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# Synthesis, Spectroscopic Characterization of Cobalt ComplexfromC$_{16}$H$_{19}$N$_3$O$_3$S and photodegradation using prepared Nano TiO$_2$catalyst

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## Abstract:

Coblatcomplex has been prepared by reaction between $C_{16}H_{19}N_3O_3S$ (L) as ligand and metal salt (II). The prepared complex were characterized by infrared spectra, electromic spectra, magnetic susceptibility, molar conductivity measurement and metal analysis by atomic absorption and (C.H.N) analysis. From these studies tetrahedral geometry structure for the complex was suggested. The photodegredation of complex were study using photoreaction cell and preparednanoTiO$_2$ catalyst in different conditions (concentration, temperatures, pH).The results show that the recation is of a first order with activation energy equal to (6.6512 kJ /mol).

**Keywords:** Amoxicillin derivation, Photodegradation, Photo-catalysis, nanoTiO$_2$**.**

## Introduction:

The knowledge of the interaction between metal ions and antibiotics is of great importance because these reactions can influence the synthesis of metal antibiotics depending on the idea of metal ion interaction with absorbed drugs, for example penicillin in human body can interact with metal ions which are present in a form of free ions or coordinative bounded to proteins, enzymes, amino acid, nucleic bases, nucleotides and other bio ligands(1, 2).

Metals have an esteemed place in medicinal chemistry, most antibiotics do not need metal ions for their biological activities, but there are a number of antibiotics that require metal ions to function properly, such as bleomycin.

streponigrin and bacitracin drugs have gained recognition and are more effective than pure drugs (3),this is due to the fact that metal ions can interact with many different kinds of biomolecules including DNA, RNA, proteins and lipids rendering their unique and specific bioactivities(4, 5). M.A. Hussein, etal(6)(2009) studied Cu(II), Co(II), Ni(II) and Fe(III) chelates of amoxicillin (AMX) antibiotic drug proved that dentate chelate was through –NH$_2$,–that AMX acts with mentioned metal chlorides as a tri –NH, and oxygen of carbonyl β-lactam groups. K.D. Radosavljevic, etal(7), studied TiO$_2$ the application of synthesized by sol-gel method in the photocatalytic degradation of amoxicillin. Effect of the pH value on the photodegradation of AMX was studied at five different pH values. The adjustments of acidic were (pH 3 and 5) and alkaline medium were (pH 9 and 11).The present study was mainly aimed to prepare Co-complex from Amoxicillin derivative and study the photolysis of the prepared complex as a model antibiotic pollutant under UV light illumination and in the presence of TiO$_2$ suspension**.** Another objective was prepare nano TiO$_2$ by Sol-Gel method characterization the TiO$_2$ surface morphologies and Crystallography and examine the effects of catalyst concentration, pH, tempture and light intensity on the photocatalytic degradation of Co-complex. The third objective was to evaluate kineticsparameters of reaction.

## Material and Methods:

The metal salt (CoCl$_2$.6H$_2$O) used in this work were obtained from Fluka, FTIR were of Shimadzu 8400 Fourier transform infrared spectroscopy, which wavelength range of (4000-200) cm$^{-1}$ and UV-Vis 1600A Shimadzu was used to record the electronic spectra at wave length range of (190-1100) nm .The metal analysis was conducted via a Perkin Elmer 500 Atomic Absorption Spectrophotometer. Conductivity Meter 220 with Gall encamp was used to calculate the molar conductivity in ethanol as a solvent at room temperature.M.F.B-6000.01 was used as a melting device. Magnetic susceptibility balance model MSB-MKT was used for magnetic moment measurement.

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### Preparationof ligand$C_{16}H_{19}N_3O_3S$ (L).

Thelig and Methyl-6-[2-amino-2-(4-hydroxyphenyl) acetamido]-2,2-dimethyl-5-oxo-1-thia-4-azabicyclo[3.2.0] heptane-3-carboxylate was prepared according to Obeed and Khalidan (8).

