Intercalation of ZnO with 2-mercapto – N` - (2-mercaptoacetyl) and Study of its controlled released.

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Received:- 10/5/2017 Accepted:-13/7/2017

Abstract:-

Metal nanoparticles have been extensively studied over the last decade. Nanomaterials were an important subject in basic and applied sciences. Nanoparticles of zinc oxide compounds have received considerable attention because of their unique antibacterial, antifungal, properties.
The aim of this work is to prepare a hybrid Nano-crystalline composite of zinc oxide molecules using the chemical method and the diagnosis of ZnO-NPs using FT-IR, AFM and XRD, the intercalation of 2- (2-mercaptoacetyl) with ZnO accurse by the ion exchange method. The shape of the fibrous particles was determined by the atomic force microscope. The dimensions of these compounds ranged from 24.6*30.8 nm and Mean grain size (57.1) nm . The release ratio of the atoms of this compound (pH = 4,6,8,13), . The reaction is governed by the pseudo second order reactions.

***Keywords:* ZnO-NPs, 2-mercapto – N` - (2-mercaptoacetyl), controlled release, intercalation.**

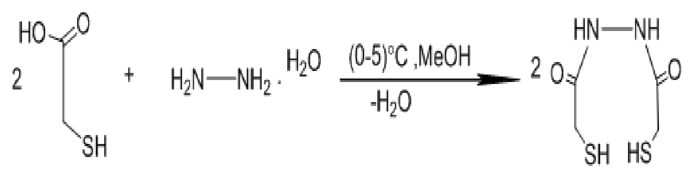
Chemistry Classifiication QD540 -801

Introduction:-

The field of engineering science is one amongst of every for most active analysis areas in trendy materials science [1,2]. Nanoparticles exhibit new or improved properties that are supported with specific characteristics like size, distribution, and morphology. There are spectacular developments within the field of engineering science among the recent past years, with numerous developed methodologies to synthesize nanoparticles with specific type and size that are counted for specific needs. New applications of nanoparticles and nanomaterials are increasing rapidly; whereas operative with nanomaterials has allowed a much better understanding of biology [3,4]. As a consequence, there's the potential of providing novel ways that concerning the treatment of diseases that were previously powerful to specialize in as a result of size restrictions.

For medical science applications, the synthesis of bio-functional nanoparticles is extraordinarily very important, and it's recently caught the attention of various analysis groups, making a constantly evolving area [5–8].The growing demands for environmental friendly nanoparticles, researchers have utilization inexperienced methods for the synthesis of varied metal chemical compound nanoparticles for pharmaceutical applications[9,10]. Various ways are used to synthesize the ZnO nanostructured materials (such as optical device deposition and thermal evaporation) that are typically treated at high temperature[11,12].However, it still remains the nice challenge to develop. New approach for fabricating ZnO nanomaterials, significantly for preparing aluminum -doped ZnO nanorods by the easy method of low price, straightforward operation, etc. Soft chemistry route is a good and promising approach to synthesize ultrafine ZnO nanorods and nanowires at low temperature [13,14]. Our work focusing on intercalation of ZnO with Mercaptohydrazine compound [15], via the ion exchange method to obtain a nano particular used as carrier and control release of the organic molecules

This investigation used mercabtoacetamide as suitable reagents in the determination of some metal ions is well-established[16]. Due to the presence of nitrogen, Sulphur and oxygen atoms at suitable positions, N-alkyl-2-mercaptoacetamides are reported to behave as monofunctional bidentate ligands and also show linkage isomerism in their complexes[17- 20]. In most of the metal complexes these ligands are reported to behave as N,S bonded or O,S$^-$ bonded ligands[21-24]. However ,some complexes have also been reported in which these ligands are bonded through N and O atoms with metals. In addition to above , these ligands are reported to exist in following two conformational isomeric forms.



Experimental Methods:

500mg of [2-mercapto – N` - (2-mercaptoacetyl] was dissolved in 100ml the previous solution was added to 0.5gm ZnOdi- Layer in 50ml distilled water at a conical flask with stirring for three hours, the mixture was placed in a shaker water bath and set at 70^{o}C for 18 hours , centrifuged and washed four times with de ionized water, dried in oven at 70^{o}C,then crushed. Prepared some solution in different p H which controlled by 0.1N of HCl for acidic and by 0.1N of NaOH for base.

