CHEMISTRY & CHEMICAL TECHNOLOGY

Chem. Chem. Technol., 2024,
Vol. 18, No. 3, pp. 350–355

Chemistry

MICROWAVE-ASSISTED SYNTHESIS OF BIS-(HYDROXYBENZYLIDENE)-CYCLOALKANONES *VIA* ACID CATALYZED CLAISEN-SCHMIDT CONDENSATION

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https://doi.org/10.23939/chcht18.03.350

Abstract. In the present study, bis-(hydroxybenzylidene)cycloalkanone derivatives were synthesized by Claisen – Schmidt condensation using cycloalkanones and arylaldehydes in the presence of HCl as an acid catalyst. The synthetic reaction was carried out under microwave irradiation. The structure of the synthesized compounds was determined by UV, IR, ^{1}H NMR spectroscopic methods. The obtained reaction yields were not optimal due to the self-polymerization of p-hydroxybenzaldehyde in an acid solution.

Keywords: bis-(hydroxybenzylidene)cycloalkanones, Claisen – Schmidt condensation, acid catalyst, self-polymerization.

1. Introduction

Curcumin (1,7-bis-(4-hydroxy-3-methoxyphenyl)-1,6-heptadiene-3,5-dione, Fig. 1), a major bioactive phytochemical isolated from the rhizome of turmeric (*Curcuma longa L.*), has been used for centuries as a dietary pigment and spice. There are secondary metabolites in turmeric, namely curcuminoids, which consist of curcumin (the main compound), desmethoxycurcumin, and bisdemethoxycurcumin[1, 2]. Curcumin has been found to have a wide range of traditional pharmaceutical applications in such diseases as external/internal wounds, liver diseases (especially jaundice), blood cleansing, microbial effects, and anti-inflammatory. In addition, curcumin has pharmacological effects such as antioxidant, antimutagenic, antibacterial, anticancer, and may also be neuroprotective[3]. Its utility in clinical practice is limited due to its low aqueous solubility, poor absorption, and metabolic instability, resulting in poor oral bioavailability.

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Curcumin has three different groups, namely a methoxy, a phenolic hydroxyl and a β-diketone group[4] (Fig. 1). The hydroxy phenolic and β-diketone groups play an important role in the antioxidant process as they can act as free radical scavengers[5].

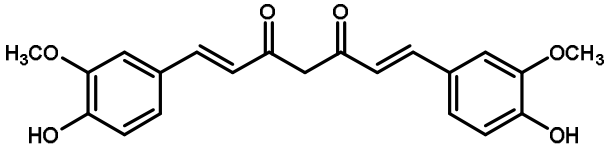


Fig. 1. Curcumin

However, curcumin is not well absorbed in the digestive tract and the presence of the β-diketone group causes curcumin to be easily metabolized into compounds that are more polar and easily excreted, resulting in low plasma concentrations. To overcome this condition, the β-diketone group is modified to a mono-ketone group and it is hoped that the curcumin analogue will become more stable and have equal or greater activity[6, 7]. To overcome the limitations of curcumin, various mono-ketone analogues of curcumin have been synthesized, one of which is a dibenzylidenecycloalkanone derivative.

The compound dibenzylidenecycloalkanone can be synthesized from the reaction between cycloalkanone and substituted benzaldehyde via the Claisen – Schmidt condensation reaction, which can occur in either acid or base catalysts[8]. In the previous study, dibenzylidenecycloalkanone was synthesised using sulphuric acid, hydrochloric acid, sulphamic acid, sodium hydroxide, potassium hydroxide, and NaOH/ZrO_2 montmorillonite[9, 10]. In a previous study, they synthesized the compound 2,6-bis(4-hydroxybenzylidene)cyclohexanone using NaOH as the basic catalyst; however, the reaction occurred with an excess of NaOH solution, take a longer reaction time, and the yield is variable[3]. Therefore, in this study, we carried out the reaction using HCl as the catalyst.

Several methods can be carried out for the synthesis of dibenzylidenecycloalkanone by stirring, reflux,

grinding, sonochemical method, and microwave irradiation. In this study, the synthesis reaction was carried out using microwave irradiation. This method has several advantages: it is more environmentally friendly, easy to handle, shorter reaction time, high purity results[11].