Amixture of amoxicillin (0.02mol) and an excess of absolute methanol (50ml) with 1-2 drops of concentrate sulfuric acid was placed in 100 ml round bottom flask. The mixture was refluxed in a bath water ($50^{O}$C) for 3 hrs. The solution was cooled and poured into crushed ice. The obtained precipitate was filtered, dried and recrystallized in ethanol.

### Preparation of complex.

An amount of (0.236g of$CoCl_2.6H_2O$)was dissolved in 10 ml of ethanol and mixed with ( 0.93 g of ligand dissolved in 15 ml ethanol) in molar ratio 1:2 (M: L) in a round bottom flask under heating and refluxed for 3 hrs. The colored precipitate was filtered then washed with ethanol and dried then keeped in desiccator.

### Preparation of ($TiO_2$) Nano particles using (Sol-Gel) method.

Five milliliters of glacial acetic acid (99.5%) were added drop wise to 50 ml of distilled ethanol (99.9%) and vigorously stirred for about 1 hr., then (6.3) ml of Titaniumtetraisopropoxide (TTIP 99%) were added drop wise to the clear cooled glacial acetic acid- ethanol solution with continuous stirring for about 1 hr, dryingfor 1hr at 50 $^{O}$C and calciniedat 400 $^{O}$C for 3 hrs(9).

### Photolysis Procedure.

Thermostatic cylinder photo cell (75 ml) and medium pressure mercury lamp were used. A series of standard solutions of Co- complex was prepared with a concentration of $[1\text{-}3.5]\times10^{-4}$ M and the absorption values were recorded at a wavelength of (450) nm.

### Results and Discussion:

The physio – chemical properties were summarized in table (1).The complex powder was stable at room temperature with color listed in table (1). A molar ratio of (1:2)(M: L)was use to prepared complex, which can be suggested according to the analytical data.

**Table 1. Analytical and some physical data for the ligand and its cobalt complex.**

| Compounds | Elemental analysis Found (cal.) | | | | M.P °C | Color | %Yield | $\mu_s$ $cm^{-1}$ |
|---|---|---|---|---|---|---|---|---|
| | C | H | N | Co | | | | |
| $C_{16}H_{19}N_3O_3S$(L) | 48.04<br>(49.1) | 5.26<br>(5.30) | 18.2<br>(18.9) | 0.00 | 196 | Light yellow | 87 | 0.00 |
| $Co(C_{32}H_{41}N_6O_{7.5}S_2)Cl_2$ | 38.67<br>(39.5) | 4.59<br>(5.11) | 12.35<br>(13.9) | 5.73<br>(6.12) | 211 | Greenish blue | 78 | 80 |

### Infrared spectrum.

The FTIR spectrum of ligand exhibit a strong band at (1778) $cm^{-1}$ due to the presence of carbonyl group of ester that appears usually around (1700) $cm^{-1}$, (10) β-lactam carbonyl group occurred at a significant value about (1724)$cm^{-1}$.The band of the ν(NH) group appeared at the (3178.5) $cm^{-1}$ in the spectrum. If the band of carbonyl group of ester did not shift in complex means that the carbonyl did not participate in coordination. The bands attributed to the β-lactam was shifted to higher wave number in complex about (15.8)$cm^{-1}$ indicating that β-lactam carbonyl group was involved in complex formation (11). The bands of ν(NH) group were shifted to higher wave number in complex about (54)$cm^{-1}$so the ligand behaves as a bidentate through the coordinationvia oxygen and nitrogen atoms. This results are further supported by the occurrence of M-O and M-N bands in the spectrum of the complex at the range mentioned in literatures(12, 13).The other important characteristic peaks in the FT-IR spectrumare listed in table (2).

**Table 2. FT IR bands for Ligand (L)and its complex (A)**

| Comp. | νNH | νC=O Est& Amid | νCH Alph arom | νC=O β-lactam | νC=C | νC-O | νM-N | νM-O | M-Cl | $\nu H_2O$ |
|---|---|---|---|---|---|---|---|---|---|---|
| L | 3032 | 1778<br>1689 | 2970<br>3151 | 1724 | 1616 | 1242 | - | - | - | 3444 |
| Co-complex | 3086 | 1778<br>1648 | 2974<br>3155 | 1739.8 | 1616 | 1230 | 532 | 415 | 288 | 3475 |

## Electronic spectrum.

The electronic absorption spectrumis used to predict the geometry according to the shape and number of observed peaks beside those calculated onces depending on the information of Racah parameter ($B^-$), 10Dq and nephelauxetic factor (β), free ion ($B_o$) using Tanabe Sugano diagram.

