Multicomponent Synthesis and Investigations Fluorescence Activity of Chromenone–Pyrazole Compounds

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Abstract
A synthetic method is described to produce some chromenone-pyrazole derivatives through a one-pot multicomponent reaction using SrFe12O19 as a magnetic catalyst. This method provides quite a few merits, including the use of an effective and easy separable nanocatalyst, high yields of products, short reaction time, and easy work-up. Two of the products showed fluorescence properties, which have detected mercury ions without any interference with other ions. They can detect a tiny amount of mercury ions, which were comparable with other chemosensors. The detection limit is $4 \times 10^{-7}$ and $3 \times 10^{-8}$ M, respectively, for the compound I and II, respectively, which were considered very low amounts. The effect of mercury on health and environmental pollution is essential in medical science.

Keywords 4-Hydroxy-6-methyl-2H-pyran-2-one • Multiple-component reactions • Hydrazine derivative • Salicylaldehyde • Chromenone–Pyrazole compounds • Fluorescence compounds • Hg$^{2+}$ Chemosensor

Introduction
Coumarin is a familiar scaffold found in many natural products isolated from a wide range of species, especially plants. Dicoumarol is an anticoagulant agent that was discovered by Professor Link in 1933. This compound is a metabolized product of coumarin in the infected sweet clover (Melilotus) by some molds such as Penicillium nigricans. Warfarin is another important and famous coumarin derivative, which is used as an oral anticoagulant agent [1]. In addition to the pharmaceutical application of coumarin derivatives, some of them are beneficial sensors, such as 3-aceetoacetylcoumarin (Fig. 1), which was used as a probe to find intracellular hydrazine in glioma cell lines because of the formation of a fluorescent coumarin-pyrazole product [2].

In recent years, a combination structure of coumarin and pyrazole has become an attractive scaffold in medicinal chemistry. Saeed et al. designed coumarinyl pyrazolinyl thioamide derivatives and determined their potential antioxidant activity against the urease enzyme [3].

Among the various pyrazoles prepared by Wardakhan and Louca, coumarin-pyrazole compounds showed the highest activity against Candida albicans [4]. Moreover, the antibacterial activity of quinolinepyrazoline-based coumarinyl thiazole derivatives has been assigned [5]. Al-Ayed developed the synthesis of antioxidant coumarin containing a 4-arylbut-3-en-2-one moiety through the condensation of 3-acetylcoumarin with aryl aldehydes [6].

Mercury pollution is one of the most challenging problems since its accumulation in the human body can lead to various neurological damages [7, 8]. Some industrial activities, including fossil-fuel combustion, [9] gold mining, [10] solid-waste burning, [11] and natural sources, such as forest fires and volcanic emissions [12] lead to rapid enhancement of Hg$^{2+}$ levels in the environment. Additionally, ionic mercury can be converted to methylmercury naturally, which is more toxic than the mercury element and its salts [13]. When methylmercury enters the environment, it results in bioaccumulation and high concentrations among populations of some species through the food chain [14]. The Food and Drug Administration (FDA) and the United States Environmental Protection Agency (US EPA) advise the maximum allowable concentration for methylmercury and mercury salts as 1 ppm (1 mg/L) [15] and 2 ppb (0.002 mg/L), [16]
respectively. Hence, an appropriate and fast technique is essential to detect Hg$^{2+}$. In recent years, extensive studies have been performed to develop sensitive and straightforward methods, such as resonating microcantilevers, [17] voltammetry, [18] colorimetric, and fluorescence spectroscopy [19] for quick recognition of mercury ions.

In continuation of our research, [20–23] herein a synthetic method for the synthesis of chromenone-pyrazole derivatives through a one-pot multicomponent reaction using SrFe$_{12}$O$_{19}$ [24, 25] as a magnetic catalyst is described, and then the fluorescence properties of chromenone-pyrazole compounds against metal cations were investigated.

### Experimental Section

#### General Procedure for the Preparation of the SrFe$_{12}$O$_{19}$ Catalyst

SrFe$_{12}$O$_{19}$ magnetic nanoparticles (MNPs) were prepared by employing a simple sol–gel auto-combustion method, as reported before in literature [26, 27].

#### General Procedure for the Synthesis of Coumarin-Pyrazole Derivatives (4a–g)

A combination of salicylaldehyde derivative 1 (1 mmol) and 4-hydroxy-6-methyl-2$H$-pyran-2-one 2 (1 mmol, 0.13 g), and SrFe$_{12}$O$_{19}$ (0.02 g) was heated at the temperature of 120 °C about approximately 15 min. Then, hydrazine (1 mmol) was added to the reaction mixture, which was stirred under the same conditions within (3–7 min) until reaction completion (traced by TLC method). Then, the raw product was dissolved in EtOAc, and the magnetic catalyst was separated using an external magnet. By evaporating the solvent, the product was precipitated out, and then crystals were filtered off, washed well with $n$-hexane, and then with water, respectively.

