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Green and Eco-Friendly Strategies to Perform a New Series of Chromene Carbonitrile Derivatives Incorporating of Hydrazineyilidene Thiazolidenone Moiety.

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ABSTRACT: 4H-chromene-3-carbonitrile derivatives have a great deal of attention as six-membered heterocyclic compounds containing oxygen that possess broad spectrum of biological ,pharmacological and industrial activity, and also it was prepared via multicomponent reactions using one of the most important green chemistry techniques; grinding and microwave irradiation. The first step involved the preparation of hydrazineylidene thiazolidinone as active building block (H1) through a multicomponent reaction, employing grinding followed by microwave irradiation at (400 W) for (2 min) between thiosemicarbazide and p-hydroxy benzaldehyde. The resulting intermediate (l) was subsequently subjected to cyclization with ethyl chloroacetate using grinding and microwave irradiation at (200 W) for (8 min) to prepare (H1). Finally, the compound (H1) was employed as a key component in a one-pot multicomponent reaction with malononitrile and substituted benzaldehydes to achieve chromene carbonitrile derivatives (H2 – 8). All prepared compounds were illustrated by the available physical and spectral methods.

1. INTRODUCTION

Chromenes, also known as benzopyrans, are important heterocyclic compounds consisting of a phenyl ring fused with an oxine ring(Katiyar et al., 2022). Chromenes are structurally classified into two main categories, namely 4H-chromene and 2H-chromene, depending on the position of the double bond and the hydrogen atom within the pyran ring(Liu et al., 2025). This type of heterocyclic compounds have demonstrated significant anticancer activity. Recent studies have shown that 4H-chromene derivatives exhibit selective cytotoxicity toward various cancer cell lines, including breast cancer (Sant et al., 2026), lung cancer (Abbaspour et al., 2024), and colon cancer (Yousif et al., 2023). Furthermore, the presence of functional groups such as amino and cyano groups within the chromene framework can enhance the antitumor activity of these compounds by increasing their ability to interact with biological targets within cancer cells (Elshemy et al., 2022).

The growing importance of chromene derivatives can be attributed to their broad spectrum of biological activities. These compounds have been reported to exhibit antiproliferative(Abdelall et al., 2022; Mansouri et al., 2011), antioxidant (Badiger et al., 2024; Vanga et al., 2024), antimicrobial (Agrawal et al., 2024; Gaikwad et al., 2025), anti-inflammatory (Chung et al., 2016; dos Reis et al., 2022a, 2022b) , and antimalarial activities (Parthiban et al., 2015) , in addition to its anti-obesity and antiviral activities (Chávez-Jiménez et al., 2025; Inthanon et al., 2025; Kadu et al., 2026) , as well as other important biological applications.

The low toxicity of chromene, structural variety, high effectiveness, and lipophilic nature, which facilitates its penetration into biological membranes, have contributed to the increasing research interest in the development of these heterocyclic compounds

(Dash & Kumar, 2023).Therefore, chromene derivatives remain important targets in the design and development of pharmaceutical compounds, while their distinctive properties continue to stimulate the development of modern synthetic methodologies in organic chemistry(Bibak & Poursattar Marjani, 2023; Raj & Lee, 2020).

According to the above advanteges of chromene, in this work we prepared a novel type of chromene derivatives including an active unite building represented by hydrazineylidene thiazolidinone in order to increase its activity.

2. MATERIAL AND METHODS

All chemicals in this study are produced by Fluka and BDH. The melting point of the prepared compounds were measured using apparatus model (SMP 10), the FT-IR were measured by Burker FT-IR device in region between (400-4000 cm-1). The H1-NMR were measured by Burker ascent device (400 MHz). The measurement was carried out in university Of Basrah. TMS as an internal reference, Using DMSO as a solvent.

2.1 Synthesis of (E)-2-(((E)-4-hydroxybenzylidene)hydrazineylidene)thiazolidin-4-one. (H_1) (Ayyash et al., 2014; El-Naggar et al., 2021)

A mixture of equimolar (1mmol) of 4-hydroxybenzaldehyde and thiosemicarbazide were placed in a beaker (50ml) in an acidic medium (1ml) of glacial acetic acid. The mixture was thoroughly grinding with a glass rod for (1 min) to ensure complete homogeneity, then the resulting mixture was subjected to microwave irradiation at (400 W) for (2 min). After cooling, an alcoholic ethyl chloroacetate solution (1ml ethanol /1.1ml), as well as anhydrous sodium acetate (1mg) added to the previously prepared reaction mixture. The mixture was subsequently subjected to microwave irradiation at (200 W) for (8 min). The progress of the reaction monitored by (TLC). Upon completion of the reaction, the reaction mixture allowed to cool, poured into ice-water. The precipitate was collected by filtration then dried and recrystallized from appropriate solvent to produce compound 91% as yellow crystal and m.p> 300 0C.

