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Research Article

PREPARATION OF COPPER, NICKEL AND ZINC (II) NANO-OXIDE BY USING ION COMPLEXES WITH THE LIGAND 2-AMINO-4-METHYL PYRIDINE

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ABSTRACT
This research studies the spectral and magnetic properties of two-nuclear ion complex Cu (II) and mononuclear complex Ni (II) and zinc ion (II) by using ligand-2 - amino-4 using methyl pyridine and copper nitrate salts of (II), nickel nitrate (II) and zinc nitrate (II) by synthesis method. For complex preparation, methanol is used in this study. The alcohol was used as solvent in order to give the metal a chance to appear as a multi-core alkoxide bridge. Complex synthesis is done in one step and using elemental analysis (CHN), infrared spectroscopy (FT-IR), ultra-violet-visible spectroscopy (UV-Vis) and magnetic properties, identification was done. After synthesizing complex by heating it at 800 °C for 2 hours, fragmented or isolated ligands from complex and metal ions were converted into nano-particles of copper oxide, nickel oxide and zinc oxide. After the synthesis of nanoparticles, identifying the shape and size of nanoparticles, by using infrared spectroscopy (FT-IR), X-ray diffraction (XRD) and scanning electron microscopy (SEM) is performed.

***Keywords**: Nanoparticles of Copper, Nickel and Zinc, Complex Thermal Decomposition, Identification of Nanoparticles, Techniques, X-Ray Diffraction (XRD), Techniques (SEM)*

ABSTRACT
The aim of this study is to produce nanoparticles of copper oxide, nickel and zinc by using bivalent complex and ligand 2-amino-4-methyl pyridine respectively. The method used in this research was thermal decomposition of prepared complexes and production of nanoparticles. Since, the metal oxide nanoparticles are used in various applications, producing them are very important. The metal oxide nanoparticles can be used in medicine, which uses metal oxide nanoparticles for different cancer treatments.
Nanotechnology is the science and technology that has attracted a lot of attention recently, this technology is a new approach in all fields, the ability to produce materials and new systems by manipulating atomic and molecular level.
This technology is used in medicine, biotechnology, materials, physics, mechanics, electricity, electronics and chemistry to the extent that it can be named as one of the great revolutions of the world. It's a new way to solve problems and answer the many questions raised in the various sciences that humanity has failed to resolve or respond to them.
For this reason, this technology as industrial and scientific revolution of the century are remembered. Since, the properties at the nanometer scale is changed favorably, nano-technology has opened a window to the material world which its production make it possible to build materials and equipment performance better (University of Medical Sciences, 2008).
The prefix nano is derived from the Greek word Nanus which means too small and can be used as a prefix for each unit such as seconds or liters and is meant a billionth (10^{-9}), so Nanotechnology will work in areas where dimensions in the nanometer range are used. Although a lot of articles are survived about nanotechnology, but little consensus about definitions related to the field of nanotechnology are presented.
In a recent report of the Royal Society and the British Academy of Engineering, the term of Nanotechnology is explained in this way: the design, characterization, production and application of structures, systems design and the use of controlled size and shape of materials at the nanoscale.

INTRODUCTION

The aim of this study was to produce nanoparticles of copper oxide, nickel and zinc using bivalent complex and ligand 2-amino-4-methyl pyridine respectively. The method used in this research complex thermal decomposition and the production of nanoparticles is prepared. Since, the metal oxide nanoparticles in various applications in the field of production of utmost importance. Including the use of metal oxide nanoparticles can be used in medicine, which uses metal oxide nanoparticles for cancer treatment is different. Nanotechnology is the science and technology that has attracted a lot of attention recently, this technology is a new approach in all fields, the ability to produce materials and new systems by manipulating atomic and molecular level has range. The application of this technology in medicine, biotechnology, materials, physics, mechanics, electricity, electronics and chemistry to the extent that it can be named as one of the great revolutions of the world. It's a new way to solve problems and answer the many questions raised in the various sciences that humanity has failed to resolve or respond to them yet. For this reason, this technology as industrial and scientific revolution of the XXI century is remembered. Since the properties at the nanometer scale is changed favorably, nano-technology has opened a window to the world for its production of building materials and equipment performance is more possible (University of Medical Sciences, 2008). The prefix Nano means dwarf which is derived from the Greek word Nanvs or too small and can be used as a prefix for each unit such as seconds or liters meaning a billionth (9-10) is the same, so Nanotechnology will work in areas where dimensions in the nanometer range. Although there is huge discussions about nanotechnology, but there is little consensus about definitions related to the field of nanotechnology there. In a recent report of the Royal Society and the British Academy of Engineering, which is defined Nanotechnology, Nano- technology is the design, characterization, production and application of structures, systems design and the use of controlled size and shape of materials at the nanoscale.

