Nano-Synthesis, characterization and spectroscopic Studies of chromium (III) complex derived from new quinoline-2-one for solar cell fabrication

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Abstract. In this work, Schiff base ligand (L) has been synthesized by condensation reaction of N-amino quinoline-2-one with 4-chlorobenzaldehyde in ethanol, for 5 hours. The synthesized ligand was characterized using (¹³C,¹H NMR), (U.V-Vis), X-ray diffraction (XRD), (FT-IR), (C.H.N) elemental analysis, atomic force microscope (AFM) and melting point. The chromium complex was obtained by (2:1) (L: M) molar ratio and then characterized by FT-IR, UV-Vis, molar conductivity, magnetic susceptibility measurements, AFM, XRD and flame atomic absorption technique (FAA). The results confirmed an octahedral geometry of chrome ion (III). Drop casting techniques was used to prepare nano-thin films of the synthesized compounds. The aim of this study was to fabricate solar cells using the prepared nano- thin films. To achieve, the morphological, structural and optical properties of the nano-thin films were studied, and then they were precipitated on the silicon slides. The fabricated solar cells showed a high efficiency promising to be used for improving silicon solar cells.

Keywords: Quinolone, Nano Scale, Chromium (III) complex, silicon Solar cell

1. Introduction

Nanochemistry is one of the modern sciences, which has taken a wide field in many agricultural, industrial and medical applications, as well as energy and pollution fields [1-6]. Renewable energy - green chemistry is still one of the major environmental and economic solutions for increasing rapid pollution, generated by fossil fuels and other traditional sources of energy [7-10]. The natural and available sun light is widely used for saving energy in green chemistry reactions process [11]. The solar energy is still the richest, most incomplete and cleanest of all renewable energies. This energy used to generate the solar cells, also known as photovoltaics, which are photovoltaic devices that convert the energy from the sun to electricity. Electron-hole pairs are created by the energy incidents of the photovoltaic material to overcome the energy band gap of the photovoltaic equipment to allow the current flow generally p-n (semiconductor) junction, due to the special composition of solar cells that then generates current free electrons travel in a single direction [11,12]. Schiff base compounds have been used to improve the efficiency of the solar cell by generating a photocurrent that can be used as an electron accepter [13]. For this regard, coumarin is the basic part that can be used as a starting material for improving the photoelectric activity via quinolone compounds [14-16]. Quinolone compounds have good optical characteristics and the potential to be applied to laser instruments, fluorescent samples and light-emitting diodes [17]. Therefore, several quinolone complexes can be used to generate solar cells with visible absorption, low cost and non-toxic [18]. The aim of this study including to synthesize of chromium (III) complex derived from new Quinoline-2- one and test its effectiveness to increase the efficiency of the solar cell.
2. Experimental part

2.1. Chemical Materials and Methods

Flukes and Mark supplied all chemical materials. Melting points were determined Using SMP30 melting point system. carbon-13 and proton NMR spectra were collected by the University of Al-Albayt, Amman, Jordan, with BrukerDMX-500 (300 M Hz). Infrared spectra collected using (KBr) on Shimadzu, (4800S) (FTIR) spectrophotometer in a range between 4000 to 400 cm^{-1}. The Electronic spectrophotometer (U.V-Vis) range between 200 to 900 nm were used to obtain the electronic spectra. Complex magnet sensitivity was measured using magnetic sensitivity balance, Model (MSB MK1). Model (MSB MK1). The C.H.N. Spr Analyses prepares by EURO company [ EA (EA3000)] elemental analyser was conduc
ted with elemental ligand microscope research. A phoenix-986 AA spectrophotometer was used for the metal content of the complex using atomic absorption (A.A) technology. The molar conductivity of the complex was calculated by the Inolap Multi 740, WTW 8236 Weilhiem (WTW) for (10-3 M) complex solution within DMSO. X-ray diffraction (XRD-6000, Shimadzu). Microscope optical (Leice dm 2500p). Atomic force microscope known (AA 3000 scanning sample microscope) was used to detect the topography of the nano-films.