2.2 Synthesis of 2-amino-6-((1E)-((4-oxothiazolidin-2-ylidene)hydrazineylidene) methyl)-4H-chromene-3-carbonitrile compounds. ($H_{2\text{-}8}$). (Kidwai et al., 2005, 2010)

In a beaker (50 ml), equimolar amounts (1mmol) of the H1, malononitrile and substituted benzaldehyde were mixed in a (1ml) of saturated potassium carbonate (K_2SO_4 / H_2O) solution. The mixture was thoroughly grinding with a glass rod for (1 min) to ensure complete homogeneity, then the resulting mixture was subjected to microwave irradiation (MWI) at a power of (400–600 Watt) for (1–3 min). The progress of the reaction were monitored by (TLC). TLC using a benzene: chloroform solvent system (1:7, v/v). Upon completion of the reaction, the reaction mixture allowed to cool, poured into ice-water (20ml) with shaking. The precipitate was collected by filtration then washed thoroughly with cold water, dried and recrystallized from appropriate solvent to produce compound ($H_{2\text{-}8}$) in good yield with physical properties listed in Table (1).

Table (1). Physical data of compound ($H_{2\text{-}8}$)

Comp. No.	Molecular Formula	M.Wt	M.P (°C)	Power (Watt)	Time (min)	Yield (%)	Color	R_f Benzene Chloroform (7:1)
H2	$C_{22}H_{17}N_5O_2S$	415	101-102	400	3	86	Brown	0.743
H3	$C_{21}H_{17}N_5O_3S$	419	276-278	600	2	90	Yellow	0.642
H4	$C_{20}H_{14}ClN_5O_2S$	423	278-281	600	2	74	Yellow	0.681
H5	$C_{20}H_{15}N_5O_2S$	389	274-276	600	2	94	Yellow	0.593
H6	$C_{21}H_{15}N_5O_4S$	433	284-286	600	3	90	Green	0.582
H7	$C_{20}H_{15}N_5O_3S$	405	308-310	400	2	83	Yellow	0.395
H8	$C_{21}H_{17}N_5O_4S$	435	292-294	400	2	82	Yellow	0.610

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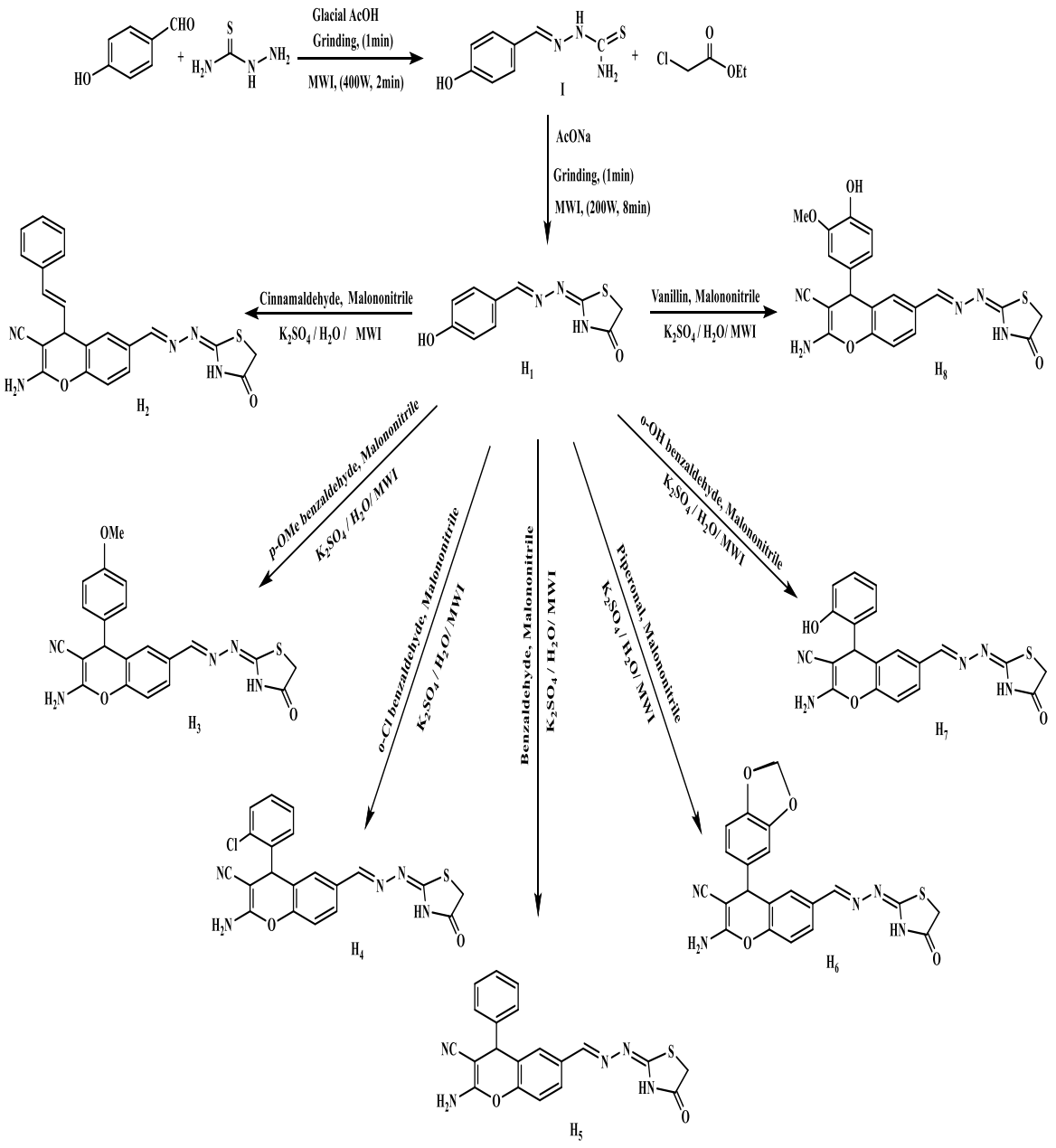
3. RESULTS AND DISCUSSION