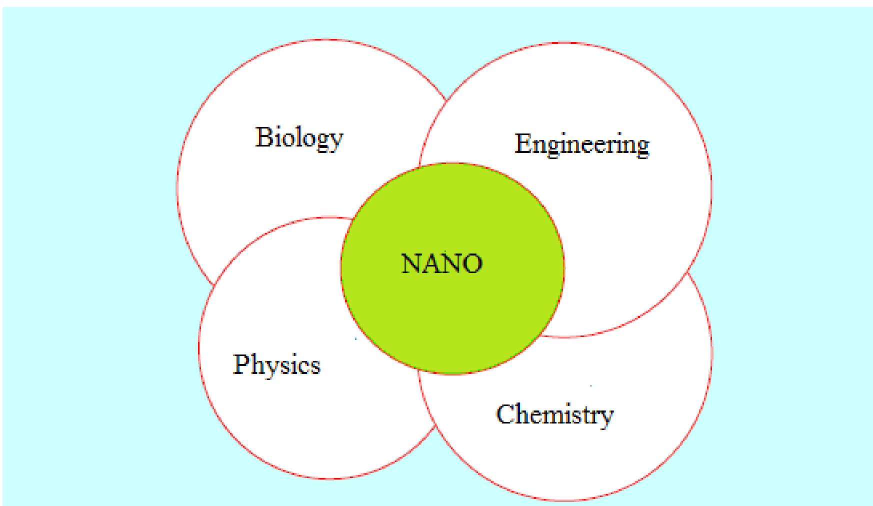

Figure 1: Shows the Relationship between Nanotechnology and Other Sciences

History of Nanotechnology

American physicist Richard Feynman proposed the idea of nanotechnology, because Richard Feynman contributions a lot of values to quantum electrodynamics (something far from nanotechnology), he had received the Nobel Prize in Physics, 1960 conference entitled. A lot of space down there "to discuss the capabilities and enables the production of nano-scale payment, Feynman proposed manipulate single atoms in order to build a new small structures with very different modes of action. Now this objective has been achieved by using a scanning tunneling microscope, and he conceived making the circuits on the nanometer scale (as of powerful computers), although Feynman thought was not reflected by scientists of that time, but now many of his assumptions convert to reality (Naderi, 2014). Two major events led to the

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rapid development of nanotechnology in the 1980s. The first invention of the scanning tunneling microscope in 1981, was the unrivaled clarity of atoms and bonds to provide separately and the second one was the Florin exploration in 1985. Today, nanotechnology is evolving at 4 generation and the evolution of it is the molecular system.

Some Applications of Nanotechnology

One of the applications of nanotechnology is in the food industry. Scientists and industrialists, use nanotechnology in the food industry in various sectors. For example, the food processing and food packaging products. Types of nanomaterials used in this industry. Include polymer Nano-composites impenetrable and silver and copper nanoparticles and Nano-composites and Nano-sensors. The use of this material is a useful way to prevent gas permeability and pathogens (Naderi, 2014).

Types of Nanomaterials in Terms of the Dimension

Nano materials can be classified into four groups according to the dimension:

1- Materials Zero-Dimensional (Quantum Dots): The material in all directions coordinate, length is very small, for example, atomic clusters with dimensions of 20 nm.

2- Materials dimensional (quantum wire) material only in line with the larger size of the unit, such as nano fibers only in the longitudinal axis of the unit are Fiber.

3- Two-dimensional (quantum wells) in both directions have a length of more than one unit, such as clay minerals that constitute the small plates.

4- The three-dimensional materials: materials in three dimensions, such as cluster size larger than the unit (Nourani, 2011)

Elemental Analysis Results

Analysis of the elements carbon, hydrogen and nitrogen, as well as theoretical and experimental results of atomic absorption measurements to calculate the percentage of copper, nickel and zinc complex are shown in the table (1-3).