2. Experimental

2.1. Materials

All reagents and chemicals were of p.a. purity, purchased from commercial sources, and used without further purification. The yield of synthesis is calculated from the isolated product. The melting point was determined in an open capillary tube and was not corrected. The synthesis was carried out using a domestic microwave oven. All synthesized compounds were characterized by spectroscopic data (UV, UATR, ^{1}H NMR). UV spectra were recorded on a Shimadzu UV-160. UATR spectra were recorded on a Perkin Elmer UATR spectrophotometer. ^{1}H NMR spectra were recorded at room temperature on a JEOL ECS-400 MHz FT-NMR spectrometer using $CHCl_3$-d or DMSO-d_6 as solvent and tetramethylsilane as internal standard. The purity of the synthesized compounds and the progress of the reaction were monitored by thin layer chromatography (TLC) on Merck pre-coated silica gel 60 F_{254} aluminum sheets, visualized by UV light.

2.2. Methods

Dibenzylidenecycloalkanones (**3a-e**) were obtained from the reaction of cyclopentanone (**1a**) or cyclohexanone (**1b**) with arylaldehyde (**2a-c**) *via* a cross-aldol condensation reaction. The synthesis of compounds (3a-e) was carried out using an acid catalyst, while compounds (**3a-b**) were synthesized using both acid and base catalysts (Fig. 2). All syntheses were carried out under microwave irradiation.

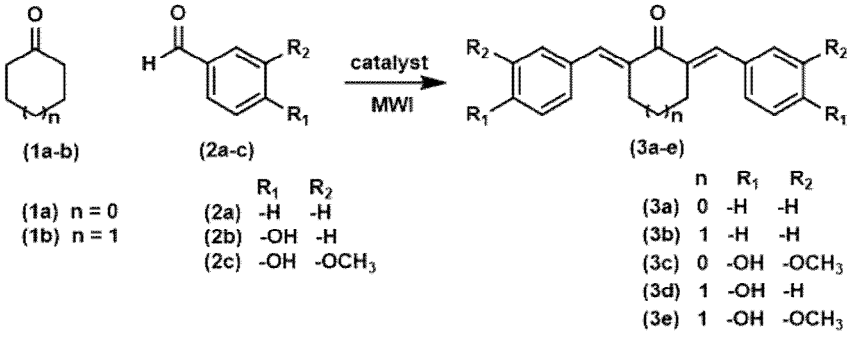


Fig. 2. Reaction pathway for the synthesis of dibenzylidencycloalkanones (3a-e)

General Procedure for (3a-e) by Claisen-Schmidt Condensation in Acid Solution

To a solution of 1 mmol of cycloalkanone in EtOH (2.5 mL), 2 mmol of arylaldehyde was added. The solution was stirred at room temperature for 5 min and then 1 mL of HCl was added dropwise. The mixture was irradiated in a microwave oven at 480 W for an appropriate time. The completion of the reaction was monitored by TLC. When the aldehyde was completely consumed, the reaction mixture was cooled to room temperature. The solid precipitate obtained was filtered on a Buchner funnel, washed three times with 50 % acetic acid solution, filtered, and then washed thoroughly with cold water to remove any acid present. The solid thus obtained was dried and purified by recrystallization.

General Procedure for (3a-b) by Claisen-Schmidt Condensation Using NaOH as a Catalyst

A solution of 1 mmol of cycloalkanone (**1a** or **1b**) and 2 mmol of benzaldehyde (**2a**) in ethanol (2.5 mL) was stirred for 10 min. Slowly add 2 mmol 10 % NaOH solution (0.8 mL) while stirring for 5 min. The mixture was then irradiated in a microwave oven at 480 W for 10 min. The completion of the reaction was monitored by performing TLC. When the aldehyde was completely consumed, the reaction mixture was cooled to room temperature. The solid precipitate obtained was filtered through a Buchner funnel and then washed thoroughly with cold water to remove any alkali present. The solid thus obtained was dried and purified by recrystallization.

Compound (**3a**)

2E,5E-dibenzylidenecyclopentanone (= 2,5-di((E)-benzylidene)cyclopentan-1-one)

Yellowish powder; mp. 191–193 ^{o}C. UV (EtOH, λ_{max}, nm) 356. IR (UATR, ν, cm^{-1}) 3021 (aryl, C-H), 2920 (alkyl, C-H), 1687 (conjugated C=O), 1604 (alkene, C=C), 1601 and 1442 (aryl, C=C). ^{1}H NMR (DMSO-d6, *d*, ppm) 7.59 (*d*, *J* = 8.4 Hz, 4H), 7.57 (*s*, 2H), 7.47–7.32 (*m*, 6H), 3.09 (*t*, *J* = 1.2 Hz, 4H)