Characterizations

X-Ray powder diffraction patterns (PXRD) were obtained with a Shimadzu XRD-6000 powder diffractometer using (λ=1.5406 Å), scan rate = 1 degrees/min.

The surface morphology and bulk structure of the sample were observed by Atomic force microscope (AFM) and FT-IR .

Results and disscussions

XRD Powder X-ray diffraction

Powder x-ray diffraction patterns of ZnO nanoparticular resulted from ZnO intercalation products with [2-mercapto – N` - (2-mercaptoacetyl] are shown in Fig.[1]. All the materials show sharp diffraction peaks,where indicative of well- ordered and high crystallinity. The intercalation of an inorganic anion resulted in an expansion of the basal spacing of ZnO. This is due to the size and spatial orientation of the anion in the organic interlamellar of the ZnO. Intercalation of [2-mercapto – N` - (2-mercaptoacetyl] show well-ordered phase nanohybrid. The observation of the other harmonics with the interlayer distance this was showed by AFM paragraph as fiber structure. This indicates that [2-mercapto – N` - (2-mercaptoacetyl] Find its own way and assemble its self for the formation of monophasic nanohybrids, resulting in the formation of well-defined nanohybrid. In the same time the peaks belong to ZnO was reduce in intensity comparing to the more intense pattern of [2-mercapto – N` - (2-mercaptoacetyl] Intercalated with ZnO.

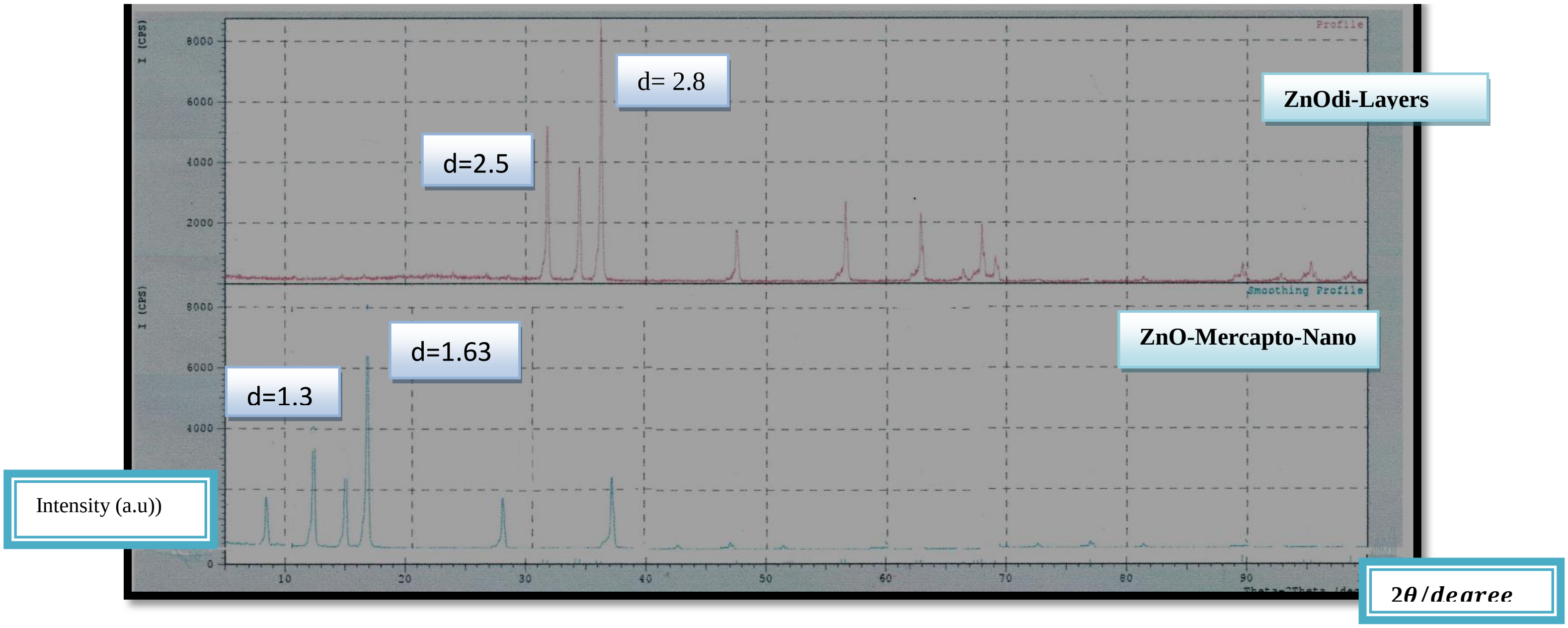


Fig [1] :- XRD spectrum of ZnO di-Layers and intercalated ZnO-Nanoparticles (ZnO-Mer.) .

