Development of down flow hanging sponge reactor as a new process and technology for sewage sludge treatment

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ABSTRACT

There are numerous sewage treatment techniques to select from, ranging from decentralized systems to big centralized systems that involve a network of pipes and pump stations that transport sewage sludge to a treatment plant. Sewage sludge treatment is a sort of waste water treatment that tries to remove contaminants from sewage sludge in order to generate effluent appropriate for discharge to the surrounding environment of an intended reuse use, preventing water contamination from raw sewage discharges. The presence of organic, inorganic, and biological contaminants in sewage sludge has a number of environmental and health consequences. Human and animal exposure to hazardous pollutants through ingestion of food and drinks has serious consequences, including impaired animal growth and development, cancer sickness, organ damage, nervous system damage, and, in extreme circumstances, death. To alleviate the detrimental effects of hazardous contaminants on human, animal, and environmental health, a range of remediation procedures have been developed. Chemical and biological remediation procedures have been shown to be particularly effective in the treatment of hazardous contaminants in sewage sludge. A novel technology involves combining a new amino organic ligand with sewage sludge under various conditions such as concentration and time, and then passing the sewage through a modified Bio-active Sorbent System. The primary goal of this study was to remove infamous heavy metals and fecal coliforms utilizing a hydrazide ligand. Cu(II),Mn(II), Zn(II), Cd(II), Fe(II), Pb(II), and Cr(II) were the metal ions. To begin, the experiment was carried out in a laboratory at 25C^{o} utilizing sewage metal ions and varied concentration ratios of the ligand (L) and metal salts (M) like [1 L: 2M] and [1 L: 5M]. The results showed that raising the concentration of the ligand to the metal salts improved the efficacy of heavy metal and bacterial elimination. At [1 L:2M] molar ratio and 25 C^{o}, the removal efficiency of heavy metals was in the range of [10% - 22%] after 30 minutes and increased to [20% - 45%] after 60 minutes. After 30 minutes, the removal efficiency increased to (12%-19.5%) and reached (21%-38%) after 60 minutes. Furthermore, utilizing dose 3.0

mg, the results of the Bio-active Sorbent System revealed that heavy metal content reduces as the dose increases. At a dose of 1.0 mg.L^{-1}.Cu concentration reduced from 4.25 to 0.64 mgL^{-1} (85.0%). The Cr concentration dropped from 5.24 to 0.35 mgL^{-1} (95.0%). However, the Mn concentration fell from 2.25 to 0.43 mgL^{-1} (80.9%) and the Cd concentration fell from 4.02 to 1.19 mgL^{-1} (70.0%). The Zn concentration fell from 1.87 to 0.32 mgL^{-1} (82.0%). Using 3.0 mgL^{-1} as well The Cu concentration was reduced from 4.25 to 0.1 mgL^{-1} (86.0%). The Cr concentration dropped from 5.24 to 0.29 mgL^{-1} (95.0%). Mn concentration fell from 2.15 to 0.41mgL-1 (88.0%). However, the Fe concentration dropped from 5.01 to 1.15 mgL^{-1} (77.4%) while the Cd concentration dropped from 4.3 to 1.1 mgL^{-1} (85.0%). The Pb concentration dropped from 187 to 0.21mgL^{-1} (89.0%). The Zn concentration dropped from 1.87 to 0.04 mgL^{-1} (98.0%). Finally, the number of fecal coliforms was reduced from 5.4x106 to 3x103and (99.9%).

Keywords*: Organic ligand ,* Bio-active Sorbent System, removal of *notorious heavy metal, fecal coliforms.*