3-(3-Methyl-1H-pyrazol-5-yl)-2H-chromen-2-one (4d): Yield: 96%, yellow powder, mp: 182–184 °C, FT-IR (KBr) $\nu_{\text{max}}$: 3427 (NH), 3037, 3091 (CH stretch), 1720 (-O-C=O), 1605 (C=O), 1576, 1474 (C=C), 1382 (CH$_3$) cm$^{-1}$, $^1$H NMR (500 MHz, DMSO-d$_6$): 2.3 (s, 3H, CH$_3$), 6.7 (s, 1H, Pyrazole ring C-H), 7.3 (dd, 1H, Ar–H), 7.4 (d, 1H, Ar–H), 7.6 (t, 1H, Ar–H), 7.8 (s, 1H, Ar–H), 8.5 (s, 1H, Ar–H), 12.8 (s, 1H, NH) ppm. $^{13}$C NMR (125 MHz, DMSO-d$_6$): 10.9, 105, 115.83, 119.2, 124.5, 128.5, 131.3, 136.59, 152.63, 158.8 ppm.

5-Hydroxy-3-(3-methyl-1-phenyl-1H-pyrazol-5-yl)-2H-chromen-2-one (4f): Yield: 96%, white powder, mp: 280–281 °C, FT-IR (KBr) $\nu_{\text{max}}$: 3415 (OH), 33070, 2923 (CH), 1684 (C=O), 1605, 1585, 1534 (C=C), 1374 (CH$_3$) cm$^{-1}$, $^1$H NMR (500 MHz, DMSO-d$_6$): 2.3 (s, 3H, CH$_3$), 6.5 (s, 1H, Pyrazole ring C-H), 7.1 (d, 1H, Ar–H), 7.1 (d, 1H, Ar–H), 7.2 (t, 1H, Ar–H), 7.3 (m, 1H, Ar–H), 7.4 (m, 4H, Ar–H), 10.2 (s, 1H, Ar–H) ppm. $^{13}$C NMR (125 MHz, DMSO-d$_6$): 13.2 (CH$_3$), 109.9, 118.2, 118.8, 118.8, 119.5, 123.3, 124.8, 127.0, 129.1, 136.8, 140.3, 141.9, 144.0, 144.4, 148.3, 157.9 ppm.

### Table 1 Optimizing the reaction condition for the synthesis of chromenone-pyrazole

| no | Catalyst | Solvent | Condition | Time (h) | Yield (%) |
|----|----------|---------|-----------|----------|-----------|
| 1  | SiO$_2$-Pr-SO$_3$H | - | 120 °C | >4 | - |
| 2  | SrFe$_{12}$O$_{19}$ | EtOH: H$_2$O (1:1) | Reflux | 2.5 | 67 |
| 3  | SrFe$_{12}$O$_{19}$ | EtOH | Reflux | 3 min | 52 |
| 4  | SrFe$_{12}$O$_{19}$ | H$_2$O | Reflux | 1.5 | 75 |
| 5  | SrFe$_{12}$O$_{19}$ | - | 120 °C | 22 min | 95 |
6-Bromo-3-(3-methyl-1H-pyrazol-5-yl)-2H-chromen-2-one (4g)

Yield: 89%, yellow powder, mp: 241–239 °C, FT-IR (KBr) \( \nu_{\text{max}}: 3442 (\text{NH}), 3106, 2957 (\text{CH}), 1721 (\text{C=O}), 1598 (\text{C=N}), 1566, 1474, 1436 (\text{C=C}) \text{ cm}^{-1}. \) \( ^1\text{H NMR (125 MHz, DMSO-} \text{d}^6): 2.3 (s, 3H, \text{CH}_3), 6.7 (s, 1H, Pyrazole ring C-H), 7.4 (d, 1H, Ar–H), 7.7 (d, 1H, Ar–H), 8.1 (s, 1H, Ar–CH), 8.5 (s, 1H, Ar–H) \text{ ppm.} \) \( ^{13}\text{C NMR (125 MHz, DMSO-} \text{d}^6): 9.9, 104.7, 115.8, 117.7, 120.9, 121.2, 133.1, 134.7, 138.8, 143.8, 151.3, 158.1 \text{ ppm.} \)