The electronic spectrum of the ligand shows three main bands, the first and the second absorption bands appeared at (42372 and 36363) cm$^{-1}$ are due to $(\pi - \pi^*)$ transitions located on (C=C)group. The third absorption appeared at (31250) cm$^{-1}$ attributed to $(n - \pi^*)$ may be located on the (C =O) group (14).

## Co- complex.

The magnetic measurements showed the cobalt ion in its greenish blue complex to be paramagnetic with ($d^7$)configuration in distorted tetrahedral environment (15),in infrared region the($\nu_1$) is found as a weak band at (3451)cm$^{-1}$ due to transition $^4A_2 \rightarrow {}^4T_2$ the ($\nu_3$) was calculated as the average to three bands and assigned to the transition $^4A_2 \rightarrow {}^4T_1(p)$ at about (16977) cm$^{-1}$,while the ($\nu_2$) was calculated by reference to T.S.D for $d^7$ configuration (16,17) to the transition $^4A_2 \rightarrow {}^4T_2(f)$. The Racahparameter $B^-$, nephelauxetic factor β and the value of (10 Dq) were also estimated as describe in table(3).

**Table 3. The electronic spectrum data and their assignment of the ligand and prepared cobalt complex**

| Comp. | Band cm$^{-1}$ | Assignment | $B^0$cm$^{-1}$ | Dq/$B^-$ | β | 10Dq cm$^{-1}$ |
|---|---|---|---|---|---|---|
| L | 67613<br>42372<br>31250 | <br>π-π$^*$<br>n- π* | ------ | ------ | ------ | ------ |
| Co-complex | 3451<br>5217(cal.)<br>16977 | $^4A_2 \rightarrow {}^4T_2$<br>$^4A_2 \rightarrow {}^4T_2(F)$<br>$^4A_2 \rightarrow {}^4T_1(p)$ | 970 | 0.47 | 0.76 | 3470 |

## Characterization of Nanostructure $TiO_2$.

Atomic force microscope was used to detect ($TiO_2$) nanoparticles structure and to get morphological information. Fig(1, 2) show typical surface AFM image (in three and two dimensions). The nano particle is spherical in shape(18)and has an average diameter of (52.21) nm.

The Scanning Electron Microscope (SEM) show the surface morphology (Fig 3) of synthesized $TiO_2$nano powder by sol- gel method after calcinations at (400°C) which illustrate the small size of the particle show that the synthesized nanoparticles are spherical and have homogenous morphology. (18,19).