Table 1: Results of Elemental Analysis and Theoretical Data

Row	**Complex**	**%C Experimental Theories**	**%H Experimental Theories**	**%N Experimental Theories**	**% Metal Experimental Theories**
1	$[Cu_2(L)_4(O\text{-}CH_3)_2(H_2O)](NO_3)_2$	39/05 40/57	5/55 5/17	17/51 17/06	15/89 14/94
2	$[Ni(L)_2(H_2O)_2](NO_3)_2$	33/13 34/53	4/63 5/86	19/32 19/61	13/49 13/97
3	$[Zn(L)_2(H_2O)_3](NO_3)_2$	32/63 33/46	4/56 4/39	19/02 19/93	14/8 15/67

According to the analysis of the elemental and the theoretical result we can see that complex No. 1 in methanol and sodium nitrate copper (II) three water, and is in 2 core situation and (OCH_3) is located as a bridge between the 2 core copper. Complex No. 2 in methanol and sodium nitrate, nickel (II) 6 water was prepared as a single core. Complex number 3 in methanol and the nitrate salt (II) 4 water was prepared as a single core.

In table 2, the complexes molecular weight are shown.

Table 2: Molecular Weight Complexes

Complex number 1	M_W= 763/56 gr
Complex number 2	M_W=434/ 97 gr
Complex number 3	M_W= 459/66 gr

Electron Spectrum

In these complex the transfers of π π* are shown due to aromatic ligands and because it ( ) is the alcohol solvent and also there are water molecules in the copper salt and the presence of amine groups on the heterocyclic ring ligand, cause n traffic control δ^* that we had a bit of intensity and also appear in a lower wave length.

Complex Electron Spectrum

A) Complex Number 1: [Cu_2 (L) $_4$ (O-CH_3)$_2$] (H_2O) (NO_3)$_2$

The characteristics of the whole d d 'ion Cu^{2+} is strip flat and low molar absorption intensity (Slavin, 1968).

The complex is a wide strip with λmax$_=$611 nm transfers in the visible spectrum dd can be seen. In the range of ultraviolet light in this complex the band with $\lambda_{max=611}$ nm is observed which is related to transfer σ^* n in the complex, and the band with λmax =365 nm of ligand molecules related to transfer n π* that is from features of dual-core copper compounds (II) with $Cu_2O_2N_4$, the band in λmax= 291 nm is related to the transfer π * ligand, this transfer is related to the ligand aromatic double bonds (Drago, 1977; Kida *et al.,* 1973; Mornet *et al.,* 2004; Gubin *et al.,* 2005).

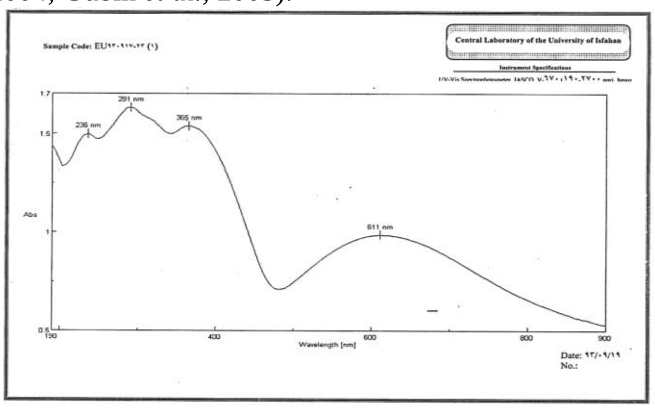

Figure 1: Number of Complex Electron Spectrum: [Cu_2 (L)$_4$(O-CH_3)$_2$](NO_3)$_2$(H_2O)

B) The Complex Number Two: [Ni (L)$_2$ (H_2O)$_2$] (NO_3)$_2$

In this complex we see two bands almost flat with low molar absorption rate that is related to the transfer d d in nickel, nickel element is d^8, which is the transmission of d to d like d^2. Tapes observed tapes with λmax=671 nm and λmax=753 nm is related to the transfer d to d. Full bar with λmax=389 nm is related to the transmission bar, the band with λmax=279 nm * π π ligands for charge transition and transfer band with λmax=389 nm is related to δ^* π in complex.

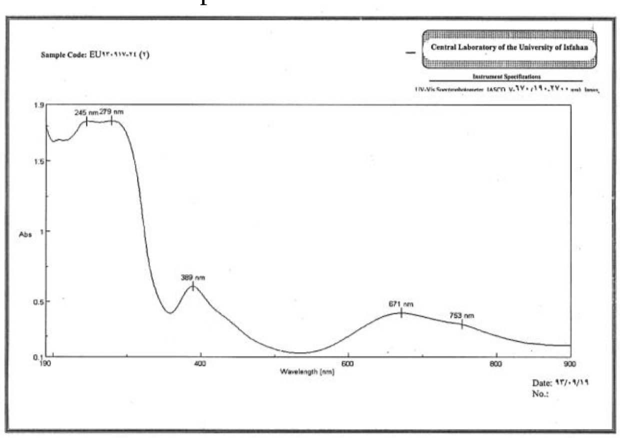

Figure 2: Complex Electron Spectrum II: [Ni (L)$_2$] (H_2O)$_2$ (NO_3)$_2$

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C) The Complex Number Three: [Zn $(L)_2$] $(NO_3)_2$ $(H_2O)_3$