Results and Discussion

Chromenone-pyrazole can be synthesized via a three-component reaction of salicylaldehydes 1, 4-hydroxy-6-methyl-2H-pyron-2-one 2, and phenylhydrazine 3. Initially, the reaction conditions were optimized, and the results were shown in Table 1. Among the employed catalysts of SiO\(_2\)-Pr-SO\(_3\)H and SrFe\(_{12}\)O\(_{19}\), in the case of the last one, the reaction time significantly reduced and gave the highest yield of product. This reaction was then investigated in the presence of SrFe\(_{12}\)O\(_{19}\) magnetic catalyst in different solvents, including EtOH, H\(_2\)O, and a mixture of H\(_2\)O and EtOH, besides under solvent-free conditions. By comparing the obtained results, it was found that solvent-free condition at 120 °C is the best condition in terms of reaction efficiency and time.

Afterward, this reaction was generalized with several salicylaldehydes and hydrazine compounds under the optimized conditions (Scheme 1), and the derivatives have been classified in Table 2. After completing the reaction (monitored by TLC), the raw product was dissolved in boiling EtOAc; furthermore, the catalyst was simply separated from the solution using a magnet, and the product was obtained via gradual solvent evaporation. The new products were characterized by melting point, FT-IR, GC–MS, and NMR spectral data. Melting points of synthesized derivatives were compared with data reported in the literature, as illustrated in Table 2.

The proposed mechanism for this reaction was presented in Scheme 2. At first, enol carbon of 4-hydroxy-6-methyl-2H-pyron-2-one 2 was added to the activated carbonyl group of salicylaldehyde 1 through the Knoevenagel condensation, and then the dehydration process gave the adduct product 6, which is then cyclized intramolecularly to gain intermediate 7. The dehydration process of compound 7 leads to the ring-opening of the pyran moiety to

Table 2 Synthesis of chromenone-pyrazoles derivatives 4a-4g in the presence of nanomagnetic SrFe\(_{12}\)O\(_{19}\) in solvent-free condition

| Entry | No. | R\(^1\) | R\(^2\) | R\(^3\) | Time (min) | Yield (%) | m.p. (°C) | m.p. [Lit.] (°C) |
|-------|-----|---------|---------|---------|------------|-----------|-----------|-----------------|
| 1     | 4a  | NO\(_2\) | H       | Ph      | 22         | 88        | 168-170   | [28]            |
| 2     | 4b  | Br      | H       | Ph      | 20         | 89        | 160-158   | [29]            |
| 3     | 4c  | H       | H       | Ph      | 20         | 89        | 125-127   | [29]            |
| 4     | 4d  | H       | H       | H       | 18         | 96        | 182-184   | New             |
| 5     | 4e  | H       | H       | 4-NO\(_2\)-Ph | 20   | 87        | 270-272   | New             |
| 6     | 4f  | H       | OH      | Ph      | 20         | 96        | 281-280   | New             |
| 7     | 4g  | Br      | H       | H       | 18         | 89        | 241-239   | New             |
give chromene 8, which was reacted hydrazine compound 3, followed by dehydration, cyclization, and another dehydration process to yield the final product 4.

In comparison with the published methods in literature as shown in Table 3, the present methodology has several advantages such as the use of inexpensive magnetic catalyst, simple procedure, the short reaction time, and the high purity of products. The nanomagnetic catalyst can be easily separated from the reaction mixture using an external magnet.

### Investigation of Fluorescence Properties and Sensitivity of Chromenone-pyrazole Compounds (L₁, L₂) Against Metal Cations in Absolute EtOH

The fluorescence responses of chromenone-pyrazole derivatives were examined toward metal cations using fluorescence spectroscopy. As shown in Fig. 2, the fluorescence spectra of ethanolic solution of all derivatives (1 × 10⁻⁵ M) were recorded, and only 4d and 4g showed the fluorescence emission with λₑₓ = 348 nm and λₑₓ = 300 nm, respectively.

### Table 3 Comparison of different reported conditions in the literature for the synthesis of chromenone-pyrazoles derivatives 4a-4c

| Entry | Catalyst   | Solvent          | Condition     | Time (h) | Yield (%) | Ref |
|-------|------------|------------------|---------------|----------|-----------|-----|
| 1     | Piperidine | CH₃CN            | Reflux        | 3        | 60        | [28]|
| 2     | Meglumine  | EtOH:HO (1:1)    | Reflux        | 1.5      | 75        | [29]|
presence of the aryl group on the nitrogen atom of the pyrazole
group leads to the lack of fluorescence emission because the
non-bonding electrons of the nitrogen atom participate in the
resonance of the phenyl ring. Thus, the resonance energy of
the pyrazole ring is decreased. Therefore, participating in the
non-bonding electrons of the nitrogen atom in the pyrazole
ring’s resonance is essential for the product’s fluorescence
properties.