FT-IR Spectrum:-

FTIR spectrum of the zinc oxide compound: The spectrum of the zinc oxide compound showed a number of bands at frequencies 466cm^{-1} and cm^{-1} 534 indicating Zn-O as shown in Fig[2].

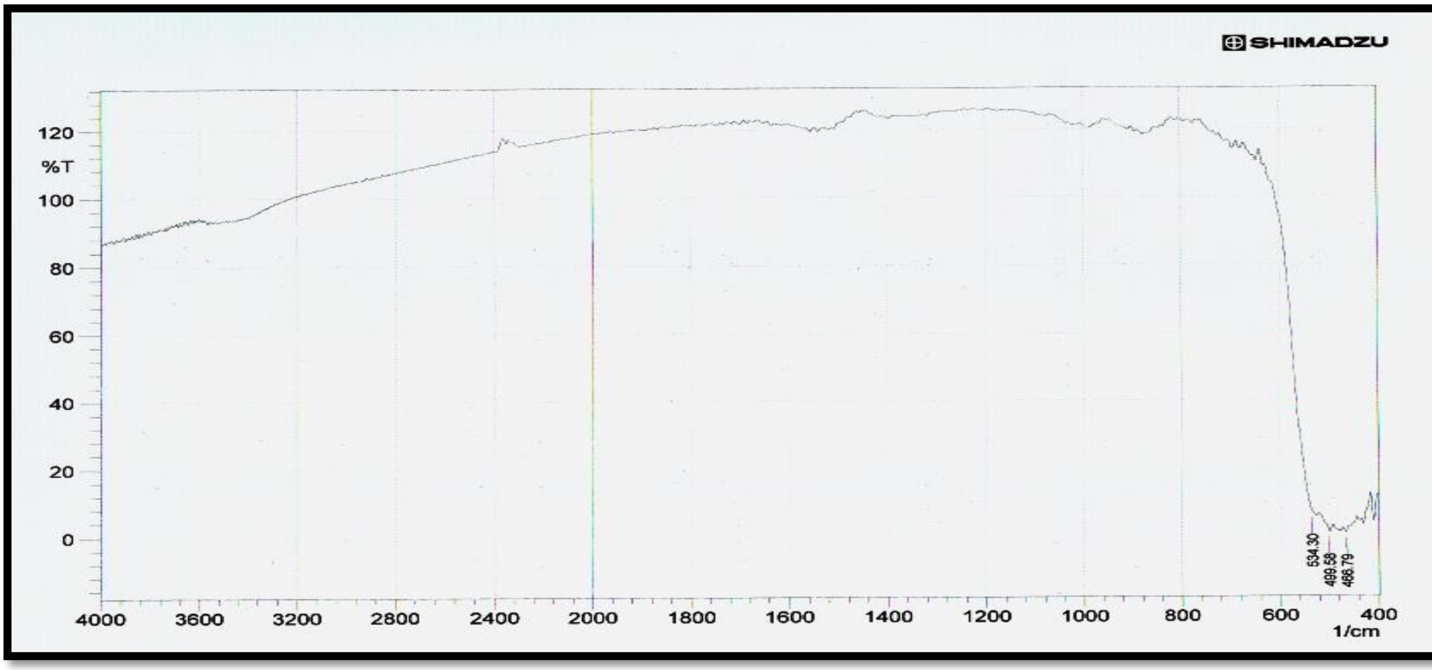
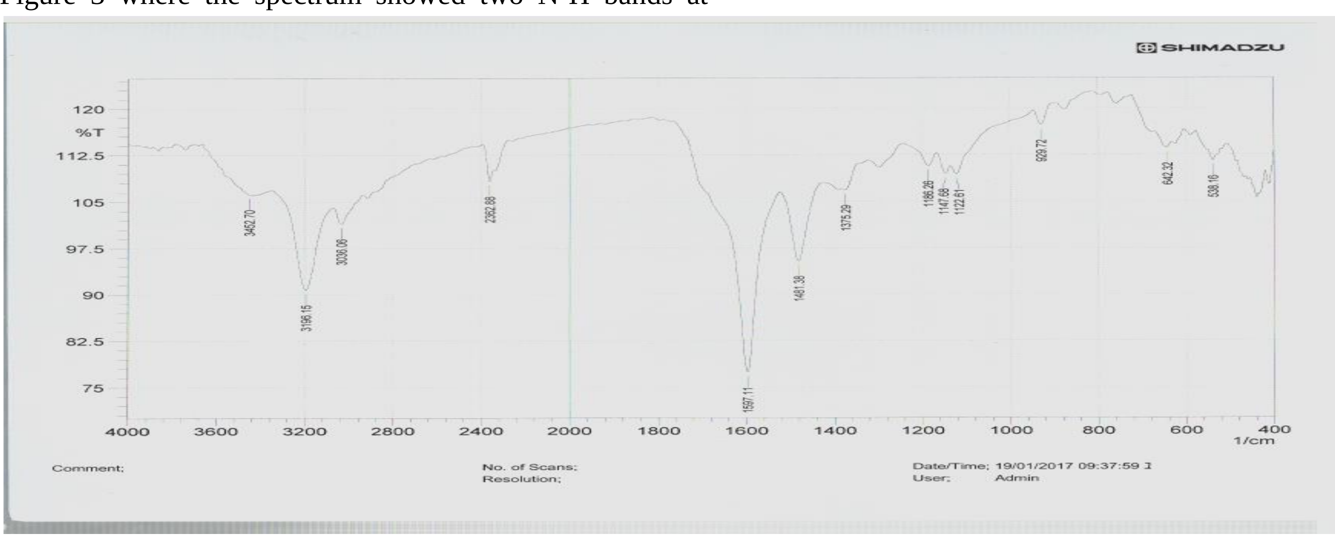


