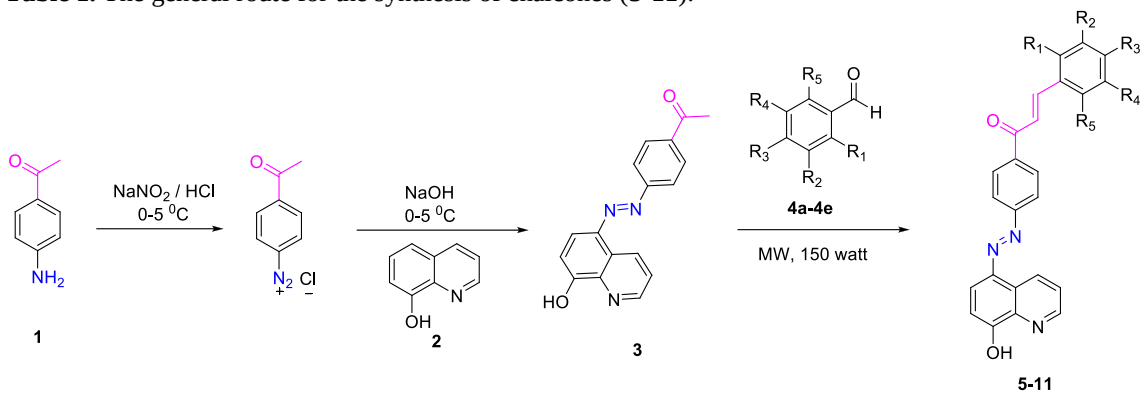
137.1, 136.6, 133.1, 130.5, 128.2, 125.4, 124.0, 122.3, 121.7, 120.5, 120.1, 112.0; Anal. Calcd. for $C_{24}H_{16}BrN_3O_2$: C, 62.90; H, 3.52; N, 9.17. Found: C, 62.94; H, 3.55; N, 9.21.

3. Results and Discussion

Initially, (E)-1-(4-((8-hydroxyquinolin-5-yl)diazenyl)phenyl)ethanone (3) was synthesized by diazotization and azo coupling of p-aminoacetophenone (1) according to earlier developed procedure (Mchugh et al., 2004; Makhloufa et al., 2017). Therefore, it has been converted into the corresponding diazonium salt by using a treatment with sodium nitrite and HCI at 0-5 ^{0}C to give the corresponding benzenediazonium chlorides, which were used directly in the next step without further purification. Subsequently, the diazonium salt was coupled with 8-hydroxyquinoline (2) in a dilute NaOH solution at 0-5 ^{0}C for 4h leading to the formation of (E)-1-(4-((8-hydroxyquinolin-5-yl)diazenyl)phenyl)ethanone (3). The crude product is purified by recrystallization. The FT-IR spectra of initial compound 3, observed a broad O-H stretching vibration band at 3270 cm^{-1}. The characteristic band of C=O stretching showed at 1670 cm^{-1}. The N=N stretching was observed at 1500 cm^{-1} in the FT-IR spectrum (see supplementary data, figure 8 for compound 3). Synthesis of chalcones using the Claisen-Schmidt condensation is a cornerstone of our synthesis strategy. Thus, firstly, chalcone derivatives were synthesized by the Claisen-Schmidt condensation equivalents of the corresponding substituted benzaldehydes (4a-4e) with (E)-1-(4-((8-hydroxyquinolin-5-yl)diazenyl)phenyl)ethanone (3) in the presence of NaOH in EtOH at microwave irradiation. After completion of the reaction, the mixture was purificated by recrystallized affords the pure chalcones (5-11) in good yield. The structures of all the compounds were confirmed by FT-IR, NMR spectroscopy and elemental analysis. The crude product was contaminated with some starting materials which could easily be removed using recrystallization. All synthesized compounds and elemental analysis results of chalcones 5-11 are given in Table 1 and Table 2 respectively.

The FT-IR spectra of chalcone 5, observed a broad O-H stretching vibration band at 3285 cm^{-1}. The characteristic band of C=O stretching showed at 1675 cm^{-1}. This bathochromic shift is due to the conjugation of the π-electrons of the benzene ring and the olefinic linkage. The N=N stretching was observed at 1510 cm^{-1} in the FT-IR spectrum (see supplementary data, figure 1 for chalcone 5). The ^{1}H NMR spectrum of a prototype of these chalcones, 5, olefinic proton adjacent to the carbonyl group appeared as doublet at δ 7.88ppm, with J value of 15.4 Hz. indicating that the ethylene moiety in the enone linkage is in a trans-conformation in the chalcone. The aromatic protons appear at δ 9.32 (d, 1H, J = 8 Hz), 8.90 (d, 1H, J = 8 Hz), 7.99-8.10 (m, 6H), 7.69 (d, 1H, J = 8 Hz,), 7.50 (d, 1H, J = 8.2 Hz), 7.11-7.41 (m, 5H), respectively, and the phenolic proton at δ 4.85 ppm. The trans-(E) geometry of the chalcone 5-11 double bond was evident by the large olefinic coupling constant between the relevant signals in the ^{1}H NMR spectrum (J = 15.4-16.0 Hz). The ^{13}C NMR spectrum of these chalcone indicated characteristic α-β unsaturated carbonyl carbon at δ 189.9 ppm. The olefinic carbons were visible at δ 144.2 and 122.8 ppm respectively. The NMR spectra (^{1}H and ^{13}C NMR) are consistent with the proposed structures. Finally, the structures of all the compounds were confirmed by FT-IR, NMR spectroscopy and elemental analysis. FT-IR spectra of all compunds are given in supplementary data (Figure 1-8)

Table 1. The general route for the synthesis of chalcones (**5-11**).



Compounds	R_1	R_2	R_3	R_4	R_5
5	H	H	H	H	H
6	CH_3	H	H	H	H
7	H	CH_3	H	H	H
8	H	H	CH_3	H	H
9	OCH_3	H	H	H	H
10	H	OCH_3	H	H	H
11	H	H	Br	H	H

Table 2. Elemental analysis results of chalcones **5-11.**

Compounds	**Theoretically calculated (%)**			**Found (%)**		
	C	**H**	**N**	**C**	**H**	**N**
5	75.97	4.52	11.08	75.93	4.49	11.03
6	76.32	4.87	10.68	76.30	4.83	10.65
7	76.32	4.87	10.68	76.30	4.84	10.65
8	76.32	4.87	10.68	76.36	4.91	10.61
9	73.34	4.68	10.26	73.35	4.70	10.30
10	73.34	4.68	10.26	73.38	4.70	10.29
11	62.90	3.52	9.17	62.94	3.55	9.21

4. Conclusion

In summary, we have synthesized a one-pot green synthesis for a novel type of chalcones containing 8-hydroxyquinoline via Claisen-Schmidt condensation between (E)-1-(4-((8-hydroxyquinolin-5-yl)diazenyl)phenyl)ethanone (3) and a variety of substituted benzaldehydes (4a-4e) via an environmentally benign approach, by the microwave irradiation. All structures has been confirmed by FT-IR, NMR and elemental analysis. Chalcones and quinolines are the most valuable synthones for synthetic organic chemists, and by means of proper planning and design of these active compounds can give inspiration for the

synthesis of many important and pharmaceutical compounds.

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