The novel 2-amino-4H-chromene-3-carbonitrile compounds (H_{2-8}) have been prepared as show in the Scheme (1), the reaction of H_1, malononitrile and substituted benzaldehyde via one-pot multicomponent reactions using green chemistry techniques represented by grinding and microwave irradiation with yield enhancement. This novel efficient method has the advantages of mild reaction conditions, simple work-up, high efficiency, atom economy, short reaction times, low cost and green reaction conditions.



Scheme (1): Synthetic pathway for compounds (H_{2-8})

As outlined in Scheme 2, the proposed reaction sequence proceeds through Knoevenagel condensation, followed by Michael addition then intramolecular cyclization reaction to afford the 4H-chromene carbonitrile derivatives.

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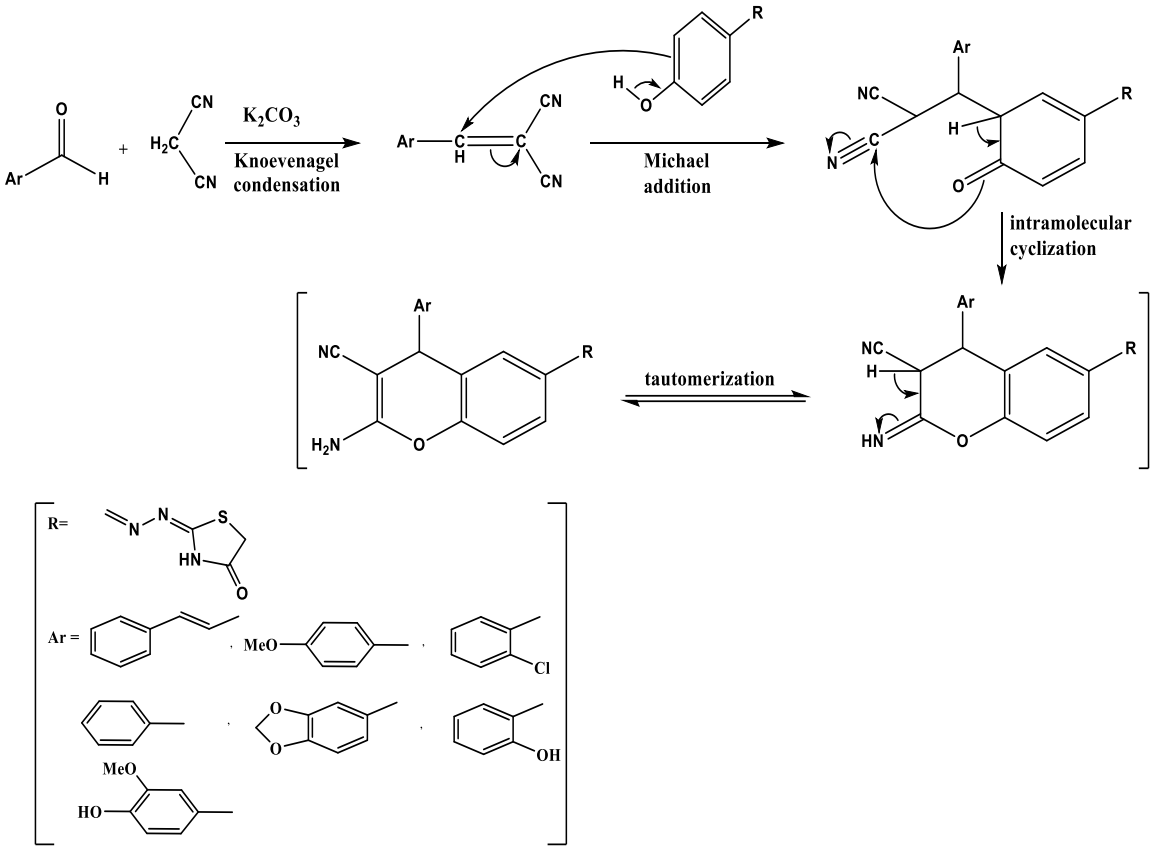

Scheme (2): Synthesis mechanism of compounds (H_{2-8})

The measurement of compound (H_1) are consistent with those published in scientific literature, m.p: (dec.300 ˚C).