The transfer d-d is not observed in this complex, because it does not have the zinc (Zn) d^{10} and transfers d-d, and band poorly observed λmax=390 nm related to the transfer of load (Charge Transfer), a Charge with λmax=214 nm transfer related to δ* n has less wavelength and more energy of other tapes. Band transfer λmax=299 nm related to * π π ligand is aromatic.

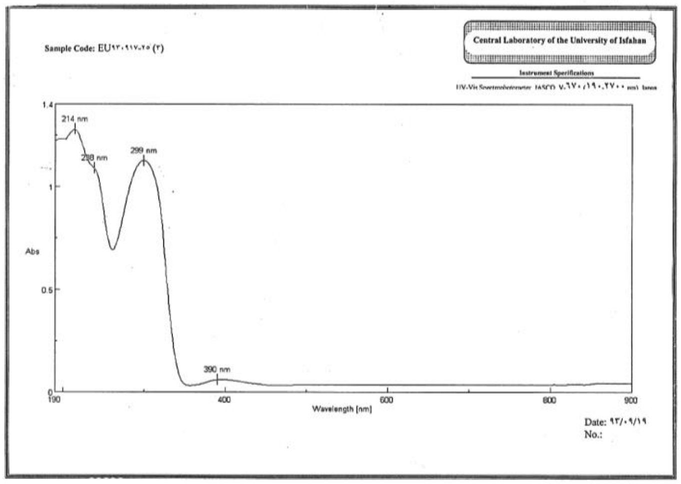

Figure 3: Electron Whole Complex Number Three: [Zn $(L)_2$] $(NO_3)_2$ $(H_2O)_3$

Review the Results of the Infrared Spectrum Complexes

In the infrared spectrum, the whole spectrum of complex and Ligand are compared to each other, the whole ligand complex spectrum in the range of 200-3500cm^{-1} are checked. For symmetric and non-symmetric stretching vibration, the ring Cu_2O_2 range of 440-540 cm^{-1} in some references and territories 440-575 cm^{-1} have been reported (Shimizu *et al.*, 2005).

The spectrum of the non-coordinate nitrates is reported 1100-1300 cm^{-1} and a single branch in the area 760 or 820 cm^{-1} and in some references the nitrate ion is 1328-1385 and 827-1051 cm^{-1} (Socrates, 2001; Baker, 2001).

In this technique by using compressed KBr pills, we try to identify complexes synthesized, first spectrum ligand 2 - methyl pyridine 4 - and then infrared spectrum is put under investigation.

Spectrum Ligand- 2 - Methyl Pyridine -4

Skeletal strip is obvious in the 1300-1600 cm^{-1} area. CH bending out of plane in the range of 690-900 cm^{-1} can be seen. 1100-1300 cm^{-1} peak can be related to bend plate and 3142 cm^{-1} peak can be relied to CH stretching and two peaks in the area of 3323cm^{-1} and 3421 cm^{-1} assigned to amino group. Peak C-N stretch in Area 1290 cm^{-1} has appeared. A peak appeared in the area 2872 cm^{-1} is related to symmetric stretching methyl group and the peak appeared in the area 2962 cm^{-1} is related to non- symmetric vibration stretching methyl group.

Checking the Infrared Spectrum Complexes

A) *Checking the Infrared Spectrum Complex Number One: [Cu2 (L) 4(O-CH3)2] (NO3)2 (H2O)*

In This complex, in addition to the peak of the ligand, delivery of symmetric and non- stretching vibration of the unit Cu_2O_2 is important. The peak of this area of 452-575 cm^{-1} to symmetric and non-symmetric stretching vibration ring Cu_2O_2 is attributed which is 512 and 560 cm^{-1} tape. Strip appeared in 400 cm^{-1} of the Cu-N, which is here in 432cm^{-1} .

B) Checking the Infrared Spectrum of Complex Two: [Ni $(L)_2$] $(H_2O)_2$ $(NO_3)_2$

In This complex, in addition to the peak of the ligand, complex peaks can also be seen. The single-core complex containing the metal-nitrogen bond and a metal-oxygen bond. Strip appeared in 400 cm^{-1} related to the metal-nitrogen bond which appeared here in 440 cm^{-1} and strip appeared in 640 cm^{-1} of the metal-oxygen bond.