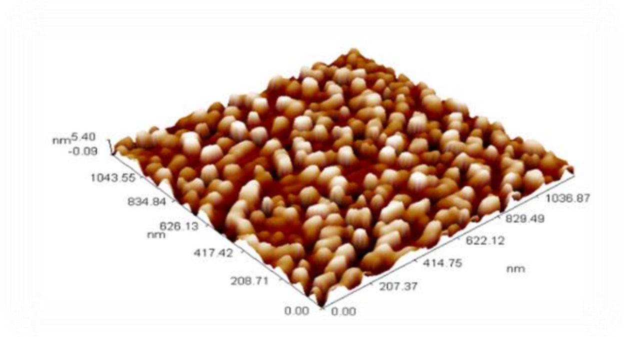

Figure 1. The AFM -3D image

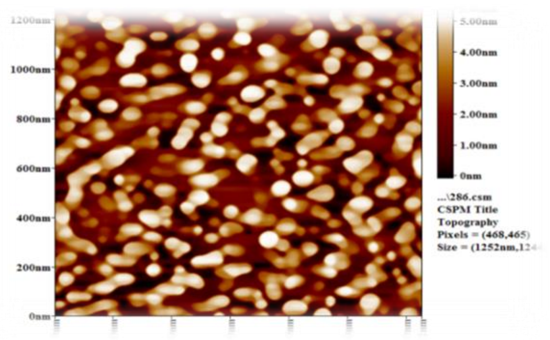

Figure 2. The AFM 2D image

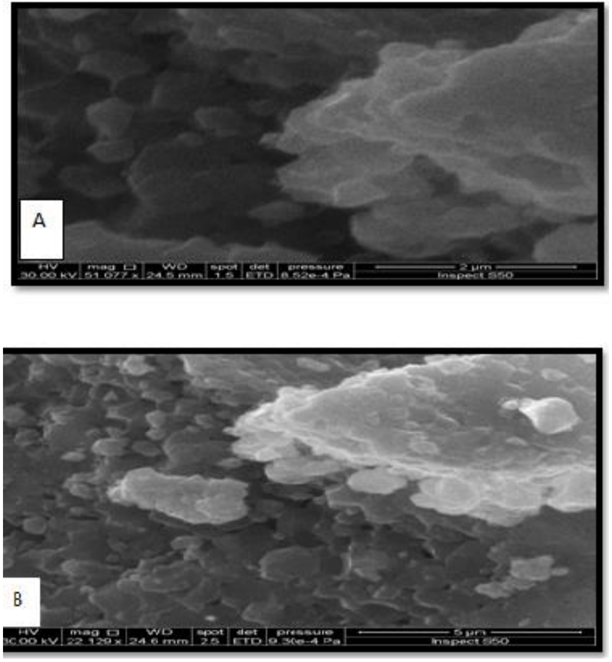

Figure 3. SEM images of synthesized $TiO_2$nano powder by sol- gel after calcinations at 400°C.(A) 2µm,(B)5 µm

## XRD- Diffraction

XRD Technique was used to investigate the phase transformation of the prepared sample, Fig (4) shows the XRD pattern for the calcinated sample calcinations at (400 °C) for (2hr), the anatase phase was only observed. Themain diffraction peaks were at 2θ =25.30, 37.83 and 48.05 corresponding to (101), (004), (200) planes respectively (20, 21).Crystalline sizeis 10.23 nm calculated by using the Scherrer formula.

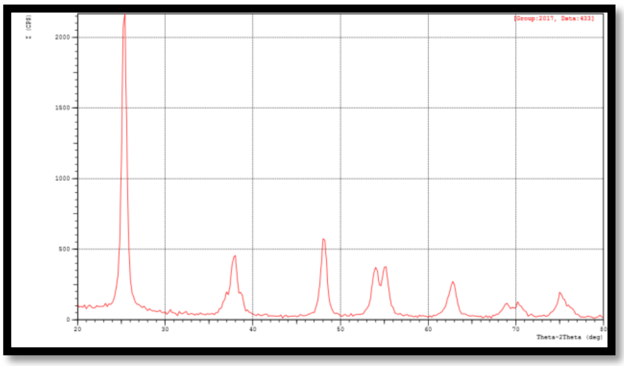

**Figure 4. TheXRD-Diffraction of $TiO_2$ nano particles at calcinations temperatures, 400°C.**

## Photodegradation of Co-complex

The removal degradation percentage of Co-complex was defined in Eq(1).

Degradation (%) = $[(C_0 - C_t)/ C_0] \times 100$….. (1) . Where $C_o$ is the initial concentration and $C_t$ is the concentration at irradiation time. The calibration curve used to calculate $C_t$ is shown in figure (5).