2.2. Synthesis of starting material

The starting compound [N-amino quinoline-2-one (S)] was synthesized according to process in Scheme.1. A weight of 0.05 mole of coumarin (sigma Aldrich) and 0.5 mole of hydrazine hydrate 99% (sigma Aldrich) was dissolved in 30 ml absolute ethanol and mixed well. The mixture was heated and refluxing for 12 hours. After that, a light-yellow precipitate was collected, washed using absolute ethanol and dried. The precipitate was recrystallized using ethanol to produce a clear sample [19], the yield percent of product was 92%, the melting point ranged between 131 to 133 °C, the Molecular weight (160 g \text{ mole}) and the chemical formula was (C_{9}H_{8}N_{2}O). Table.1 showed the theoretical (percent) analysis of C (67.5), N(17.5), H (5); experimental (percent) C(67.37) ; N(17.41) ; H(5.28).

2.3. Synthesis of Schiff base

The Schiff base ligand (L) was synthesized via condensation reaction (Scheme1) of N-amino quinoline-2-one (1.6 g, 0.01mole) and 4-chlorobenzaldehyde (1.41 g, 0.01mole) in absolute ethanol 25 ml. Drops of glacial acetic acid were added as a catalyst to adjust the pH to 6, then the mix solution was left under refluxing for 7 hours. The yellow precipitate was then washed several times using absolute ethanol and dried. The obtained precipitate was recrystallized using ethanol to produce a clear sample. The yield percent of product was 85%, the melting point ranged between 219 to 221°C, molecular weight (272.5 g \text{ mole}) and the chemical formula was(C_{16}H_{11}ON_{2}Cl). Elemental analysis, theoretical (%) C (70.45), H (4.03), N (10.27); experimental (%) C (69.08), H (3.96), N (10.35) as shown in Table.1.

![Scheme 1](image-url)

**Scheme.1.** Synthesis of ligand (L) (1-((4-chlorobenzylidene)amino)quinolin-2(1H)-one.

2.4. Synthesis of chromium complex

Chromium complex was synthesized by mixing ethanolic solution of ligand (L_{1}) (0.545 g, 2 mmole) with (0.266 g, 1mmol) of CrCl_{3}.6H_{2}O and dissolved in ethanol for a final molar ratio 2:1 (L_{1}:M). The mixture was stirred and refluxed for 6 hours. The green solid precipitate was filtered and washed with excess of ethanol. The crude product was recrystallized in tetrahydrofuran (THF) to get a clear sample.
of a yield 78%, melting point (299-302 °C) and a molecular weight (703.5 g/mol). The physical properties of the starting materials and the synthesized compounds are shown in Table 1.

### Table 1. The physical properties of the synthesized compounds (S, L & M).

| Symbol | General formula | Molecular weight (g/mole) | M. P C° | Yield % | Color     | Theoretical (exp.) |
|--------|-----------------|---------------------------|---------|---------|-----------|-------------------|
|        |                 |                           |         |         |           | C%        | H%        | N%        | M%        |
| S      | C₇H₈N₂O        | 100                       | 131–133 | 92      | Light yellow | 67.5      | 5         | 17.5      | -         |
|        |                 |                           |         |         |           | (67.37)   | (5.28)    | (17.41)   | -         |
| L      | C₁₆H₁₈N₆OCl    | 272.5                     | 219-221 | 85      | Yellow    | 70.45     | 4.03      | 10.27     | -         |
|        |                 |                           |         |         |           | (69.08)   | (3.96)    | (10.35)   | -         |
| M      | [Cr(L₂)Cl₂]Cl  | 703.5                     | 299-302 | 78      | Green     | 54.58     | 3.12      | 7.96      | 7.39      |
|        |                 |                           |         |         |           | (54.18)   | (3.01)    | (7.81)    | (7.27)    |

### 2.5 Thin Film Deposition [20]

Drop casting method was used to deposit (L, M) thin films on glass and silicon substrates, (Figure 1). This method is regarded the best way used to get various thin films, with high productivity from a simple apparatus and no waste or another of material.