1. INTRODUCTION

Treatment of sewage sludge (or biosolids) is the residue that collects in sewage treatment plants. The solid, semisolid, or slurry leftover material is sewage sludge. The semi-solid residue left over from industrial waste water or sewage treatment methods is known as sludge. It can also refer to the settled suspension created by traditional drinking water treatment and a range of other industrial processes. The phrase is widely used as a generic name for solids withdrawn from suspension in a liquid; this soupy mixture frequently contains significant volumes of interstitial water (between the solid particles) [1]. In 1992, the United States imposed a ban on ocean dumping of human sewage sludge, leaving towns with the costly option of disposing of sewage waste in landfills [2].Given that sewage contains more than only heavy metals[3] and microorganisms like Clostridium difficile[4], Corporations like Synagro adapted the nutrient concept and offered sludge to farmers as "bio-solids" as a free fertilizer. **[5].**Organic matter, which accounts for roughly half of the solid part of most sludge, enhances the physical state of soils**[6 - 9]**.Increased organic matter content reduces bulk density **[7] ,** Increases aggregate stability **[7,10,11**], water holding capacity **[7,9,12- 14]**, and water infiltration **[7,8, 15]**.Improving soil physical characteristics boosts soil productivity **[8]**. Higher productivity results from an increase in the amount of water available to crops in coarse-textured soils. Productivity increases in fine-textured soils because aggregation of soil particles enhances infiltration, porosity, and aeration. The enhancement of soil productivity by sewage sludge treatment has been demonstrated dramatically in tests utilizing severely disturbed soils **[16-18]**, The term pollutant is defined in the EPA 503 rule. Pollution limitations for sludge components have been established by the EPA. A pollutant is an organic substance, an inorganic substance, a combination of

organic and inorganic substances, or a pathogenic organism that, after discharge and upon exposure, ingestion, inhalation, or assimilation into an organism either directly from the environment or indirectly through ingestion through the food chain, could, based on information available to the EPA Administrator, cause death, disease, behavioral abnormalities, cancer, genetic mutations, phlegm, or phlegm production. Trace elements are defined as elements that occur in trace amounts in natural systems yet are toxic to living organisms when present in excessive concentrations. **[20] .** The plant soil system includes three defensive systems that might limit potentially dangerous trace elements in a plant's aerial portions, hence reducing health issues in animals or humans. Thousands of different sludge components that are untested/undetectedly dumped from modern society and end up in sludge (pharmaceuticals, micro particles, etc.) have been proved to be harmful to both human and environmental health **[21].** The application of sewage sludge to land can have a variety of beneficial impacts. Several benefits of land application of municipal sewage sludge include providing nutrients (N, P, secondary nutrients, and micronutrients) to crops, enhancing soil physical qualities, and raising soil organic matter content. Although these are evident advantages, there are certain issues that must be handled. The most frequently expressed concerns are: 1) the potential for soil, plant, animal, and human damage as a result of toxic metal treatments; and 2) the potential for disease transfer. The use of sewage sludge might cause a shift in soil pH. When municipal sewage sludge was added to soils, the pH increased **[22, 23]**. Metals can be found in various forms in municipal sewage sludge. Their shape is determined by the chemical characteristics of the sludge as well as the chemistry of the metal **[24, 25]**. Cadmium concentration rose in plant tissue when soil Cu levels increased **[26]**; the amount of the rise was also controlled by Cr, Cd, and Zn levels. These interactions are exceedingly intricate and little is known about them. Other metals' presence and concentrations can have a significant impact on the absorption of the metal in question **[27]**. In 2009, the Environmental Protection Agency (EPA) released the Targeted National Sewage Sludge Study, which reports on the quantities of metals, chemicals, hormones, and other constituents in a statistical sample of sewage sludge. Among the highlights are the following: On average, silver is present to the extent of 20 mg/kg of sludge, a near economically recoverable level; nevertheless, some sludge of extremely high quality includes up to 200 milli-grammes of silver per kilo-gramme of sludge; one outlier demonstrated a silver lode of 800-900 mg per kilo-gramme of sludge. According to the EPA, lead, arsenic, chromium, and cadmium are present in quantifiable concentrations in 100% of national sewage sludge in the United States, although thallium is present in only 94.1% of sludge **[28]**. Chemicals are more sensitive to surface erosion than infiltration because they stay adsorbed to surface soil particles **[29].**

2.Materials and Instruments

Chemicals : All chemicals used to prepare the ligand and its metal complexes were of analytical quality and were utilized without additional purification. Sigma-Aldrich Company offered metal salts. Sigma-Aldrich Company also provided diethyl oxalate (Assay 99.99%), proplenediamine (Assay 98%), H2SO4 (Assay 99.7%), and ethanol (Assay 99.8%).