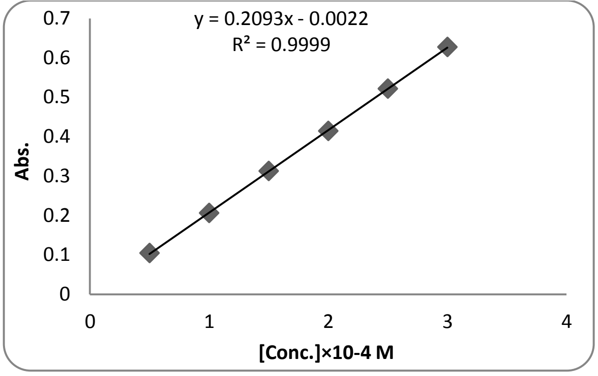

**Figure 5. The Calibration curve for Co-complex a wavelength (450) nm, at25°C.**

## Effect of light.

According to the results, fig (6) show that no photodegradation for complex was happen in dark and get photodegradation in UV, whereas UV plays an important role in photo degradation by using $TiO_2$. Reactions are initiated when a photon of higher energy level or equal to the band gap energy is absorbed by $TiO_2$ catalyst promoting an electron ($e^-$) from the valence band to the conduction band with simultaneous generation of a positive hole (h+) in the valence band. Themechanism of radical's generation (${}^{\bullet}OH$ and $\bullet O_2^-$) is presented as in the following equation

$TiO_2+h\nu \rightarrow TiO_2\,(e^-+h^+)$ …………..1

$H_2O+TiO_2\,(h^+)\rightarrow TiO_2+{}^{\bullet}OH+ H^+$ ……2

$O_2+TiO_2\,(e^-)\rightarrow TiO_2+\bullet O_2^-$ ……………..3

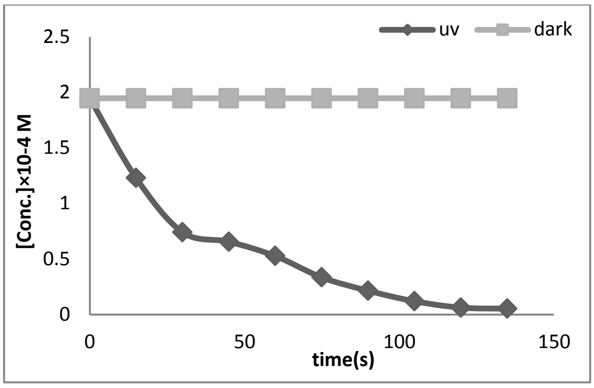

**Figure 6. Effect of light and dark condition on photodegradation of Co-complex with $TiO_2$ nanoparticles (0.003g)at 25°C.**

## Effect of Catalyst amount.

Fig (7) includes the results obtained for the [$2\times10^{-4}$] M Co-complex using a lamp (MPML) and different amounts of the nano-photo catalyst ($TiO_2$)(1 -5) $\times10^{-3}$g with keeping all other parameters fixed.

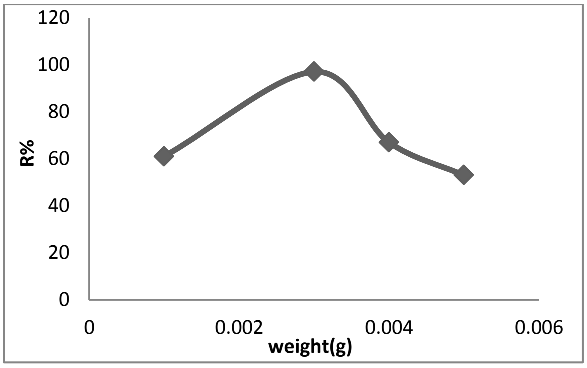

**Figure 7. Influence of catalyst loading ($TiO_2$)on Co-complex($2\times10^{-4}$ M)photodegradation at 25°C.**

The best amount of the catalyst (0.003 g) isused in a series of experiments to study the photodegradation of different concentrations of the Co-complex and the results obtained shown in figure (8).