The FT-IR spectra was shown abroad band of NH at (3171 cm^{-1}) and C=O lactam at (1686 cm^{-1})(Ayyash et al., 2014). Whereas the FT-IR spectra of compounds (H_{2-8}) was shown distinctive absorption bands in the range of (3441–3216 cm^{-1}), which belong to the primary (NH_2) group. In addition to the sharp absorption bands appeared in the range of (2226–2194 cm^{-1}), belong to the (CN) group. These spectral features provide strong evidence supporting the proposed structural assignments of the synthesized compounds. Furthermore, the other absorption bands also supported the validity of these structures, and their data are listed in the Table (2).

Table (2). FT-IR data of compounds (H_{2-8})

Comp No.	NH_2	NH	CN	C=O	C=N	C-O-C		Others
						asym	sym	
H2	3333	3030	2221	1673	1606	1177	1071	C=C =1575
H3	3216	3151	2222	1684	1640	1163	1021	CH_3: asym =2924 sym =2690

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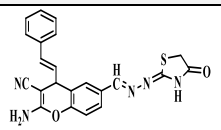
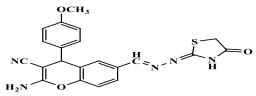
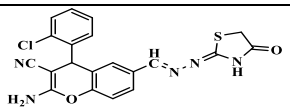
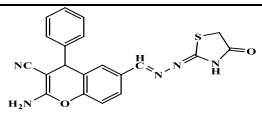
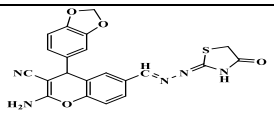
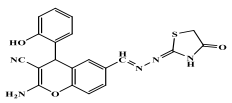
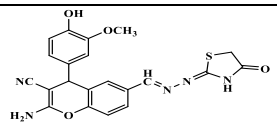
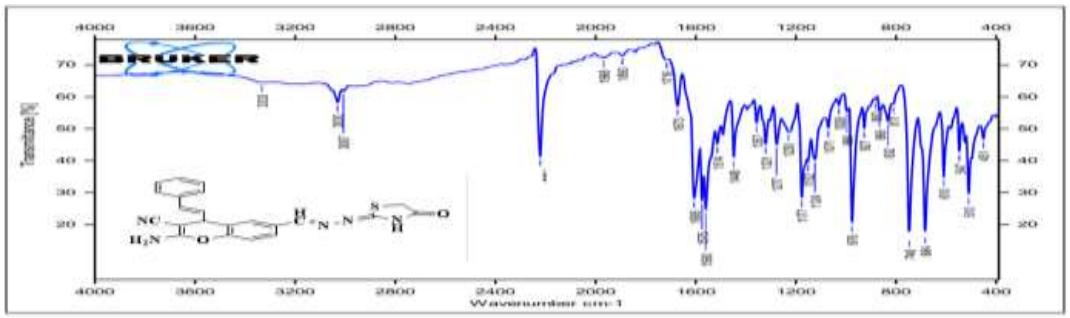
H4	**3220**	**3152**	**2224**	**1684**	**1639**	**1229**	**1051**	**C-C l =756**
H5	**3247**	**3170**	**2194**	**1684**	**1640**	**1163**	**1079**	———
H6	**3214**	**3033**	**2226**	**1686**	**1640**	**1229**	**1034**	———
H7	**3350**	**3167**	**2204**	**1684**	**1640**	**1230**	**1080**	**OH=3441**
H8	**3155**	**3036**	**2199**	**1684**	**1639**	**1230**	**1080**	**OH=3224**

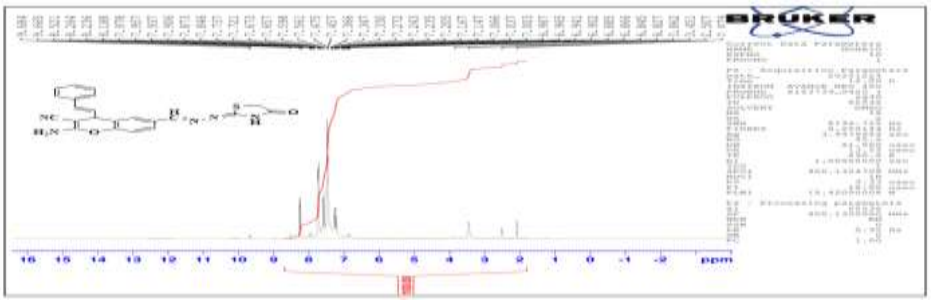
In the ^{1}H-NMR spectra, the prepared compounds exhibited characteristic signals at the expected chemical shifts, Clear signals attributable to the protons of the NH_2, CH_2-thiazolidinone, and NH–N groups were observed, in agreement with the proposed structures of the synthesized compounds. These results, together with the FT-IR spectroscopic data, contributed to confirming the structural identities of the synthesized compounds, as all observed spectral signals could be appropriately assigned to their corresponding protons, as presented in Table (3).