![Figure 1. A diagram of the drop casting method.](image)

### 2.6 Preparation of L and M /PSi/Si Hetro junction Solar Cell

The colloidal L and M Nano films were prepared via drop casting technique on Si layer. Nano scale have been taken from solution using a pipette and a volume of 5 drops were dropped onto the surface and left it for (5-6 hour) at room temperature. After drying the film is ready [9].

### 3. Results and Discussion

**HNMR spectrum:** 1HNMR spectrum was consistent with a number of types of the present protons. A single signal at μ (9.5)ppm consistent with azomethine was defined by the formation of the Schiff base (—N= CH–). Figure 1.2 showed several signals were allocated to aromatic protons in the range (6.5-8.5) ppm[21]
Figure 2. $^1$HNMR spectrum of ligand (L)

carbon-13 NMR spectrum: The $^{13}$C-NMR spectrum of ligand (L) is shown in Figure (3). The bands of carbon at (118-147, 156 and 173) ppm were attributed to the Aromatic Carbone atoms, (HC=N-) and (C=O), respectively.

Figure 3. $^{13}$CNMR spectrum of ligand (L)

The FTIR spectrum: The FTIR spectrum of 4-nitrobenzaldehyde showed a strong vibration band at (1703) cm$^{-1}$ referred to the frequency of (C=O) group stretch. The starting materiel compound [N-amino quinoline-2-one (S)] showed two vibration bands at (3298) and (3286) cm$^{-1}$ due to the vibration of the primary amine (NH$_2$), asymmetrical and symmetrical stretching. The three strong vibration bands appeared at (1639) cm$^{-1}$ referred to the extended carbonyl amide group vibration, vibration assigned at (1593,1450) and (3051) cm$^{-1}$ referred to the stretching vibration bands of $v$(C=C) and $v$(C-H) aromatic, respectively. The ligand (L) showed new bands at (1612) cm$^{-1}$ referred to stretch mode for the azomethine group $v$(HC=N-) [23,24] with no vibration band of the primary amine in the starting materiel compound (S). In addition, a vibration at (1662) cm$^{-1}$ referred to the carbonyl amid group. The bands of $v$(C-H) aliphatic, $v$(C-H) and $v$(C=C) aromatic at (2953) cm$^{-1}$, (3069) cm$^{-1}$ and (1577) cm$^{-1}$ were allocated to (2953) cm$^{-1}$, (3069) cm$^{-1}$ and (1577) cm$^{-1}$, respectively. (Figure 4) [25,26]. On complexing, the ligand spectrum was changed to a higher frequency of (1639) cm$^{-1}$, suggested the assistance of azomethine-Nitrogen groups in coordination with a chromium ion, which appeared at
(1612) cm\(^{-1}\). In complexation, the stretched vibration at (1662 cm\(^{-1}\)) was shifted to a higher frequency (1683) cm\(^{-1}\) for the prepared complex, which indicated a contribution to the coordination cycle by means of the oxygen atom of the carbonyl amide group. Weak bands in with a low frequency at 439, 574 cm\(^{-1}\) can be attributed to the υ(M-O) and υ(M-N) vibrations, respectively [26,27], (Figure 5).