Investigation of Fluorescence Properties and Sensitivity
of Chromenone-pyrazole (L1) Compounds Against Metal
Cations in Absolute EtOH. To investigate the interaction of
various cations with compound L1, Fe2+, Fe3+, Mn2+, Mg2+,
Al3+, Zn2+, Cr3+, Pb2+, Co2+, Hg2+, K+, Ca2+, Cu2+, Cd2+
and Ag+ cations (100 μL, 0.01 M) were separately added to
the ethanolic solution of L1 (3 mL, 10–5 M) and the changes
were indicated at the emission wavelength of 418 nm. The
intensity of emission peak was decreased significantly only
in the presence of Hg2+, as shown in Fig. 3.

To explore the selectivity of the L1 against mercury ions,
competitive experiments were performed against other metal
cations (Fe2+, Fe3+, Ca2+, Cd2+, Mn2+, Co2+, Pb2+, Cu2+,
Ag+, Mg2+, Ni2+, Cr3+, Zn2+, Na+, Al3+ and K+). As shown
in Fig. 4, no significant interference is observed, and there-
fore, it can be claimed that compound L1 is a selective fluo-
rescence chemosensor for the discernment of mercury ions
even in the presence of other metal cations.

Titration experiment was accomplished in absolute EtOH
solution of L1 with different amounts of Hg2+. As shown in
Fig. 5, the emission of chemosensor L1 regularly decreases
by increasing Hg2+ concentration. The Fig. 5 inset shows
good linear relations between the emission at 418 nm and
the concentration of Hg2+ with the linear equation of emission
= -218.96 [Hg2+] + 609.12 in the equivalent range
and R² = 0.987. The detection limit for chemosensor L1 to
detect mercury has been found from the following equation:
DL = (kS_d)/m, where k is a constant factor equal to 3, S_d is
the standard deviation obtained from the determination of
the sample emission intensity at 412 nm for 6 replicates, and
m is the slope of emission intensity against Hg2+ concentra-
tion. So the detection limit of L1 for Hg2+ was calculated as
4 × 10–7 M.

Investigation of Fluorescence Properties and Sensitivity
of Chromenone-pyrazole (L2) Compounds Against Metal Cations
in Absolute EtOH. Another derivative of 6-boromo-chromenone-
pyrazole (L2), also represents fluorescence properties. Thus,
the effect of cation on the fluorescence properties of L2 was
also investigated. The ligand’s interaction was selective toward
mercury ions because a sharp decrease is observed after adding
a certain aliquot of Hg2+ solution (Fig. 6). Therefore, the effect
of interfering ions was then studied to explore the selectivity
of L2 for the detection of mercury ions. Fortunately, any of the
cations did not interfere with L2-Hg2+ (Fig. 7).

The titration experiment for L2 was accomplished
as the same L1 (Fig. 8). The emission of chemosensor
L2 again decreases by the gradually increasing amount
of Hg2+. The inset in (Fig. 8) shows a well linear rela-
tion between the emission at 426 nm and the con-
centration of Hg2+ with the linear equation of emission
= -275.01 [Hg2+] + 346.23 and R² = 0.9899. The
detection limit was also calculated like the method men-
tioned above and was L2 3 × 10–8 M.

Study of pH Effect in H2O

The efficiency of acridine as a fluorescent sensor for Hg2+
was evaluated in different pH. The effect of pH in the range
2 to 8 on the fluorescence intensity of the acridine in H2O

![Fig. 2 Fluorescence emission of chromenone-pyrazole 4d (L1) and 4 g (L2)](image_url)
Fig. 3 Fluorescence emission for a new compound of $L_1$ in the presence of 100 µL (0.01 M) of metal cations (Ca$^{2+}$, Cd$^{2+}$, Co$^{2+}$, Pb$^{2+}$, Cu$^{2+}$, Ag$^+$, Mg$^{2+}$, Ni$^{2+}$, Hg$^{2+}$, Zn$^{2+}$, Al$^{3+}$, K$^+$, Hg$^{2+}$) in absolute ethanol ($\lambda_{ex}$ = 348 nm and $\lambda_{em}$ = 418 nm).

Fig. 4 Selectivity of chemosensor $L_1$ for Hg$^{2+}$ against other metal cations in an ethanolic solution ($\lambda_{ex}$ = 348 nm and $\lambda_{em}$ = 418 nm).