C) Checking the Infrared Spectrum of Complex Three: [Zn (L)$_2$] (NO$_3$)$_2$(H$_2$O)$_3$

In This complex, in addition to the peak of the ligand, complex peaks can also be seen. The single-core complex containing the metal-nitrogen bond and a metal-oxygen bond. Strip appeared in 400 cm^{-1} related to the metal-nitrogen bond which appeared here in 436 cm^{-1} and strip appeared in 570 cm^{-1} of the metal-oxygen bond.

***Magnetic Properties of Complexes Built*:**

The magnetic properties of electrons can be achieved in two ways, one of rotational motion around its axis because negatively charged electrons in this way can produce magnetic (spin moment of the electron), and the other by the movement of electrons in their orbits core (electron orbital momentum). The magnetic moment of the electron is the result of these two properties. The unit of single magnetic moment is Burmagneton.

A) Copper Complex: The copper II is (^{9}1S ^{2} 2S^2 2P ^{6} 3S ^{2} 3P ^{6} 3d).

For compounds of copper (II) magnetic moment (spin only share), regardless of the type of link is BM / 731 but spin-orbital coupling gives higher levels 11. In the compounds of copper (II) sometimes less than 1/73 MB is reported that is the magnetic moment of the interaction between unpaired electrons on adjacent copper atoms. Magnetic behavior depends on high-spin ground state (spin parallel) or low-spin (spin anticlockwise) is ferromagnetic and antiferromagnetic order 12. An important feature of dimeric complex magnetic moment decreased as a result of direct interaction of metal-metal interactions through the bridge (Welz, 1987).

*B) Nickel Complex***:** Ni (II) electron configurations is 1S ^{2} 2S ^{2} 2P ^{6} 3S ^{2} 3P ^{6} 3d ^{8}.

Magnetic moment for Ni complex is 2/96 BM. The magnetic moment of copper is reported more than nickel that is due to the makeup d^8 nickel atom and nickel complex is a mononuclear complex.

*C) Copper Complex***:** Copper (II) electron configurations is 1S ^{2} 2S ^{2} 2P ^{6} 3S ^{2} 3P ^{6} 3d ^{10}.

Magnetic moment is not reported for copper complex because it is configured in d ^{10} and no unpaired electrons.

Table 3: Magnetic Moment Produced Complexes

Complex Number	**Magnetic moment B.M**
(1)	1/54
(2)	2/96

Checking the Infrared Spectrum of Nanoparticles:

A) The Infrared Spectrum of Nanoparticles of Copper Oxide (CuO):

The whole strip appeared in the 444/44 cm^{-1} and 500 cm^{-1} related to the stretching vibration of copper oxide (CuO). Absorption peak at 3314/82 cm^{-1} is related to the absorbed water by the nanoparticles.

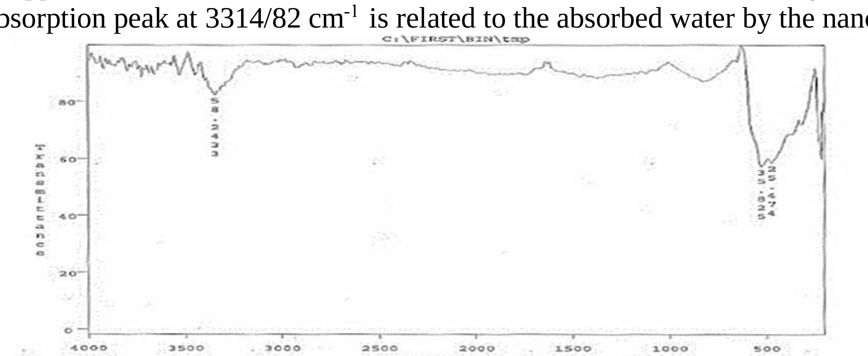

Figure 4: FT-IR Spectrum of Copper Oxide Nanoparticles

*B) Checking the Infrared Spectrum of Nickel Oxide (NiO)***:**

The whole strip appeared in the 444/44 cm^{-1} and 500 cm^{-1} related to the stretching vibration of nickel oxide (NiO). In these nanoparticles there is not absorption peak related to the absorbed water so their water is not absorbed.