Table 2. The FTIR Spectra of the synthesized compounds (cm\(^{-1}\)).

| Compound | υ(NH) | υ(C=O) | υ(C=N) | υ(C=C) | υ(C-H) | υ(C-H) | υ(NO\(_2\)) | υ (M-N) | υ (M-O) |
|----------|-------|--------|--------|--------|--------|--------|------------|--------|--------|
| C\(_{7}\)H\(_{5}\)NO\(_{3}\) (S) | 3298, 3286 | 1639(s) | - | 1593(m) | 3051 (w) | 2983(w) | - | - | - |
| C\(_{16}\)H\(_{11}\)O\(_{3}\)N\(_{3}\) (L) | - | 1662(s) | 1612(m) | 1577(m) | 3069 (w) | 2953(w) | 1514 | 1340 | - |
| [Cr(L)\(_{2}\)Cl\(_{2}\)]Cl (M) | - | 1683(m) | 1639(m) | 1593(s) | 3097 (w) | 2945(w) | 1516 | 1342 | 574 (w) | 439 (w) |

s: strong, m: medium, w: weak

Figure 4. FTIR spectrum of ligand(L)

Figure.5. FTIR spectrum of Cr (III) complex
The Electronic spectra (Uv. Visible spectrum): The uv. visible spectra (Electronic spectral) of the ligand ($10^{-3}$ M) and its complex were studied by uv. visible device using DMSO as a solvent. The spectrum of the ligand exhibited two high intense bands at 286 nm (34965 cm$^{-1}$) ($\varepsilon_{\text{max}}=1330$ molar$^{-1}$.cm$^{-1}$) and 299 nm (31746 cm$^{-1}$) ($\varepsilon_{\text{max}}=915$ molar$^{-1}$.cm$^{-1}$) due to electronic transitions ($\pi \rightarrow \pi^*$) and ($n \rightarrow \pi^*$) [28], Figure (6). The chromium (III) complex showed three bands due to (d-d) transition at 596 nm (16778 cm$^{-1}$) ($\varepsilon_{\text{max}}= 121$ molar$^{-1}$.cm$^{-1}$) and 422 nm (23696 cm$^{-1}$) ($\varepsilon_{\text{max}}=390$ molar$^{-1}$.cm$^{-1}$) due to $^4A_{2g} \rightarrow ^4T_{1g}^{(F)}$ ($\gamma_2$) and $^4A_{2g}^{(F)} \rightarrow ^4T_{1g}^{(F)}$ ($\gamma_3$) respectively[18,19], (Figure 7).

The electronic transition $^4A_{2g}^{(F)} \rightarrow ^2T_{2g}^{(F)}(\gamma_1)$ was calculated to be 9321 cm$^{-1}$. the B and 10 Dq were (834 and 5125) cm$^{-1}$, respectively. The experimental B value of the complex was 834 cm$^{-1}$ which was less than the theoretical B value of 0.865 and 1030 cm$^{-1}$ of free chromium ion. The electronic spectral bands indicated an octahedral structure around Cr(III) ion [29,30].

![Figure 6. The electronic spectra of ligand (L).](image6)

![Figure 7. The electronic spectrum of Cr(III) complex.](image7)

The Magnetic susceptibility ($\mu_{\text{eff}}$) and conductivity: The magnetic susceptibility moment ($\mu_{\text{eff}}$) of the Cr(III), d$^3$, exhibited normal magnetic moment (3.92 B.M.), in accordance with previous reported data.
The Electrical conductivity of the chrome (III) complex was \((10^{-3} \text{ mole L}^{-1})\) in DMSO solvent, at \((25^\circ C)\) showed an electrolyte behavior of the complex prepared as Molar ratio (1:1). Figure (8) shows that the molar ratio (1:2) \((\text{M: L})\) was confirmed by the study of the transition metal complex of the synthesized ligand.