Table (3). ^{1}H-NMR data of compounds ($H_{2\text{-}8}$)

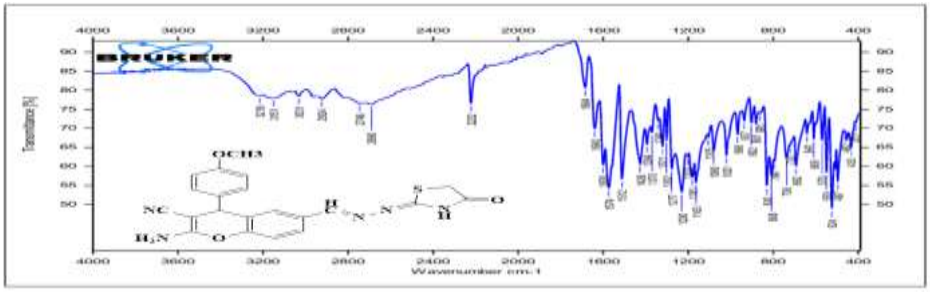
Comp.No.	Structure	^{1}H-NMR, δ (ppm)
H_2		**NH_2(s, 2.079, 2H) ; CH_2-thiazolon (s, 3.862, 2H) ; CH=CH (d-d, 6.827-6.885) ; Ar-H (m, 6.902-7.978 , 9H) ; CH=N (s, 8.23, 2H) , NH-N (s, 8.264,1H).**
H_3		**NH_2(s, 3.826, 2H) ; OCH_3 (s, 3.868, 3H) ; CH_2-thiazolinon (s, 3.876, 2H) ; Ar-H (m, 6.849 -7.982 , 8H) ; CH=N (s, 8.380, 1H) , NH-N (s, 8.432,1H).**
H_4		**NH_2(s, 3.870, 2H) ; CH_2-thiazolinon (s,4.008, 2H) ; Ar-H (m, 6.833 -8.746 , 8H) ; CH=N (s,10.044, 1H) , NH-N (s,10.145,1H).**
H_5		**NH_2(s, 3.898, 2H) ; CH_2-thiazolone (s, 4.012, 2H) ; Ar-H (m, 6.832 -7.830 , 9H) ; CH=N (s,8.281, 1H) , NH-N (s,8.389,1H).**
H_6		**NH_2(s, 3.866, 2H) ; CH_2-thiazolinone (s,4.011, 2H) ; CH_2- piperonal(s,4.408,2H) ; Ar-H (m, 6.112 -7.648 , 7H) ; CH=N (s,8.272, 1H) , NH-N (s,8.358,1H).**
H_7		**NH_2(s, 3.869, 2H) ; CH_2-thiazolinone (s, 4.012, 2H) ; Ar-H (m, 6.828 -7.808 , 8H) ; CH=N (s, 8.186, 1H) , NH-N (s, 8.277,1H)**

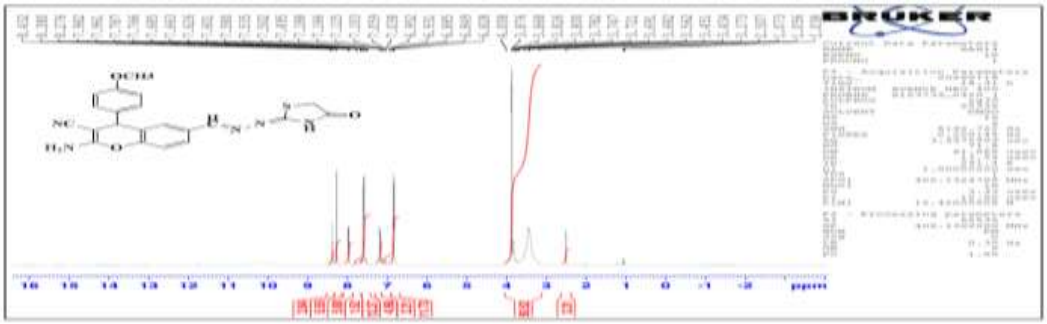
		OH (s, 8.371, 1H).
H_8		NH_2(s, 3.707, 2H) ; CH_2-thiazolinone (s,3.862, 2H) ; Ar-H (m, 6.573 -7.771 , 7H) ; CH=N (s,8.188, 1H) , NH-N (s,8.276,1H) ; OH(s,9.80,1H).