Compound (**3b**)
2Ed6E-dibenzylidenecyclohexanone (= 2,6-di((E)-benzylidene)cyclohexan-1-one)
Yellow powder; mp. 127–129 °C. UV (EtOH, λ_{max}, nm) 328. IR (UATR, ν, cm^{-1}) 3058 (aryl, C-H), 2909 (alkyl, C-H), 1660 (conjugated C=O), 1604 (alkene, C=C), 1571 and 1442 (C=C Ar). ^{1}H NMR ($CDCl_3$, *d*, ppm) 7.81 (*s*, 2H), 7.46 (*d*, *J* = 7.7 Hz, 2H), 7.40 (*t*, *J* = 7.4 Hz, 4H), 7.33 (*t*, *J* = 7.2 Hz, 4H), 2.93 (*t*, *J* = 4.2 Hz, 4H), 1.78 (*q*, 2H).

Compound (**3c**)
2E,5E-bis(3-methoxy-4-hydroxybenzylidene)cyclopentanone (= 2,5-bis((E)-4-hydroxy-3-methoxy-benzylidene)cyclopentan-1-one)
Brownish yellow powder; mp. 206–208 °C. UV-Vis (EtOH, λ_{max}, nm) 427. IR (UATR, ν, cm^{-1}) 3441 (br, OH), 3294 (aryl, C-H), 2833 (alkyl, C-H), 1616 (conjugated C=O), 1581 and 1519 (aryl, C=C) ^{1}H NMR (DMSO d-6, *d*, ppm) 7.32 (*s*, 2H), 7.20 (*s*, 2H), 7.12 (*dd*, *J* = 8.4 dan 2.0, 2H), 6.85 (*d*, *J* = 8.2, 2H, C-H), 3.80 (*s*, 6H), 3.02 (*s*, 4H).

Compound (**3d**)
2E,6E-bis(4-hydroxybenzylidene)cyclohexanone (= 2,6-bis((E)-4-hydroxybenzylidene)cyclohexan-1-one)
Greenish yellow powder; UV (EtOH, λ_{max}, nm) 375. IR (UATR, ν, cm^{-1}) 3254 (*br*, OH), 1651 (conjugated C=O), 1595 and 1572 (aryl, C=C) ^{1}H NMR (DMSO *d*6, *d*, ppm): 9.91 (*s*, 2H), 7.50 (*s*, 2H), 7.41–7.32 (*m*, 4H), 6.84–6.76 (*m*, 4H), 2.84–2.76 (*m*, 4H), 1.66 (*q*, *J* = 6.1 Hz, 2H).

Compound (**3e**)
2E,6E-bis(4-hydroxy-3-methoxybenzylidene)cyclohexanone (= 2,6-bis((E)-4-hydroxy-3-methoxy-benzylidene)cyclohexan-1-one)
greenish yellow powder; mp. 193–194 °C; UV (EtOH, λ_{max}, nm) 389. IR (UATR, ν_{max}, cm^{-1}) 3360 (*br*, OH), 1639 (conjugated C=O), 1575 and 1468 (aryl, C=C), 1284 and 1038 (alkyl ether C-O). ^{1}H NMR (DMSO-*d*6, *d*, ppm): 7.52 (*s*, 2H), 7.06 (*d*, *J* = 2, 2H), 6.99 (*s*, 2H), 6.81 (*d*, *J*= 8.2, 2H), 3.77 (*s*, 6H). 2.84 (*m*, 4H), 1.68 (*p*, *J* = 6.0, 2H).

3. Results and Discussion

In this experiment we carry out a type of acid-catalyst crossed aldol condensation called the Claisen – Schimdt reaction. The aldol reaction is widely used to synthesize new C-C bonds. In a crossed aldol synthesis, two different aldehydes or ketones (or a ketone and an aldehyde) react in the presence of a dilute base to give β-hydroxyaldehydes or β-hydroxyketones. In most base-catalyst aldol reactions, the end product is an α,β-unsaturated aldehyde (or ketone) and a separate molecule of water. A Claisen – Schmidt reaction product always has at least one double bond conjugated to both a carbonyl group and an aromatic ring.