![The suggested structure of Cr(III) complex.](image)

**Figure 8.** The suggested structure of Cr(III) complex.

**Structural properties:** The X-ray diffraction peaks consist of interference construction monochrome rays reflected from any position of lattice levels at certain angles. The distance interfaces between the levels of the diffraction angle calculated at a certain peak using *(Bragg's law)* [33]:

\[
n \lambda = 2d \sin \theta \tag{1}
\]

Where: \(n\) is an integer number that represents the interference degree \((n=1,2,3,\ldots)\), \(\lambda\) is the wavelength of the X-ray \((1.54\text{Å})\), \(d\) is the spacing between diffracting planes and \(\theta\): is the angle of the incident X-ray. There are many line profile analysis methods to determine the size - strain parameters (microstrains and crystallite sizes). One of them, the single line method which based on a Voigt function. The average crystallite size \((D)\), which can be estimated throughout the Scherrer’s formula: [34-38]:

\[
D = \frac{0.94 \lambda}{\beta \cos \theta} \tag{2}
\]

The micro strains are caused by thin, compressive or extended films to allow a deviation from the ASTM value in the c-leaf constant of the structure. The stress increase therefore occurred because of the different displacements of the atoms in relation to their location as the reference grid. The strain is based on the formula [39]:

\[
\eta = \frac{\beta \cos \theta}{4} \tag{3}
\]

\((\delta)\) can be calculated using the following relation [40]:

\[
\delta = \frac{1}{D^2} \text{ (lines/nm)}^2 \tag{4}
\]

The XRD of prepared ligand \((L)\) and chromium (III) Complex \((M)\) Nano-films by condensation reaction and deposited on glass are shown in figure (9). The XRD patterns of \(L\) include six main peaks, and four main peaks for XRD patterns of \(M\). The crystallite size \((D)\) in nm for a knowing X-ray wavelength \(\lambda\) at a diffraction angle \(\theta\) of \(L\) and \(M\) nano scale films are calculated by using Scherrer
The peak widths of a strong diffraction plane were calculated. The strain ($\eta$) and dislocation density ($\delta$) were calculated for ($\eta$ and $\delta$) of L and M Nano scale Table (3).

**Table 3. Summary of the X-RAY characterization for L and M Nano films**

| Symbol | 2 Theta (deg) | $\beta$ (deg) | D nm | $\eta \times 10^{14}$ lines$^{-2}$m$^{-4}$ | $\delta \times 10^{14}$ lines$^{-2}$m$^{-4}$ |
|--------|---------------|---------------|------|------------------------------------------|------------------------------------------|
| L      | 14.77         | 5.98          | 1.33 | 260.50                                   | 5652.46                                  |
| L      | 22.17         | 4.00          | 2.01 | 172.35                                   | 2474.15                                  |
| L      | 45.14         | 2.00          | 4.26 | 81.20                                    | 549.20                                   |
| M      | 14.73         | 6.00          | 1.32 | 261.33                                   | 5688.45                                  |
| M      | 22.15         | 4.00          | 2.00 | 172.56                                   | 2480.32                                  |
| M      | 29.64         | 3.01          | 2.71 | 127.64                                   | 1357.03                                  |

**Microstructure investigation**

The microstructure of L and M Nano samples prepared by condensation reaction was checked using the optical microscopy. The morphology of L and M Nano-films was recognized by homogeneity and colored film. The images displayed high density of tiny particles spread over the substrate region, roughness and different colors of the surfaces, without crack or void on layer surface.

**Atomic force microscopy (AFM)**

Figure (11) shows the L and M Nano films synthesized by condensation reaction and deposited on glass substrate. These Nano films were rounded spherical shapes which agglomerated particles have larger sizes. The average size was around 94 and 98 nm, respectively. The grains were uniformly distributed throughout the scanning area by 3D images (500 x500 nm) of individual columnar grains extending...
upwards. Both of the determined values of root mean square (RMS) of surface roughness average and average grain size were calculated (Table 4).

Table 4. The grain size, roughness average and Root mean square of L and M Nano films.

| Symbol | Average grain size (nm) | Roughness average (nm) | Root mean square (nm) |
|--------|-------------------------|------------------------|-----------------------|
| L      | 94.17                   | 0.29                   | 0.35                  |
| M      | 97.99                   | 0.31                   | 0.36                  |

Figure 11. The AFM images and Granularity accumulation distribution chart of L and M Nano films.