Fig.(2)FT-IR spectrum of the Zn-O compound

FTIR spectrum of the organic composite compound:

The spectrum of the organic compound showed some beams indicating the active aggregates as shown in Figure 3 where the spectrum showed two N-H bands at 3200cm^{-1} and the appearance of the S-H band at cm^{-1} 2362 ,Cm^{-1}1481 the N-N package has emerged as well as some other packages for the effective combinations of the compound Mercaptohydrazine.



Fig(3) FT-IR spectrum of the* Mercaptohydrazine *compound

FT-IR spectrum of the hybrid nanoparticle Record:

The infrared absorption spectra of the hybrid nanoparticle resulting from the reaction of zinc oxide with the composite organic compound Mercaptohydrazine showed the appearance and disappearance of many absorption revision as shown in Fig. (4). The merger process resulted in a new IR absorption spectrometer combining the two spectra of the two basic substances.

The figure shows the disappearance of the N-H vibration band at 3200 cm^{-1},The appearance of new sharp and clear band at the wave number (2360 and 2341) Cm^{-1} indicates the loss of proton from amide and its association with zinc oxides to form a new compound. And C = O packet at 1651 cm^{-1}, the Carbonyl Amide package, appeared the C-H band was shown at 2341 cm^{-1} as the C-S frequency at 952 cm^{-1}.