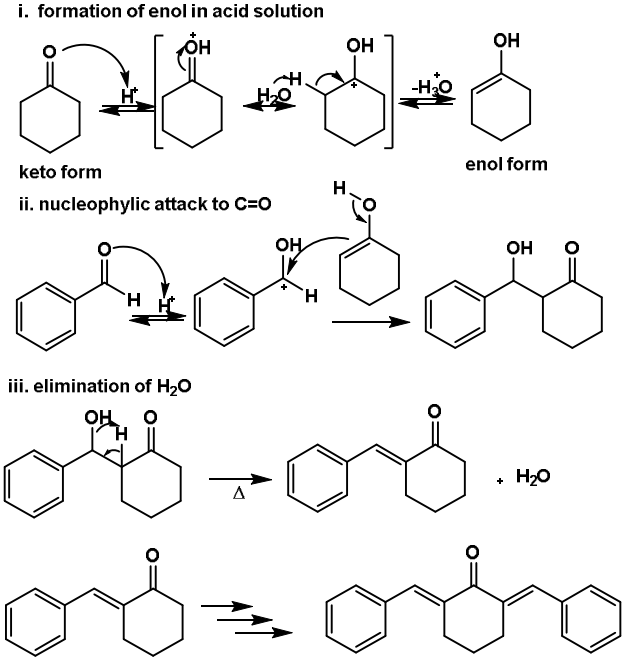


Fig. 3. Reaction mechanism of Claisen – Schmidt condensation in acid solution

Compounds (3a-e) were obtained *via* crossed aldol condensation between cycloalkanones and arylaldehyde using HCl as the catalyst. The reaction mechanism of crossed aldol condensation in the presence of acid as catalyst was carried out in three steps: (i) the acid catalyzes the keto-enol tautomerization of the cycloalkanone (ii) the enol form of the cycloalkanone, a nucleophile, attacks the protonated carbonyl of the aryl aldehyde (iii) elimination of H_2O to produce an α,β-unsaturated carbonyl (Fig. 3). As cycloalkanone has two Cα, both will be added by the benzylidene group.

3.1. Identification of Compounds (3a-e)

From the IR spectra of compounds (**3a-e**) there were new peaks at 1616–1687 cm^{-1} identified as conjugated carbonyl and the UV spectra showed a bathochromic shift compared to the spectra of the starting aryl aldehyde. The absence of a band at 2700 cm^{-1}, which is a specific band for the C-H aldehyde, confirms the absence of the -CHO group of the aryl aldehyde. This is supported by the ^1H NMR spectra, which show no signal in the chemical shift region of about 10 ppm for the aldehyde proton. The presence of two singlet protons with chemical shifts of 7.8–7.3 ppm in each compound of (**3a-e**) proved that the two benzylidene groups are attached to the carbonyl in (E, E) geometric form. The benzene ring was observed at 1595-1442 cm^{-1} in IR spectra and 6.81–7.52 ppm in ^1H NMR.

The presence of the OH-phenolic group of (**3c-e**) was also observed in the IR and 1H NMR spectra. The OH-phenolic in compound (3d) appeared at a chemical shift of 9.91 ppm. In compounds (**3c**) and (**3e**), the OH-phenolic appeared at 2.47 ppm and 3.3 ppm, respectively. This was due to the methoxy group attached to the adjacent carbon of OH phenolic giving an inductive or bond anisotropy effect which caused the OH proton to appear as a signal at an up-field chemical shift[12]. The methoxy group of (**3c**) and (**3e**) appeared as a singlet signal of 6 protons at 3.80 and 3.77 ppm respectively.

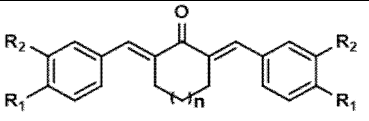
3.2. Influence of the Aryl Aldehyde Substituent

In this research, the syntheses of (3a-e) were successfully carried out by microwave irradiation. The basic mechanism of microwave irradiation is caused by the agitation of polar or ionic molecules which move due to the movement of the magnetic field. The occurrence of these magnetic motions causes the particles to attempt to orient or parallel themselves to the field, with the movement of the particles being limited by the interaction between the particles and the dielectric resistance. This results in heat being generated on the magnetic plate. Microwave irradiation differs from conventional heating because in conventional heating the oil bath or heating jacket is heated first, followed by its solvent. This type of heat distribution causes heat differences between the mantle and the solvent[11].

By microwave irradiation, the synthesis of compounds (**3c-e**) required a longer irradiation time than the synthesis of compounds (**3a-b**). The presence of OH in the para position of aryl aldehydes (**2b-c**) decreased the electrophilicity of the C=O of benzaldehydes, so that the reaction time for nucleophilic attack was longer. The addition of a methoxy group bonded to the adjacent carbon of the OH phenolic also decreased the electrophilicity of the C=O, as evidenced by the lower yields of (**3c**) and (**3e**) compared to the yield of (**3d**).