H_2





H_3

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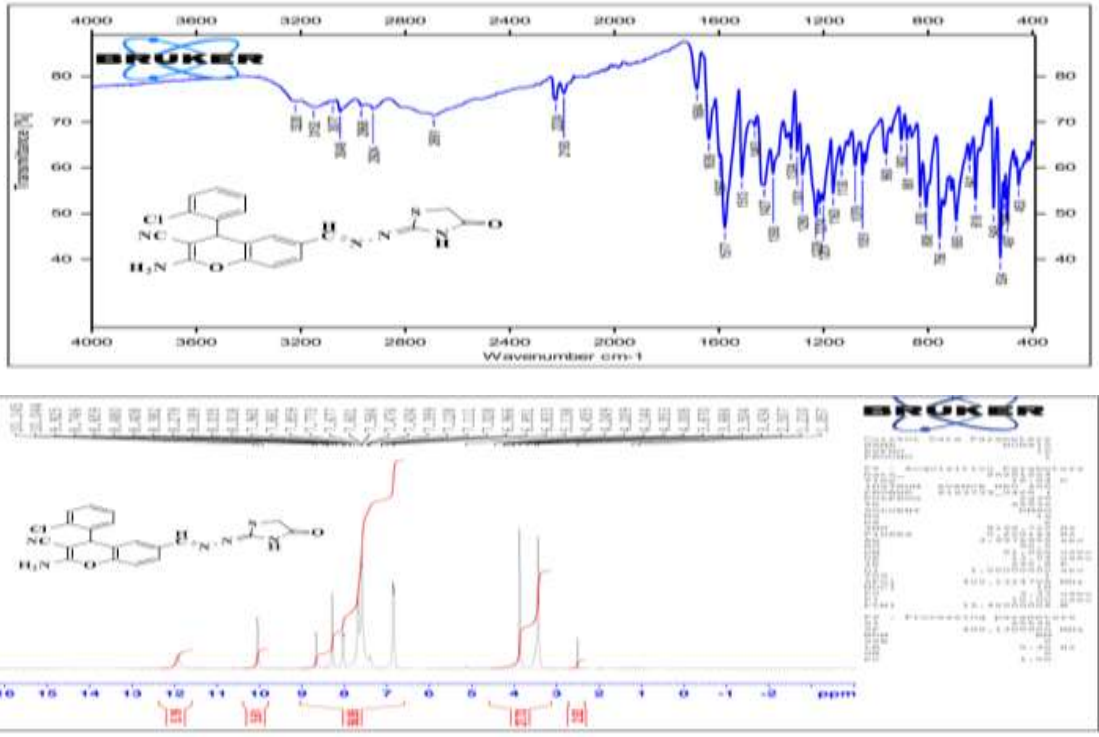