Instrumentation and measurements: The ligand and its metal complexes were tested for C, H, N, and M at Cairo University's Micro Analytical Centre. Metal ion content was determined using standard analytical procedures **[11]**. The ^{1}H-NMR spectra were obtained using a Bruker 400 MHz spectrometer. TMS is used to express chemical shifts (ppm). The FT-IR spectra of the ligand and its metal complexes were measured using KBr discs and a Jasco FT/IR 300E Fourier transform infrared spectrophotometer. Electronic spectra in the 200-900 nm region were recorded using a Perkin-Elmer 550 spectrophotometer. Thermal analyses (DTA and TGA) were carried out using a Shimadzu DT-30 thermal analyzer at a heating rate of 10 Co/min from room temperature to 800 Co. Magnetic susceptibilities were calculated.

$$(\mu \text{ eff} = 2.828 (\chi n \times T) 1/2) \qquad (1)$$

Using a Bibby conductometer type MCl, the molar conductance of 10^{-3} M complex solutions in DMSO was measured at 25C^{o}. The ESR spectra of solid complexes at room temperature were recorded using a Varian E-109 spectrophotometer, with DPPH serving as a reference material. TLC was used to certify the purity of each component.

Laboratory Preparation: The ligand (H_3L) was made by heating (10.0 g, 1.4 mol) Diethyl oxalate (10.0 g, 0.6mol) in 50 cm^3 of ethanol solution for two hours in the presence of 5 drops Conc H_2SO_4. Allow to cool to room temperature to produce Diethyl oxalate ester, which was then mixed with (8.0 g, 0.4 mol) Proplene diamine. The solution was allowed to cool at room temperature after being refluxed for two hours at 80 C with stirring. The precipitated product was filtered out and dried in air or under vacuum over anhydrous $CaCl_2$ (Figure 1), and metal complexes were formed using ligand (1L:2M) or (1L:5M).

2.1. Bio-active Sorbent System:

As shown in Scheme (1), the Bio-active Sorbent module column is made up of four comparable segments linked vertically, each containing 25 L of polyurethane foam (PF) warped with plastic material randomly distributed throughout the reactor. The PVC Bio-active Sorbent System has a capacity of 0.3 m3 and an internal diameter of 0.16 m. The reactor is 0.88 metre above ground. The reactor was filled with PF, accounting for 34% of the total

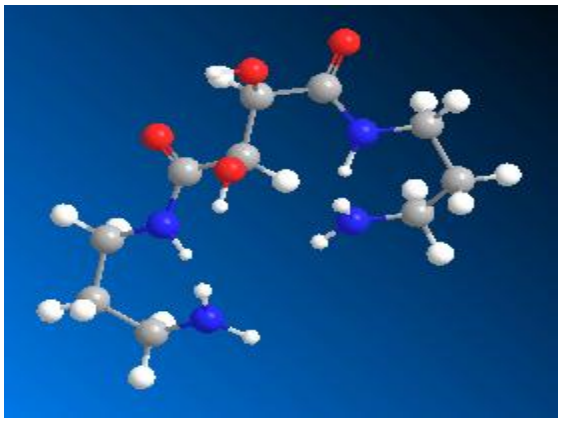
liquid reactor capacity. The PF (sponge) has the following parameters: surface area 256m^2/m^3, density 30 kg/m3, void ratio 0.9, and pore size 0.63 mm. The total capacity of the PF will be 100 L. (PF) (cylindrical) dimensions were employed for the sponge. Gravity fed wastewater effluent to the distributor, which is located on top of the Bio-active Sorbent module and rotates at 15 rpm. Chemical parameters such as COD, BOD, TSS, TDS, NO_2 and NO_3 will be assessed after a 3-hour retention time in compliance with the American Public Health Association's "Standard Methods for the Examination of Water and Wastewater." **[30].**

Scheme (1):show the sponge reactor.

Synthesis of the organic Ligand.

Diethyloxalate (10.0 g, 0.6mol) was heated in ethanol for 1 hour before adding five drops of conc. H2SO4 to the reaction solution, which was then refluxed with constant stirring for 3 hours at 60-70 C^o. Propalenediamine (4.27 g, 1.33mol) was added. The mixture was refluxed for another 2 hours with stirring, and the yellowish precipitate that developed was filtered off, washed with ethanol, and dried in air.

The structure of the (Ligand) and 3D are shown in chart (1).