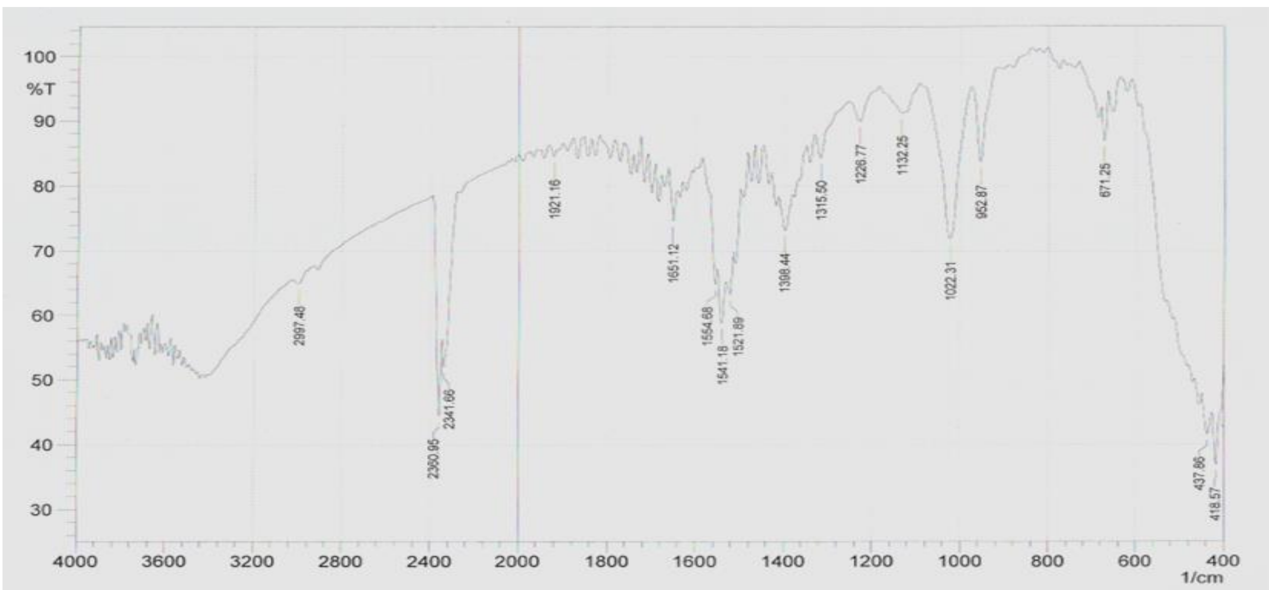


Fig. (4)FT- IR spectrum of the hybrid Zn –O nanoparticles

<u>Study of Morphology by AFM :</u>

Atomic force Microscope (AFM) was used to study the surface topography and the surface crystalline structure of the nanoparticles formed. **Fig.5** shows the topical analytical picture of surface roughness and the square root can be measured from the following equation $R_{ms} = \sqrt{\sum_{i=1}^{N} \frac{(Zi-Zav)^2}{N}}$

Where Zi represents the value of Z for each point , (Z_{av}) represent the average of (Z ,N) values representing the number of points measured, Surface roughness was found to be 11.16nm. This indicates good crystalline uniformity and excellent homogeneity. The average square root value is 7.862nm. This indicates the smoothness of the surface. The higher the mean square root, the greater the roughness of the surface and vice versa. The maximum value of the surface was 293.41nm. This value showed the topographical appearance of the membrane surface completely. The mean rate of crystalline granularity and other band was 241.41nm and the average grain size 90nm **Fig. 5** shows that the nanoparticles formed have a vertical structure and a particle size of 45.3nm.