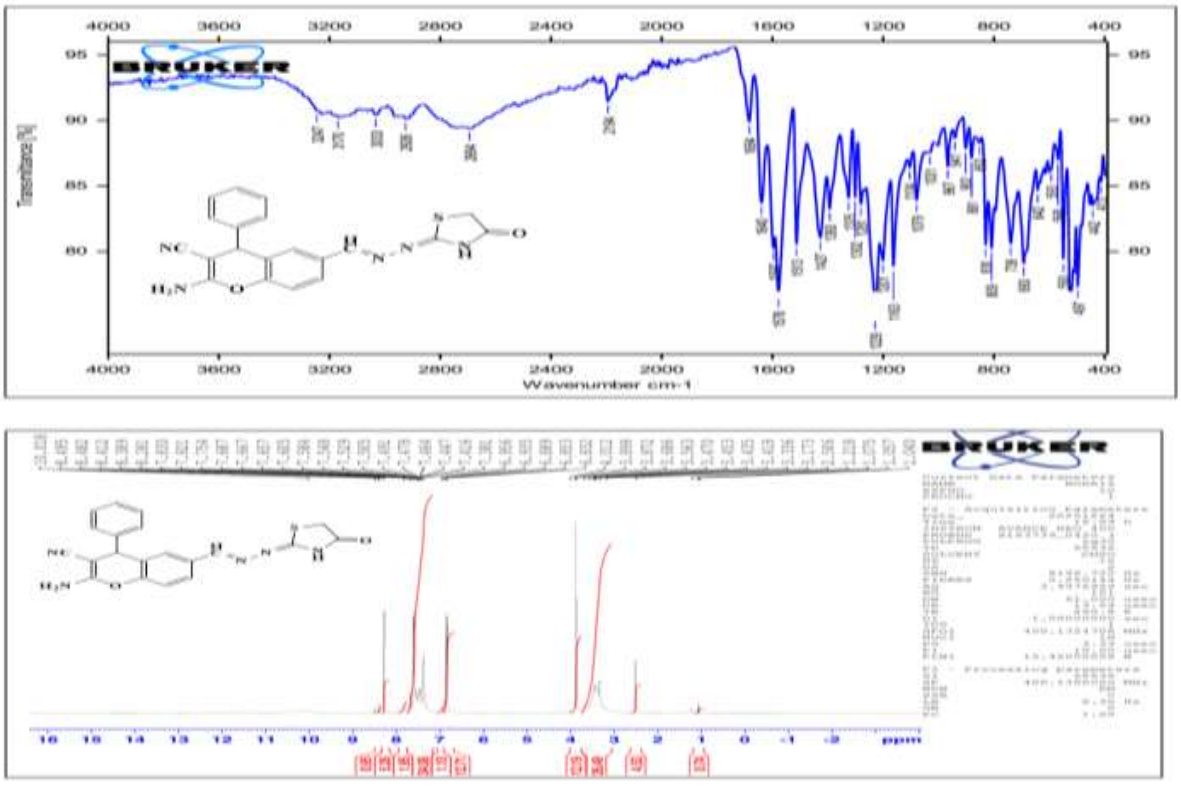
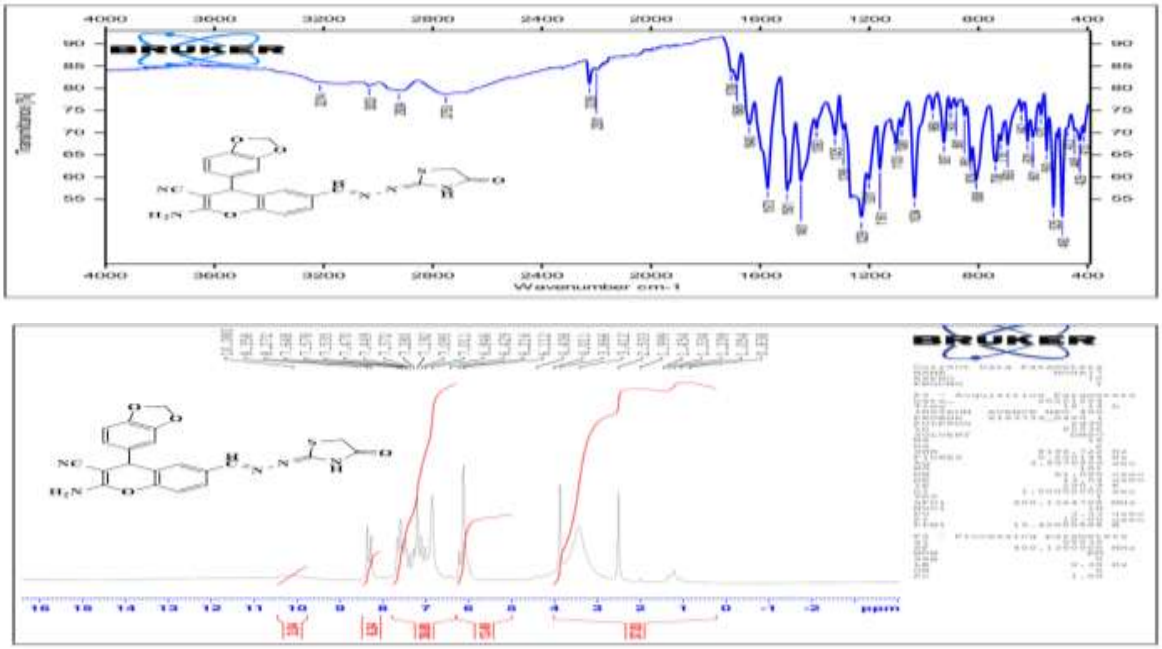