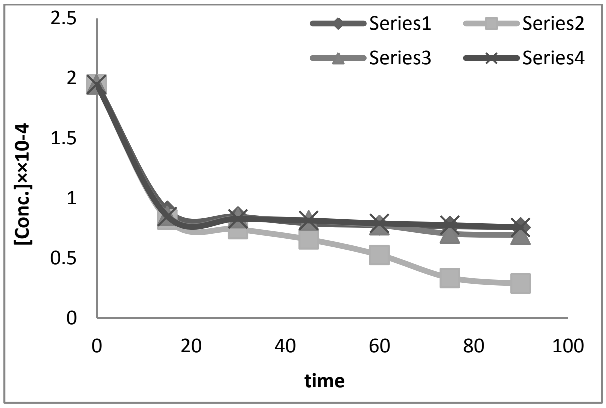

**Figure 8. Relationship between time and complex concentration with using different $TiO_2$nano particles.1.$TiO_2$ (0.001g), 2.$TiO_2$ (0.003g).3. $TiO_2$. (0.004g), 4.$TiO_2$ (0.005g).**

Fig (7), (8), show that the best concentration for photocatalyst lead to higher photodegradation was (0.003) g. Increasing the concentration of the photocatalyst ($TiO_2$) higher than (0.003)g lead to decreasing in the degradation process and mineralization rate due to aggregation of $TiO_2$ nanoparticles at high concentration causing a decrease in the number of surface active sites and an increase in the opacity and light scattering the falling light(22).

## Effect of temperature.

A series of experiments were carried out at different temperatures (278 -318) K, the rate constant (k) was calculated at each temperature, and the values are (0.003, 0.009, 0.027, 0.005, 0.006) $s^{-1}$ at (278, 288, 298, 308and 318)K respectively for the degradation of Co-complex [$2\times10^{-4}$] M Conc. with 0.003g $TiO_2$. The results showed no significant change in the rate of complex degredation.

## Effect of pH.

The photodegradation of Co-complex was studied at five different pH values under UV-light. pH value of the solution was adjusted before irradiation. The adjustments of acidic (pH 2and 4) and alkaline medium (pH 6and 10) were made using diluted HCl or NaOH respectively as shown in Fig (9).The results show complex and $TiO_2$ are both positively charged in more acidic conditions $[\,pH < 4]$. These should hinder the adsorption of complex on $TiO_2$ surface and slow the reaction (7).In alkaline medium the charge of complex and $TiO_2$is neutralized no attraction forces between the catalyst and complex.

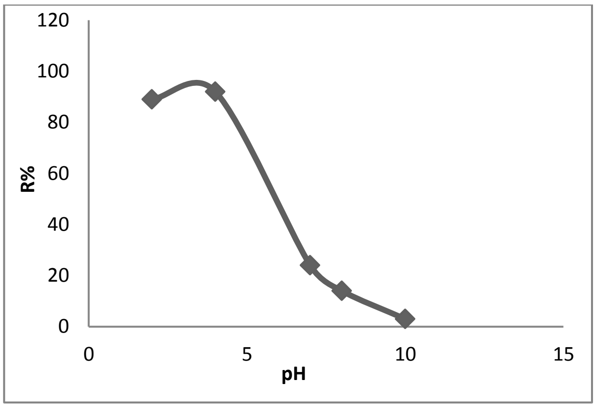

**Figure 9. Effect of pH value on the photo catalytic degradation rate of Co- complex on $TiO_2$ (0.003) g at 25$^{O}$C.**

## Effect of sunlight.

The impact was identified of sunlight radiation on the amount of photodegradation of Co-complex with and without $TiO_2$nano powder and irradiated by sunlight (2 hours), where the intensity of the sunlight $1.425\times10^{-8}$ Ein.$l^{-1}$.$Sec^{-1}$. The result was observed that photodegradation did not happen in absence of $TiO_2$nanopowder, but occurs when $TiO_2$ was used in solution inside the cell. Fig (10) shows the effect of sunlight on the Co-complex conc. Note through the fig (10) that the amount of photodegradation of the sunlight radiation is at a slow rate compared to the effect of UV, which increases the speed of photodegradation and can be explained by the low intensity of sunlight radiation. This wavelength has a significant effect on $TiO_2$ by stimulating electrons from Valence Band (V.B) to conduction Band (C.B).