In an open capillary tube, the synthesized compounds give a sharp melting point, except (**3d**) which decomposed on heating (Table 1). In a previous study, the melting point of (**3d**) can be observed by DSC method[13].

Table. 1. Physical data and reaction time of synthetic compounds



Compound Code	Cyclic, n	Substituent		Catalyst	Irradiation, min	Yield, %	Mp (^0C)	
		R_1	R_2				Obs	Lit.
(**3a**)	0	H	H	HCl	10	38	191–193	196–198[14]
				NaOH	10	77		
(**3b**)	1	H	H	HCl	10	35	117–118	109–112[15]
				NaOH	10	80		
(**3c**)	0	OH	OCH_3	HCl	17	48	206–208	212–214[16]
(**3d**)	1	OH	H	HCl	17	76	Decomp.	282.48[13]
(**3e**)	1	OH	OCH_3	HCl	17	55	193–194	210–212[17] 178–179[18]

3.3. Influence of Catalysts

Compounds (**3a**) and (**3b**) were obtained from the cross-aldol condensation reaction of cycloalkanones (**1a-b**) and benzaldehyde (**2a**), both with acid or base catalysts. The synthesis reaction carried out with the base catalyst gave higher yields (Table 1). In the acid catalyzed cross-aldol condensation, acid is required to establish the keto-enol equilibrium of the cycloalkanone. This is useful because the enol form is a nucleophile. When the cross-aldol condensation is carried out in NaOH solution, the strong OH base directly forms an enolate ion of the cycloalkanone. The enolate ion is a stronger nucleophile than the enol form, so it reacts more easily with the C=O of benzaldehyde. At the same time of irradiation (*i.e.*, 10 minutes, Table 1), the reaction carried out with the base catalyst gave a higher yield.

If we synthesize compounds (**3c-e**) from cycloalkanone (**1a-b**), arylaldehyde (**2b-c**), and NaOH solution in (1:2:2) molar ratio, the reaction cannot occur. Arylaldehydes (2b) and (2c) contain OH phenol, which is converted to phenoxide ion in the alkaline solution. Consequently, the enolate ion of the cycloalkanone is not formed.

Alternatively, the synthesis of compounds (**3c-e**) was carried out in an acidic solution. During the reaction, a dark brown by-product was formed and removed by repeated washing with 50 % acetic acid solution[19]. It is known that the condensation reaction of phenol with formaldehyde in the acidic solution produced a dark brown polymer called *Bakelite*[20]. Arylaldehydes (**2b-c**) contain OH-phenolic in para-position and aldehyde group without Ha, so *self-polymerization* in arylaldehydes (**2b-c**) is very likely to occur. This means that the yield of the reaction cannot be maximized.

4. Conclusions

Compounds (**3a-b**) can be synthesized from cycloalkanones (**1a-b**) and benzaldehyde (**2a**), both with acid or base catalysts, while compounds (**3c-e**) were obtained from the cross-aldol condensation of cycloalkanones (**1a-b**) and arylaldehydes (**2b-c**) using HCl as the catalyst. The reaction was successfully carried out in a domestic microwave oven. The presence of the OH group on aryl aldehydes (**2b-c**) caused a longer irradiation time. The yield of synthetic compounds (**3c-e**) cannot be maximized due to the *self-polymerization* of p-hydroxybenzaldehyde in the acidic solution.

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Received: August 01, 2023 / Revised: September 14, 2023 / Accepted: January 07, 2024

МІКРОХВИЛЬОВИЙ СИНТЕЗ БІС-(ГІДРОКСИБЕНЗИЛІДЕН)-ЦИКЛОАЛКАНОНІВ КИСЛОТНО-КАТАЛІЗОВАНОЮ КОНДЕНСАЦІЄЮ КЛЯЙЗЕНА – ШМІДТА

Анотація. *У роботі синтезовано похідні біс-(гідроксибензиліден)-циклоалканонів конденсацією Кляйзена – Шмідта з використанням циклоалканонів та арилальдегідів у присутності HCl як кислотного каталізатора. Реакцію синтезу здійснено в умовах мікрохвильового опромінення. Будову синтезованих сполук визначено методами УФ-, ІЧ-, ^{1}Н ЯМР-спектроскопії. Одержані виходи реакції не були оптимальними через спонтанну полімеризацію п-гідроксибензальдегіду в кислому розчині.*

Ключові слова: *біс-(гідроксибензиліден)-циклоалканони, конденсація Кляйзена – Шмідта, кислотний каталізатор, спонтанна полімеризація.*