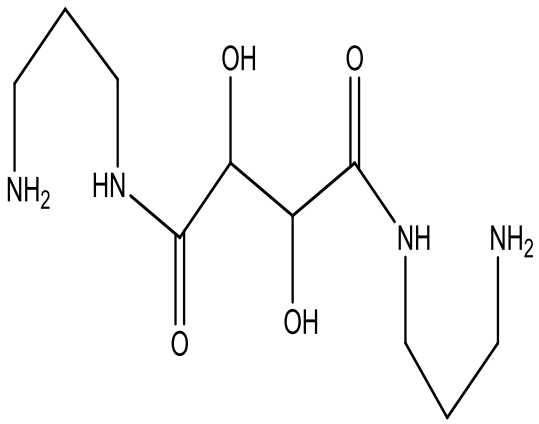


Fig (1): Molecular and 3D structure of the hydrizde ligand (1).

Ligand (1): Chemical Formula: $C_{10}H_{22}N_4O_4$, Molecular Weight: 262, Elemental Analysis: Calc: C, 45.8, H, 8.39, N, 21.7; Found (%): C, 45.23, H, 8.11, N, 20.98

IR Spectrum: υ(H-Bond) = 3580 - 3200 Cm^{-1},3190 - 2560 Cm^{-1},υ(OH) = 3412 , 3375 Cm^{-1},υ(C=O)= 1665,1620 Cm^{-1},υ(NH)= 3185 Cm-1,υ(CH_2)= 3120,2890 Cm^{-1},υ(NH_2)= 3310,2890 Cm^{-1} ,υ(OH) = 1330,1285 Cm^{-1}

Mass spectrum: H_4N_2,M/Z= 32 amu ,$C_3H_{10}N_2$,m/z=74 amu , $H_6N_2O_4$, m/z=98amu , $C_5H_{11}N_2O_2$,m/z=131 amu , $C_7H_{13}N_2O_2$,m/z=189 amu , $C_{10}H_{22}N_4O_4$, m/z=262 amu

UV-vis : 290 nm , 315 due to $\pi\rightarrow\pi^*$,$n\rightarrow\pi^*$ transitions

^{1}**H-nmr** (deuterated DMSO) : NH = 7.5 ppm , NH2= 4.7 ppm , CH_2 group 2.72 -3.35 ppm , CH 2.1 ppm , OH 5.3 ppm

Complex (1) (1L: 2 M), $Cu(OAc)_2$ .H_2O

Chemical Formula: $C_{26}H_{60}N_4O_{27}Cu$, M.Wt : 680,Coulour : Brown ,M.P : >300 C ,Cond : 9.3 Ω^{-1} $mol^{-1}Cm^{-1}$, Elemental Analysis: Calc: C, 31.76 ,H, 5.88 , N, 8.23, Cu,18.6; Found (%): C, 32.2, H, 5.1 , N, 7.82 , Cu,18.12 ,

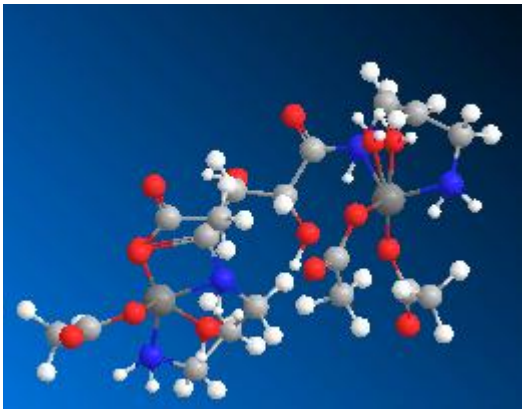
IR Spectrum : υ(H_2O) = 3530 - 3330 Cm^{-1},3320-3150 Cm^{-1},υ(H-Bond)= 3600 - 3320 Cm^{-1},3310 - 2820 Cm^{-1},υ(OH) = 3470,3410 Cm^{-1},υ(C=O)= 1635,1615 Cm^{-1},υ(NH) = 3270 Cm^{-1},υ(NH_2) = 3376, 3350 Cm^{-1},υ(OAc) = 1511,1410 Cm^{-1},υ(OH)= 1350,1295 Cm^{-1},υ(Cu O)= 628 Cm^{-1} , υ (Cu-N)=590 Cm^{-1}- **μ** eff : 1.69 B.M

mass spectrum:

H_2N_2,M/Z= 32 amu ,$C_3H_{10}N_2$,m/z=74 amu , $H_6N_2O_4$, m/z=98amu , $C_5H_{11}N_2O_2$,m/z=131 amu , $C_7H_{13}N2O_2$,m/z=189 amu , $C_{10}H_{22}N_4O_4$, m/z=262 amu and $C_{10}H_{22}N_4O_4Cu_2$, m/z=388 amu

UV-vis : 292 nm , 318 , 735 , 575 , 610 nm due to $\pi\rightarrow\pi^*$,$n\rightarrow\pi^*$ and d-d transitions .

ESR : g11=2.12 , g1 =2.06 , giso =2.08 - Octahedral structure with covalent bond character



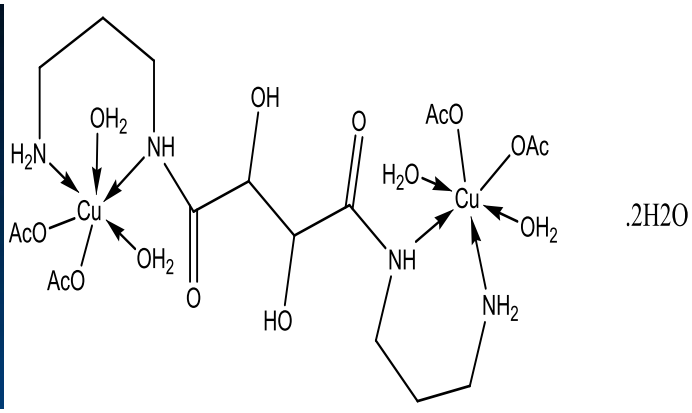


Fig (2): Molecular and 3D Structure of the complex (1).

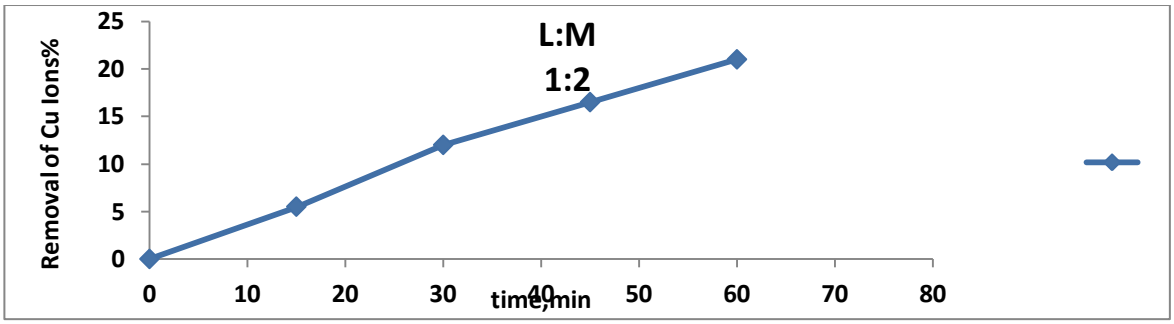


Chart (1) removal Cu ions from sewage sludge at time

At 30 C	(1L : 2M)
15 min	8%
30 min	16 %
45 min	19%
60 min	24 %

Complex (2) (1L : 5 M) , $Cu(OAc)_2$ .H_2O

Chemical Formula: $C_{26}H_{60}N_4O_{27}Cu_4$,M.Wt : 1112,Coulour : yellowish Brown ,M.P : >300 C ,Cond : 13.72 Ω^{-1} $mol^{-1}Cm^{-1}$, Elemental Analysis: Calc: C, 28.06, H, 5.39, N, 5.04, Cu,22.7; Found (%): C, 27.62, H, 5.1 , N, 4.82Cu,22.35 ,

IR Spectrum :

υ(H-Bond)= 3550 - 3320 Cm-3310 - 2780 Cm^{-1},υ(OH) = 3445 , 3400 Cm^{-1},υ(C=O)= 1638,1626 Cm^{-1},υ(NH)= 3200 Cm^{-1},υ(CH_2)= 3120,2890 Cm^{-1},υ(NH_2)= 3380,2890 Cm^{-1},υ(OH) = 1325,1295 Cm^{-1},