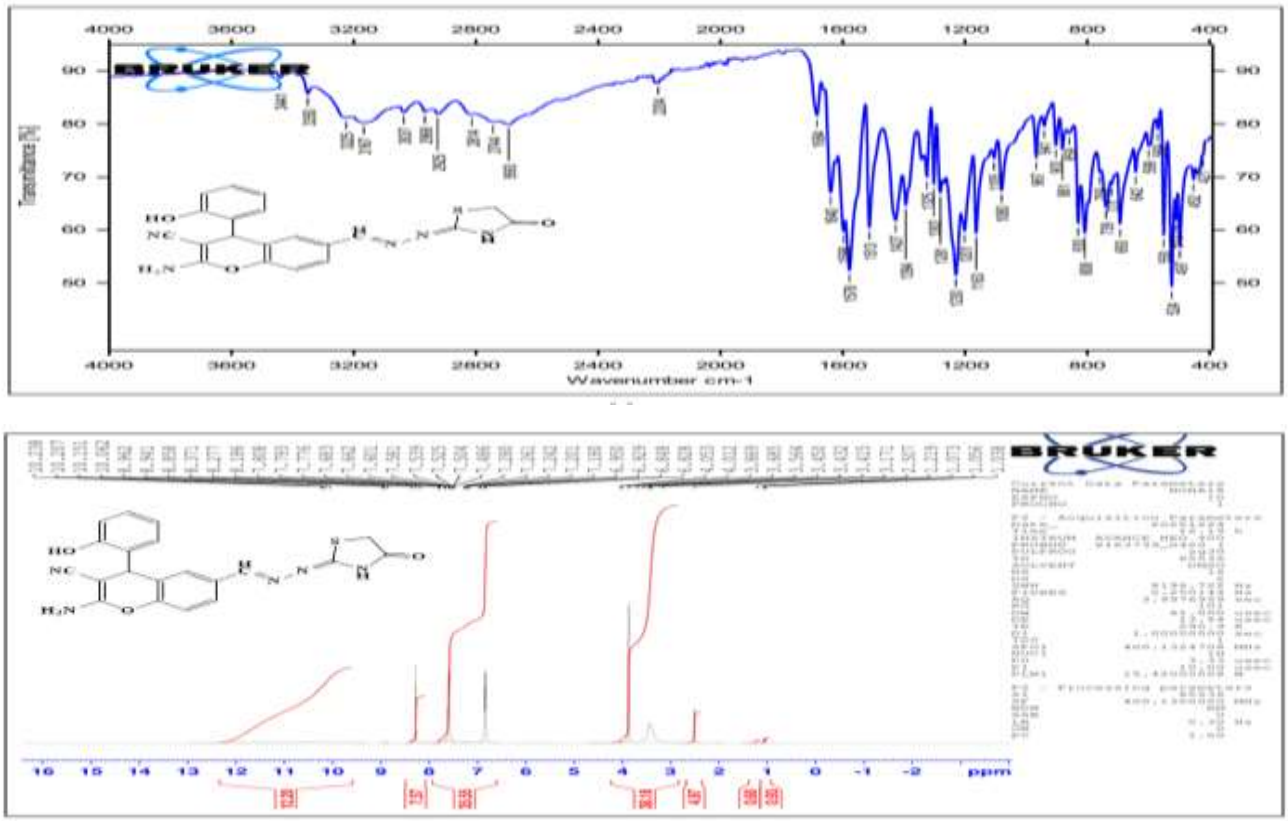
H_4



H_5



H_6



H_7

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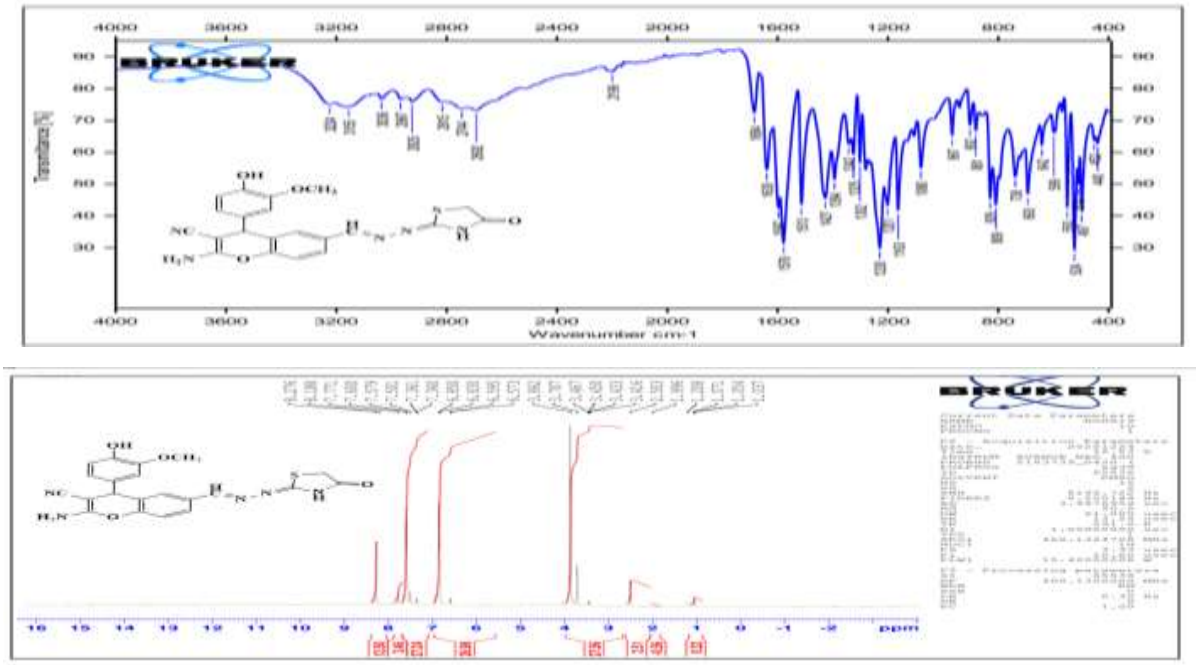

H_8

4. CONCLUSION

The current study have been successfully prepared and characterized a new series of heterocyclic compounds. These synthezide pathways have demonstrated good efficiency through the use of one-pot multicomponent reactions, grinding and microwave irradiation. These approaches contributed to reducing the reaction times and green eco-friendly reaction conditions compared with conventional methods. This study provides potential for further biological and pharmaceutical investigations on the prepared compounds.

5. ACKNOWLEDGEMENT

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