Optical properties
The transmittance remained constant at a net value for sample film and substrate, so the photo generated carriers produces as a result to passing a large of light through this wide band-gap window and absorbed in silicon. Study of the optical constants of a material has received attention for many reasons, firstly their uses in optical application as interferences filter and reflecting coating needs accurate knowledge for optical constants over a wide range of wavelengths. Figure (12) shows the spectra the L and M Nano-films synthesized by condensation reaction and deposited on a glass substrate. The transmittance of both the L and M increased sharply after~ 400 nm.

Figure 12. The Transmittance spectra for L and M Nano films.
The energy band gap of the L and M thin films determined by plotting of \((\alpha h \nu)\) versus the square of \((\alpha h \nu)\). The straight-line extrapolation gives showed an energy gap of \((\alpha h \nu)^2 = 0\), Figure. (13). The optical band gap values for L and M were 2.6 to 3.2 eV.

![Figure 13. \((\alpha h \nu)^2\) versus photon energy gap of L and M Nano films.](image)

**The Dark Current-Voltage Measurements**

Figure (14) shows the characteristics of I-V dark for reverse and forward direction of Al/L and M/ n-Si/Al solar cell. The forward current for all solar cell was less than 0.9 V, as a recombinant current existed only at low voltages. It is produced when each electron was excited to regain the balance. On the other hand, diffusion or bending region was represented by the second region at a high voltage, which depended on the resistance to serried. In this field, when the tendency voltage provides electron with sufficient energy, the barriers between both sides of the junction can be penetrated.

![Figure 14. I-V in dark characteristics for both reverse and forward.](image)

**I-V Characteristics under illumination**

The optoelectronic characteristics were a major concern for solar cells heterojunction due to its ability to determine the amount of the incident light power that converts to photocurrent. Figure (15) shows the properties of the reversed current-voltage system measuring the photocurrent under 0.1 mW/cm².
light intensity from tungsten lamp. It is worth noting that the reverse current under illumination was higher at a certain voltage for the L-and M-Si hetero junction than it was in darkness. The reverse current value under light intensity increased. The current started at low voltage was indicative of the thermal emission current [41,42].

![Image](https://via.placeholder.com/150)

**Figure.15.** illustrates the I-V characteristic of forward and reverse biasing applied.

Figure (16) shows the characteristics of L / p-Si and M / p-Si heterojunction I-V f. The shorter-circuit current, open –circuit voltage, fill factor (FF) and the Efficiency of solar cell were (19 – 22) mA, (0.41-0.31) V, (67.32-52.43) and (6.39-7.44) %, respectively. Both results confirmed that the L / p-Si and M / p-Si sandwich structures can be used as solar cells.

![Image](https://via.placeholder.com/150)

**Figure.16.** I-V characteristics of solar cell with illumination, for L/p-Si and M/p-Si

| Heterojunction | $V_{oc}$ (Volt) | $I_{sc}$ (mA) | $V_{m}$ (Volt) | $I_{m}$ (mA) | f.f % | $E_{ff}$ % |
|----------------|----------------|--------------|---------------|-------------|------|-----------|
| L/Si           | 0.5            | 19           | 0.41          | 15.6        | 67.32| 6.39      |
| M/Si           | 0.43           | 33           | 0.31          | 24          | 52.43| 7.44      |
4. Conclusion.
The average particle size of the prepared Nano films derived from the organic ligand (L) and its chromium (III) complex was (94 and 98) nm, respectively. The optical properties showed that the direct energy gap of the prepared nanoparticles (L and M) are (2.6 and 3.2eV), respectively. The obtained results may be attributed to the quantum size effect. Data of X-ray diffraction (XRD) confirmed the polycrystalline nano structures of (L and M) Only but no other phases. The accrued efficiency of the fabricated inorganic silicon solar cell (M/Si) was higher than that of the organic solar cell (L/Si).

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