Fig. 5 Fluorescence emission of chemosensor $L_1$ over addition of Hg$^{2+}$ in EtOH absolute solution; inset: plot of fluorescence emission as a function of Hg$^{2+}$ concentration ($\lambda_{ex}$ = 348 nm and $\lambda_{em}$ = 418 nm).
was studied. The pH of the solution was set by NaOH and HCl solutions, and the recorded fluorescence intensities are shown in Fig. 9. It should be mentioned which the fluorescence intensity of the ligand was not changed with and without Hg$^{2+}$ in the pH range. Therefore, the chemosensor was not affected by pH in detection of Hg$^{2+}$.

**Investigation of Fluorescence Properties and Sensitivity of Chromenone-pyrazole (L$_2$) Compounds Against Metal Cations in Absolute EtOH/H$_2$O**

One of the divertive (4 g) didn’t solve in water, so we use H$_2$O:EtOH ratio (1:1) as solvent for recording of the
fluorescence spectrum of this derivative (1 × 10⁻⁵ M). In order to investigate, the interaction of various cations with compound L₂ (4 g), Fe²⁺, Fe³⁺, Mn²⁺, Mg²⁺, Al³⁺, Zn²⁺, Cr³⁺, Pb²⁺, Co²⁺, Hg²⁺, K⁺, Ca²⁺, Cu²⁺, Cd²⁺ and Ag⁺ cations (100 μL, 0.01 M) were studied. As depicted in Figs. 10, 11, and 12, the intensity of emission peak was decreased significantly only in the presence of Hg²⁺ like ethanolic solution.

The emission of chemosensor L₂ again decreases against gradually increase of Hg²⁺ concentration.

**Comparison of Reported Chemosensors**

The presented compounds have shown very distinct advantages over the previously reported compounds in the detection and measurement of mercury ions. The analytical performance of mercury ions’ current chemosensor and some of the significant reported methods are summarized in Table 4. One of the essential factors in the synthesis of new compounds 1, 2, and 3 is using a green solvent. In this regard, EtOH is an undoubtedly better solvent than CH₃CN.
Fig. 10 Fluorescence emission of chemosensor L₂ in the presence of different metal cations (Fe³⁺, Fe²⁺, Cd²⁺, Co²⁺, Cr³⁺, Cu²⁺, Ag⁺, K⁺, Al³⁺, Zn²⁺, Pb²⁺, Mg²⁺, Hg²⁺, Ca²⁺, Ni²⁺ in H₂O:ETOH (1:1), λ ex = 300 and λ em = 418

![Fluorescence emission of chemosensor L₂](image)

Fig. 11 Selectivity of chemosensor 4g (3 mL) for Hg²⁺ (100µL, 1×10⁻³ M) in the presence metal cations in H₂O:EtOH (1:1) solution, (λ ex = 300, λ em = 418)

![Selectivity of chemosensor 4g](image)

Table 4 Comparison of the analytical performance of compounds 6 and 7 with some previously reported chemosensors

| NO | Chemosensor | Solvent | The detection limit (M) | Ref |
|----|-------------|---------|------------------------|-----|
| 1  | CH₃CN       |         | 8×10⁻⁶                 | [30]|
| 2  | CH₃CN       |         | 2.5×10⁻⁶               | [31]|
| 3  | MeCN:H₂O (4:1) |         | 4×10⁻⁶                 | [32]|
| 4  | CH₃CN (7:3) |         | 4×10⁻⁶                 | [33]|
| 5  | DMF/H₂O (95:5) |         | 5.56×10⁻⁷          | [34]|
| 6  | EtOH        |         | 4×10⁻⁷                 | This work|
| 7  | EtOH        |         | 3×10⁻⁸                 | This work|

Compound 3 showed an excellent detection limit, but the synthesis method was complicated. In the case of compound 5, while the applied solvent DMF is poisonous, the obtained detection limit is remarkably lower than the reported one. For compounds 6 and 7 as novel fluorescence sensors with excellent detection limit, a simple, green, and short time synthesis method was reported.
Fig. 12  Fluorescence emission of chemosensor L2 upon addition of Hg2+ in H2O:EtOH (1:1) solution; (λex = 300 nm and λ em = 418 nm)

Conclusions

In summary, the chromenone-pyrazoles derivatives were synthesized by the multicomponent reaction approach in solvent-free conditions using a nanomagnetic catalyst. Among the seven derivatives, only two compounds have had fluorescence properties and responded to the Hg2+ ion with the low detection limit. The two fluorescent compounds in the pyrazole group have not aromatic groups. Then, N group is the main factor in generating the fluorescence property of these compounds.

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Declarations

Ethics Approval  Not applicable.

Consent to Participate  Not applicable.

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