υ(OAc) = 1456,1340Cm^{-1},

μ eff 1.53 B.M

Mass spectrum :

$C_3H_{10}N_2$,m/z=74 amu , $H_6N_2O_4$, m/z=98amu , $C_5H_{11}N_2O_2$,m/z=131 amu , $C_7H_{13}N_2O_2$,m/z=189 amu , $C_{10}H_{22}N_4O_4$, m/z=262 amu ,$C_{10}H_{22}N_4O_4Cu_3$, m/z=451 $C_{10}H_{22}N_4O_4$ Cu_5, m/z=577 amu

UV-vis : 295 nm , 320 , 475 , 605 nm due to $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$ and d-d transitions .

ESR g_{iso} : 2.11

Covalent bond character with Octahedral geometry

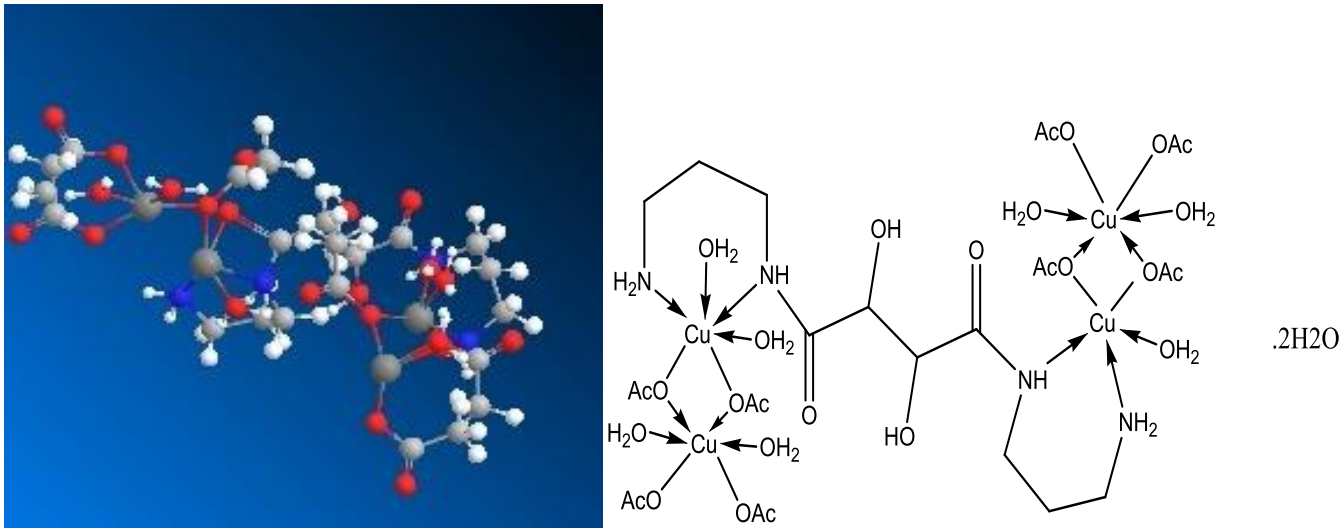
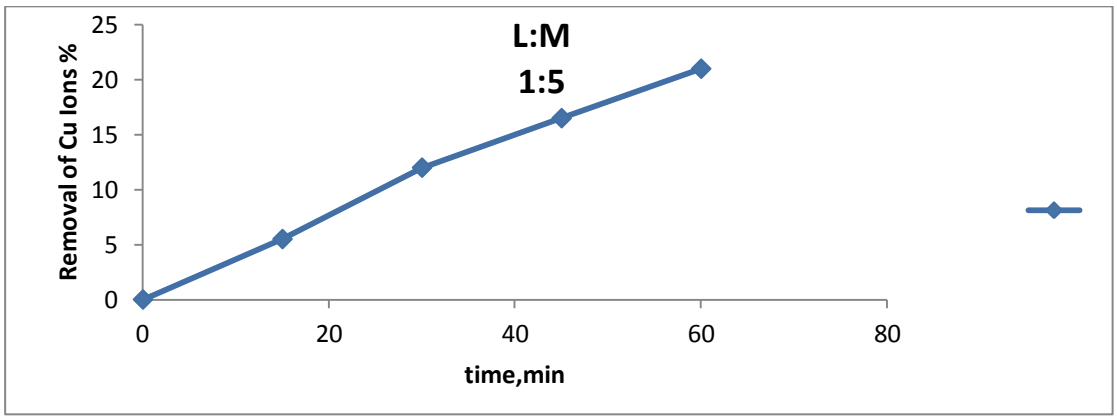