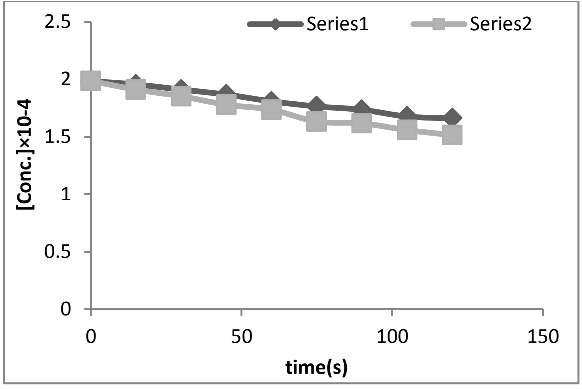

**Figure 10.Effect of sunlight on photodegradation Co- complex conc. at 25 $^{O}$C. 1.without $TiO_2$, 2. With $TiO_2$**

## Effect of light Sources.

Under constant parameters (temperature, catalyst) two different optical sources were used UV lamp and Visible Irradiation. From the results one can conclude that photodegradationoccur when using different sources of light, but the difference between them depends on the intensity or wavelength of the radiation emitted from the light source. Fig (11) shows the result of different sources (UV) and (Vis)effect. Comparing the source (UV) and (Vis), onecan show that the degradation using (UV) light was higher than Vis light .This may be because when the wavelength decrease the number of electrons moving from the valance band to conductive bandincreased(energy gab of TiO2 is 3.29 eV).

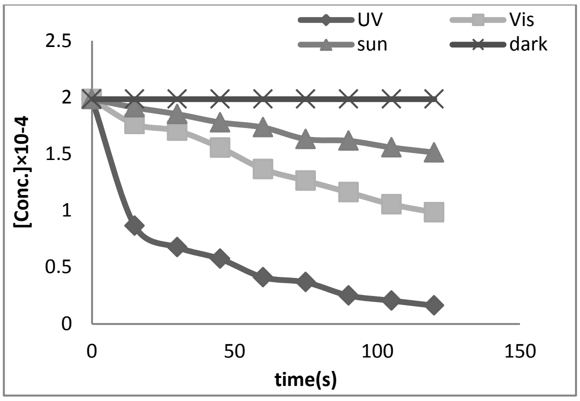

**Figure 11. The effect of different sources of light with (0.003g) $TiO_2$ on Co-complex degradation. 1-UV ,2- Vis ,3- Sun light,4- Dark**

## Kinetics Study.

The reaction order was determined using the trail method by applying different equation of order reaction 0,1 and 2

C = $C_O$-kt ………….. (5) for zero order

lnC = ln $C_O$-kt ………(6) for first order

$\frac{1}{c}=\frac{1}{c_O}$+kt ………… (7) for second order

The results obtained are shown in Fig (12).The values of correlation efficient $R^2$obtained from the plotting of the above equations are 0.898, 0.973 and 0.797 for 0, 1 and 2 reaction order respectively, which indicate that the photodegredation reaction followed the first order reaction.

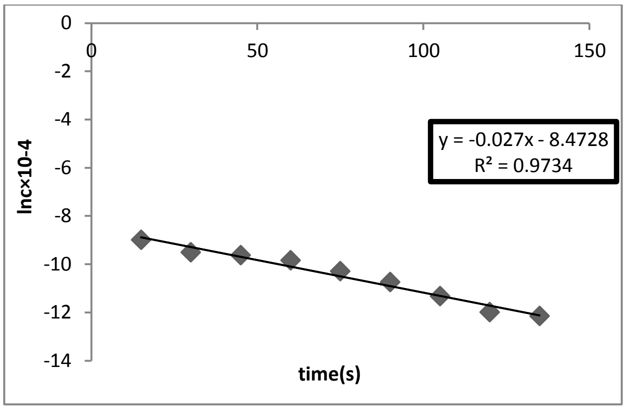

**Figure 12. The first-order kinetics reaction of Co - Complex at optimum conditions; 25 $^{O}$C, [$2×10^{-4}$]M Conc. of complex with (0.003 g). $TiO_2$**

The ctivationenergy was calculated by use Arrheniuseqation.

$ln\,k = ln\,A - Ea/RT$ …… (8).

The value of($Ea$)was calculated equal to(6.6512 kJ /mol)which indicates that the thermal activation steps are negligible in this photo catalytic process (23).The result obtained are shown in Fig (13).