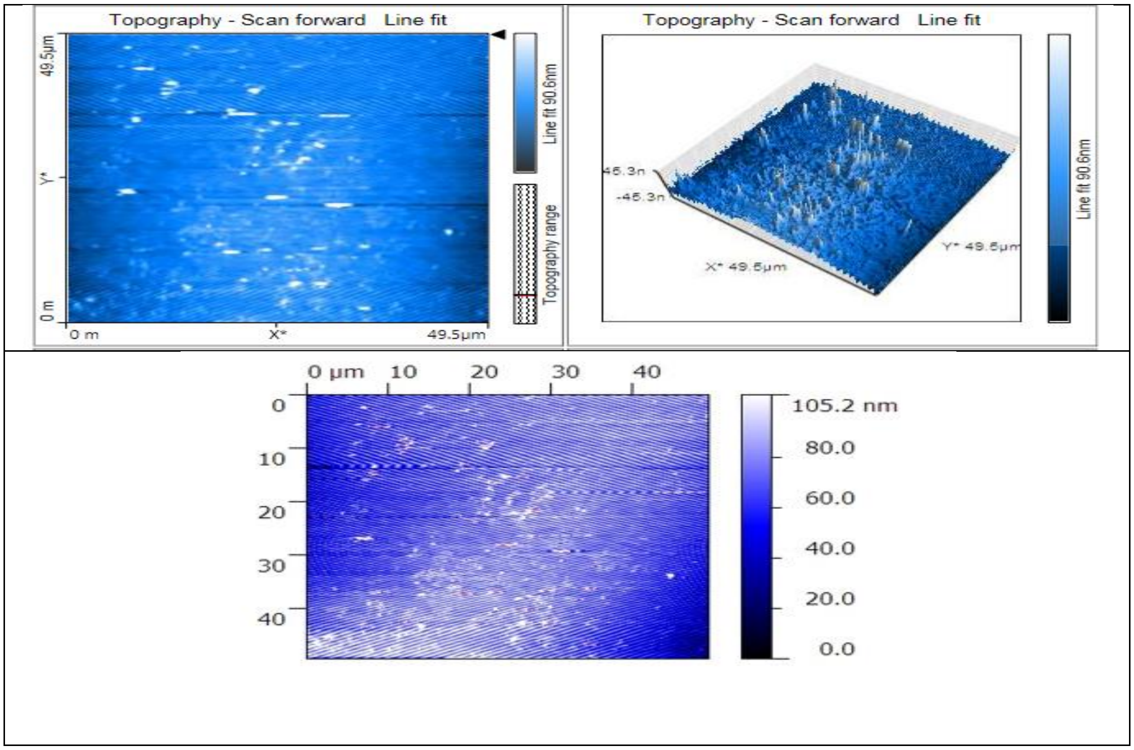


Figure (5) 3D AFM image for prepared compound

Ion exchange Kinetics :-

The ion exchange kinetics release the velocity of the separation and diffusion of ions from the solid phase to the liquid phase and the opposite occurs at the same time after overcoming all the interstellar and implicit molecular forces that impede the ion exchange process in the solution. The ion exchange kinetics are of great importance. They determine the duration of the ion exchange process and the equilibrium state after which the ion exchange process stops. The ion exchange kinetics depends on several factors: ion concentration, temperature, helper presence, and rapid propagation of ions within the exchanger and the size of the ion exchange. Therefore, ion exchange dynamics can be dealt with on the basis of zero- and first-degree interactions and the second false. By applying the equations of Ho and Mckay [25] based on the equations of Lagergran. Therefore linear equations were derived to release the ions from the surface of the steel to the water solution of each grade respectively:

$C_t = K_0\, t$ ……..(1) ---------------------Zero order

$-\log\left(1-\frac{Ct}{Cf}\right) = \frac{K_1\, t}{2.303}$ …….. (2)--------------First order

$\frac{t}{Ct} = \frac{1}{K_2 Cf^2} + \frac{t}{Cf}$ ……… (3)------ -----Second order

The release profile of mercaptohydrazine from the inter layer of ZnO-Mer. in to aqueous solution at PH(2,4,8,13) ,the accumulated Mer. release in to the aqueous solution at 120,160 and 220 minutes respectively, fitting the release with deferent’s equations , 0 order, 1^{st} order , 2nd order, the second order model is the more fitted one with R^2 =1 fig.(6).