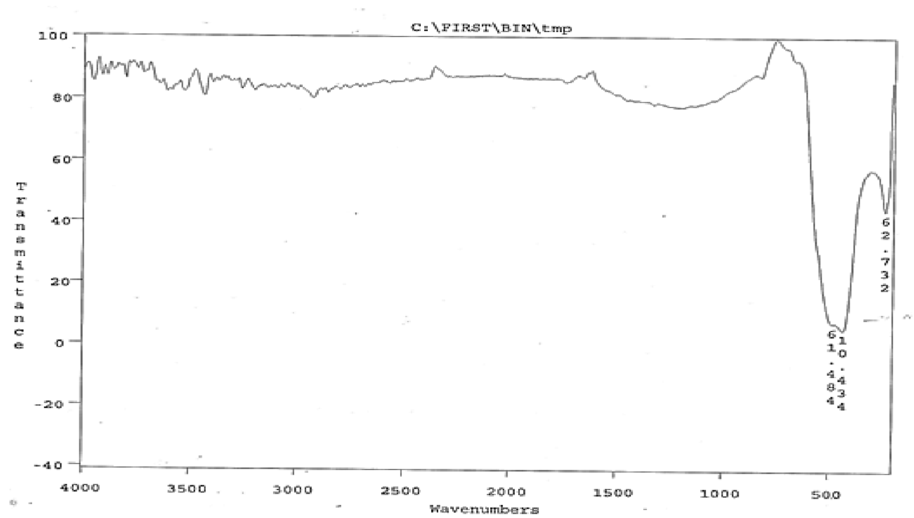

Figure 5: FT-IR Spectrum of Nickel Oxide Nanoparticles

C) Checking the Infrared Spectrum of Nanoparticles of Zinc Oxide (ZnO):
The whole strip appeared in the 500 cm^{-1} related to the stretching vibration (ZnO). Absorption peak at 3333/34 cm^{-1} is related to the absorbed water by the Nano-particles. Because of the water in the copper oxide and zinc oxide, the nanoparticles cluster structure is shown in the SEM images of the cluster areas.

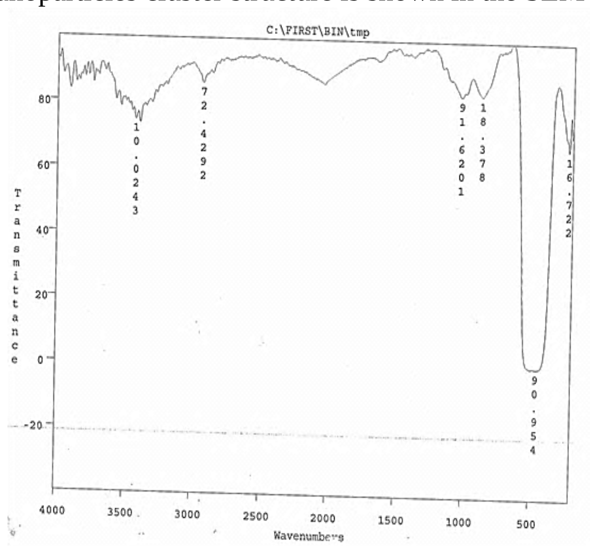

Figure 6: FT-IR Spectrum of Zinc Oxide Nanoparticles

***X-Ray Diffraction Pattern of Nanoparticles Made*:**
After preparing the characteristics of nanoparticles by diffraction techniques of (XRD) X and Fourier transform spectroscopy ((FT -IR and SEM Electron Microscope images were analyzed. The main peaks of copper oxide nanoparticles in 2θ of 35/63, 38/82, 48/83, 53/57, 58/39, 61/61, 66/31, 68/16 is appeared, the peak with the greatest intensity is observed in 38/82 = 2 θ, the width at half height for the peak is at 0/1968. Given the Peak width at half height by using the Scherer relationship D = $\frac{K\lambda}{\beta cos\theta}$ the size of the particles were calculated, θ is an angle to the peak with the greatest intensity and λ is the wavelength of the radiation, and k is a constant that is dependent on particle shape and its value to spherical particles 0/9, β is the peak width at half height. The copper oxide nanoparticles, the average particle size is 42/31

nm. In the diagram of x-rays of each peak represents a particular matter there is no double materials that have the same XRD patterns, like a fingerprint, and the number of pages in the crystal is more, the peak intensity of that is more, for example, in the pattern of copper oxide nanoparticles No. : (111) are the most common resulting in the peak intensity so the rest of the pages on this page is the most, in addition to full intensity of the peaks indicate the crystalline nature of the sample appropriate **[54].** The XRD patterns of the major nickel oxide nanoparticles peak in 2θ of 92/36, 62/43 and 75/42 appeared in 2θ=43/36 peak with the greatest intensity is observed, the width at half height for this is the peak of 0/1968. Nickel oxide nanoparticles in the size of the particles is averaged 42/51 nm.