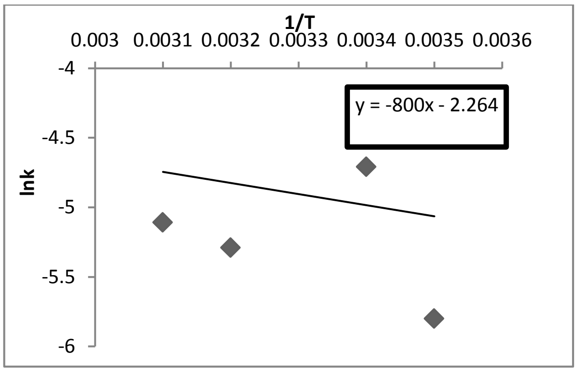

**Figure13. Arrhenius plot for the degradation of Co-Complex, with $TiO_2$ +UV.**

## Conclusions:

**1.** FT-IR Spectra of complex showed carbonyl group (C=O) for ester does not shifted in complex, therefore oxygen of carbonyl (C=O) does not participate in coordination. The bands which were attributed to the β-lactam were shifted to higher wave number in complex about (15.8) $cm^{-1}$ indicating that β-lactam carbonyl group (C=O) were involved in complex for mation. Tetrahedral geometry was suggested for the Co (II).

**2.** AFM information about $TiO_2$ nanoparticle powder show the average nanoparticle size was (52) nm, the SEM illustrates the small size and the spherical shape of the synthesized nanoparticles which have homogenous morphology**..** The XRD results showed the anatase phase has been only observed and Crystalline size is 10.23 nm has been calculated by using the Scherrer formula.

**3.**Comparing the source (UV) and (Vis), one can show that the degradation using (UV) light was higher than Vis light .This may be because when the wavelength decrease the number of electrons moving from the valance band to conductive band increased (energy gab of TiO2 is 3.29 eV)..

**4.**The best concentration for photocatalyst lead to higher photodegradation was (0.003) g. Increasing the concentration of the photocatalyst ($TiO_2$) higher than (0.003)g lead to decreasing in the degradation process and mineralization rate due to aggregation of $TiO_2$ nanoparticles at high concentration causing a decrease in the number of surface active sites.

**5.** The photocatalytic degradation of Co-complex followed a pseudo- first-order kinetic reaction; the low value of activation energy indicates that the thermal activation steps are negligible in this photo catalytic process. Therefore the photochemical system is generally temperature independent.

## Conflicts of Interest: None.

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# تحضير وتشخيص معقعد الكوبلت من الليكند $C_{16}H_{19}N_3O_3S$ والتحلل الضوئي باستخدام اوكسيد تيتانيوم الناوني كعامل مساعد

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## الخلاصة:

تم تحضير معقد الكوبلت من تفاعل اللينكد المشتق $C_{16}H_{19}N_3O_3S$ مع ملح كلوريد الكوبلت الثنائي وشخص المقعد بواسطة( جهازتحليل العناصر ومطيافية الامتصاص الذري واطياف الاشعة تحت الحمراء واطياف الاشعة الفوق البنفسجيةو المرئية والحساسية المغناطسية والتوصيلية) حيث تبين من خلال التشخيص ان الارتباط تم من خلال ذرة الاوكسجين لمجموعة الكاربونيل وذرة النايتروجين في لاكتام –β والترتيب الهندسي لمعقد الكوبلت هو هرم رباعي السطوح . تمت دراسة حركية التفكك الضوئي للمعقد المحضر بوجود ثنائي اوكسيد التيتانيوم النانوي المحضر كعامل مساعد في ظروف مختلفة(التركيز،درجة الحرارة ،الاس الهيدروجيني) وجد ان تفاعل التجزئة الضوئية من الرتبة الاولى وتم حساب طاقة التنشيط وكانت تساوي(6.6512 kJ /mol).

**الكلمات المفتاحية:** مشتق الاموكسسلين، التحلل الضوئي،العامل المساعداوكسيد التيتانيوم النانوي