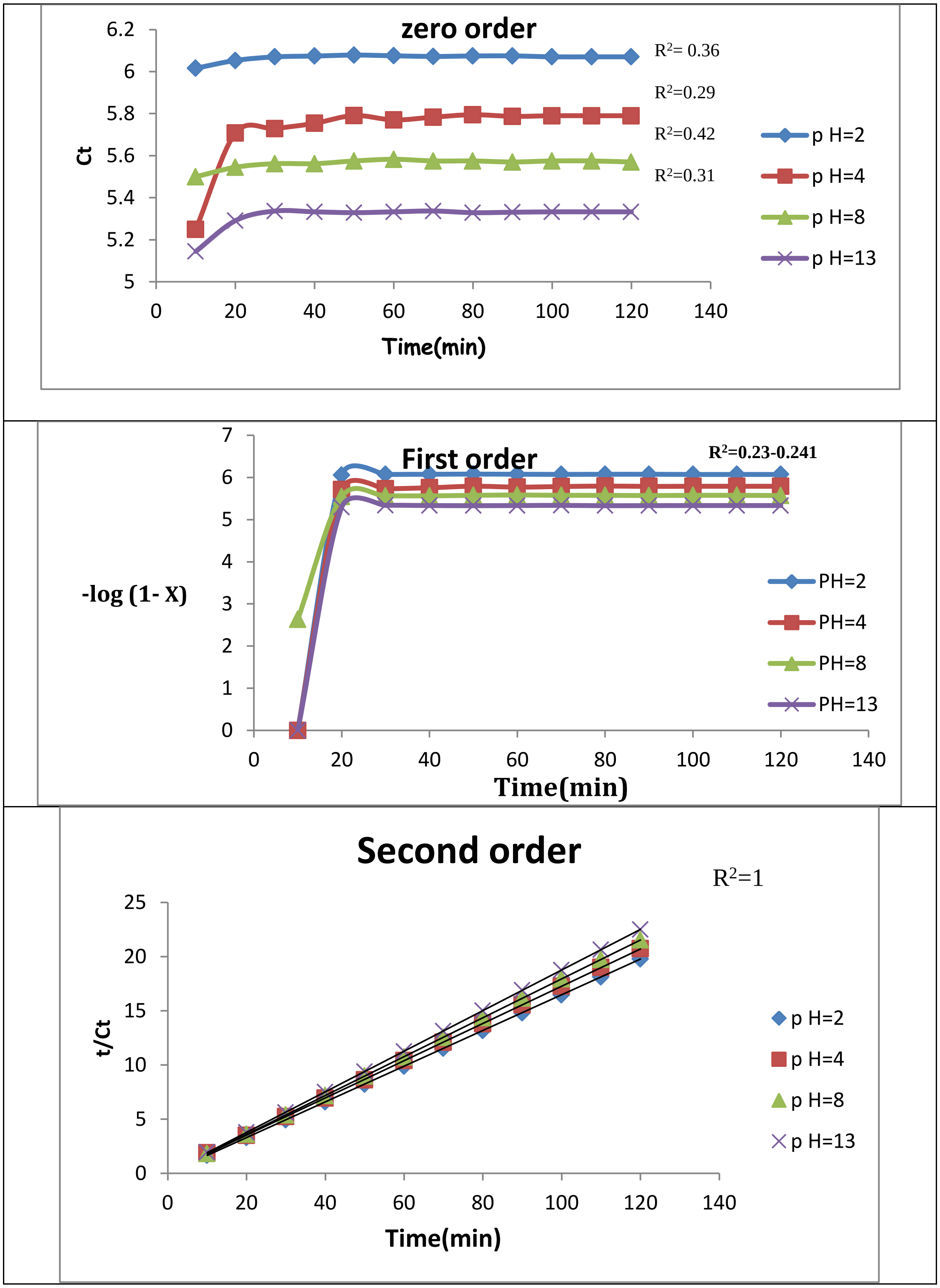


Figure (6)Zeroorder, pseudo first and pseudo second order model of the released controlled

Conclusions;-

This article aimed to investigate the possible synthesis by intercalation of Mercapto compound between the layers of ZnO to get new nanoparticles can it use as durg according to the biological significance of sulfur compounds as well as the ease of preparation of the multi-layered ZnO compound .

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إقحام أكسيد الزنك مع مركب مركبتواسيتامايد ودراسة السيطرة على تحرر جزيئاته.

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قسم الكيمياء- كلية التربية للعلوم الصرفة- جامعة كربلاء-العراق

تاريخ الاستلام:- 2017/6/18 تاريخ القبول :- 2017/10/5

الخـــلاصــــة:

درست الجسيمات النانوية المعدنية بشكل مكثف خلال العقد الماضي .المواد النانوية كانت موضوع مهم في العلوم الأساسية والتطبيقية .وقد تلقت الجسيمات النانوية لمركبات أوكسيد الزنك اهتماما كبيرا بسبب خصائصها المضادة للبكتيريا الفريدة من نوعها، المضادة للفطريات.

والهدف من هذا العمل هو تحضير مركب نانوي هجين من جزيئات أكسيد الزنك باستخدام الطريقة الكيميائية وتشخيص الجسيمات النانوية ***ZnO-NPs*** باستخدام الأشعة تحت الحمراء FT-IR ، المجهر القوة الذرية AFM و حيود الأشعة السينية(XRD)كما تمت عملية الإقحام لمركب[2-mercapto – N` - (2-mercaptoacetyl] بواسطة طريقة التبادل الأيوني، وكان شكل الجزيئات الناتجة ليفية شخصت بواسطة (مجهر القوة الذرية) حيث تبين إن أبعاد هذه المركبات تتراوح من 24.6*30.8)nm)، متوسط حجم الحبوب(57.1 nm)وتمت دراسة نسبة التحرر لذرات هذا المركب في محاليل تختلف بالدالة الحامضية (PH= 4,6,8,13) حيث تبين أن التفاعل من الدرجة الثانية الكاذبة