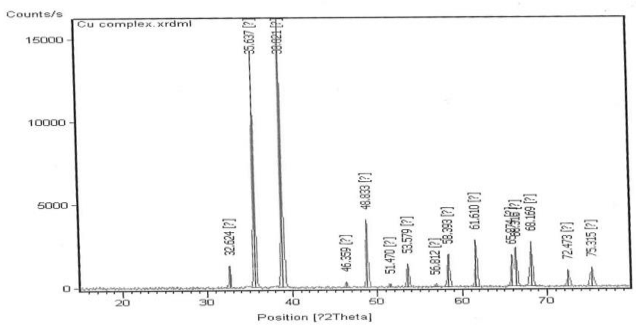

Figure 7: XRD Pattern of Copper Oxide Nanoparticles

The pattern of nickel oxide nanoparticles peak to the (111) is the most intense, resulting in No.: (111) crystal system is nickel oxide nanoparticles.

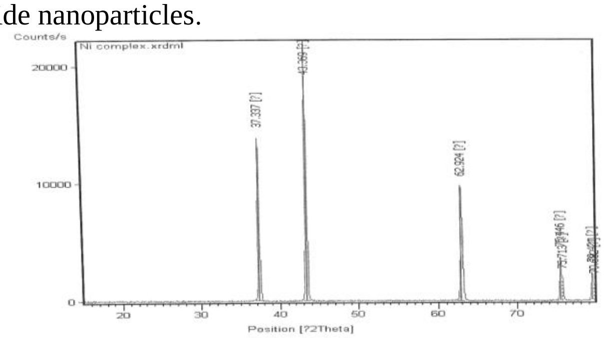

Figure 8: XRD Pattern of Nickel Oxide Nanoparticles

The pattern of XRD nanoparticles the most peaks in θ2 are 31/86, 36/52, 36/34, 47/62, 56/66, 62/91, 66/42, 67/99, 69/13 . In 2θ =36/34 the peak with the most intensity is observed. The width in half hight for the peak is 0/1476. In nanoparticles oxide copper, the size of particles are 53/58 nm. The pattern of zinc oxide nanoparticles to the peak (101) is the most intense, resulting in No. : (101) crystalline nanoparticles of zinc oxide in the system.

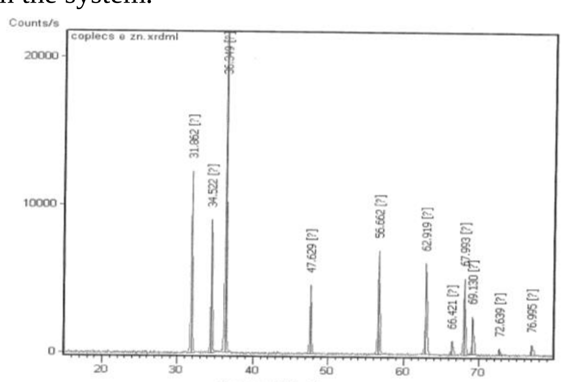

Figure 9: XRD Pattern of Zinc Oxide Nanoparticles

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The results of X-ray diffraction is shown in the following table:

Table 4: XRD Data for Copper Oxide Nanoparticles

Position Peaks (θ2)	Peak Width at Half Height (FWHM)	Specific Surface Area (SSA)	Reflection Pages	Particle Size (D) nm	The Distance between the Plates (d) Å	Fixed Network Å (a)
35/6375	0/1968	15/68	[111]	42/84	2/5173	4/3601
38/8210	0/1968	15/54	[111]	43/24	2/3177	4/0144
48/8325	0/2460	18/97	[202]	35/41	1/8636	5/2711
53/5791	0/2460	18/60	[020]	36/12	1/7091	3/4182
58/3932	0/2460	18/19	[202]	36/94	1/5791	4/4664
61/6105	0/2952	21/65	[113]	31/04	1/5042	4/9889
66/3147	0/1476	10/55	[310]	63/70	1/4084	4/4538
68/1690	0/1968	13/65	[220]	49/24	1/3746	3/3880

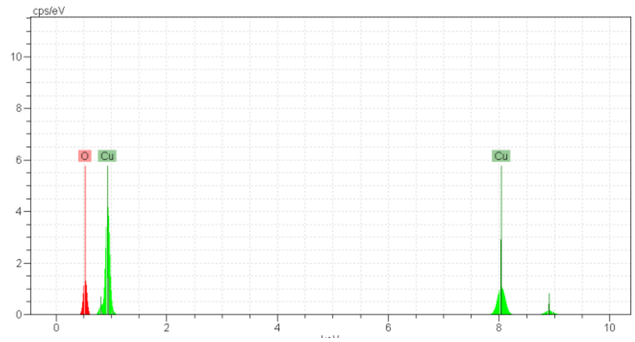
Table 5: XRD Data for Nickel Oxide Nanoparticles

Position Peaks (θ2)	Peak width at Half Height (FWHM)	Specific Surface Area (SSA)	Reflection Pages	Particle Size (D) nm	The Distance between the Plates (d) Å	Fixed Network Å (a)
43/3685	0/1968	20/50	[111]	43/89	2/0875	2/5025
62/92411	0/3444	33/21	[200]	27/09	1/4758	4/0144
75/4460	0/1800	15/91	[220]	56/55	1/2590	3/1736

Table 6: XRD Data for Zinc Oxide Nanoparticles

Position Peaks (θ2)	Peak Width at Half Height (FWHM)	Specific Surface Area (SSA)	Reflection Pages	Particle Size (D) nm	The Distance between the Plates (d) Å	Fixed Network Å (a)
31/8625	0/1476	19/28	[100]	55/46	2/8062	2/8062
34/5218	0/1968	25/05	[002]	42/70	2/5967	5/1924
36/3486	0/1476	19/05	[101]	56/13	2/4697	3/4927
47/6285	0/1968	24/00	[102]	44/57	1/9079	4/2662
56/6625	0/2460	29/02	[110]	36/63	1/6232	2/2956
62/9186	0/2460	28/29	[103]	37/80	1/4758	4/6669
66/4205	0/1476	16/78	[200]	63/74	1/4064	2/8128
67/9935	0/1476	16/63	[112]	64/32	1/3776	2/8750
69/1300	0/1476	16/52	[201]	64/76	1/3577	3/0359
72/6395	0/1476	16/16	[004]	66/19	1/3005	5/2020
76/9952	0/1800	18/71	[202]	57/15	1/2374	3/4999

***Checking EDAX Spectrum of Prepared Nanoparticles*:**
To prove the existence of metallic oxide nanoparticles and investigate the possible impurities EDAX analysis was performed on samples. EDAX spectrum of the nanoparticles prepared to the conclusion that nanoparticles are pure and contains elements of metal and oxygen. The whole of each of the elements specified in addition there is no unwanted element in nanoparticles.

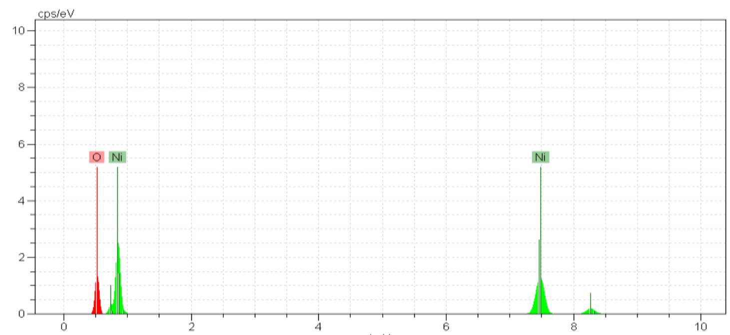


Spectra: 1-CUO
Element Series unn. C norm. C Atom. C
[wt.-%] [wt.-%] [at.-%]

Oxygen K series 9.95 9.67 29.83
Copper K series 92.99 90.33 70.17

Total: 102.9 %

Figure: 10 EDAX Spectrum of Copper Oxide Nanoparticles



Spectra: 2-NIO
Element Series unn. C norm. C Atom.
[wt.-%] [wt.-%] [at.-%]

Oxygen K series 10.39 11.45 32.18
Nickel K series 80.34 88.55 67.82

Total: 90.7 %

Figure (11) EDAX Spectrum of Nickel Oxide Nanoparticles

Spectra: 3-ZNO
Element Series unn.C norm. C Atom. C
[wt.-%] [wt.-%] [at.-%]

Oxygen K series 12.30 13.05 38.03
Zinc K series 81.90 86.95 61.97

Total: 94.2 %

Figure 12 EDAX Spectrum Zinc Oxide Nanoparticles

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