Fig (3): Molecular and 3D structure of the complex (2)



Removal % of Cu ions

Chart (2) removal Cu ions from sewage sludge at time

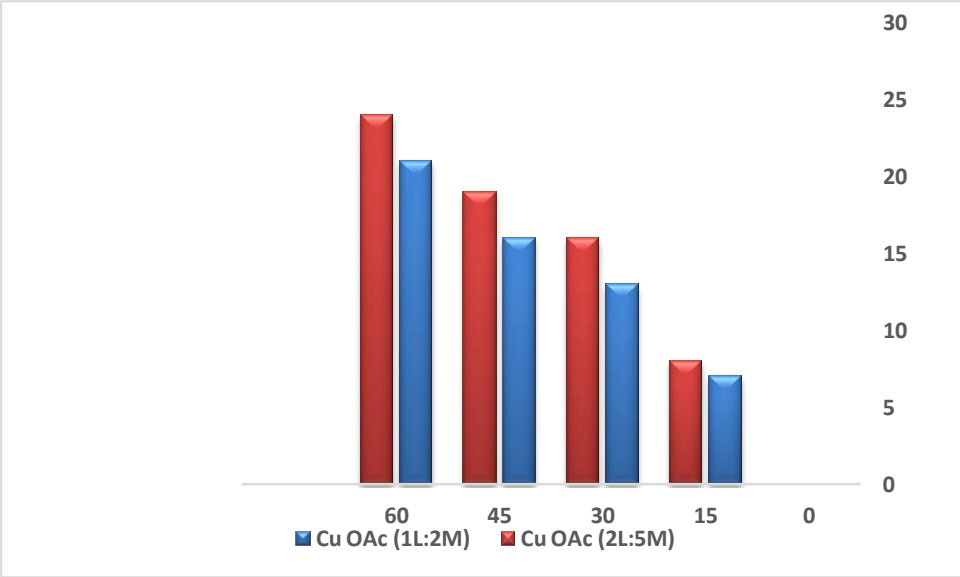
At 30 C (1L : 5M)

15 min 7%

30 min 13 %

45 min 16%

60 min 21 %



Complex (3) (1L : 2 M) , Cu(SO4) .5H2O

Chemical Formula: $C_{10}H_{32}N_4O_{17}S_2Cu_2$,M.Wt : 672,Colour : yellowish Brow n, M.P : >300 C ,Cond : 9.3 $\Omega^{-1}mol^{-1}Cm^{-1}$, Elemental Analysis: Calc: C, 17.86 , H, 4.76, N, 8. 3, Cu,18.84; Found (%): C, 17.2, H, 4.45 , N, 7.82 , Cu,18.46 ,

IR Spectrum: υ(H2O) = 3530 - 3320 Cm^{-1},3320-3150 Cm^{-1},υ(H-Bond)= 3600 - 3320 Cm^{-1},3310 - 2820 Cm^{-1},υ(OH) = 3470,3410 Cm^{-1},υ(C=O)= 1635,1615 Cm^{-1},υ(NH) = 3270 Cm^{-1},υ(NH_2) = 3376, 3350 Cm^{-1},υ(SO_4) = 1240,1140 ,730Cm^{-1},υ(OH)= 1350,1295 Cm^{-1},υ(Cu O)= 628 Cm^{-1}

μ eff 1.68 B.M

Mass spectrum:

H_2N_2,M/Z= 32 amu ,$C_3H_{10}N_2$,m/z=74 amu , $H_6N_2O_4$, m/z=98amu , $C_5H_{11}N_2O_2$,m/z=131 amu , $C_7H_{13}N_2O_2$,m/z=189 amu , $C_{10}H_{22}N_4O_4$, m/z=262 amu, $C_{10}H_{22}N_4O_4S_2$, m/z=326 amu and $C_{10}H_{22}N_4O_4S_2Cu_2$, m/z=452 amu

UV-vis : 295 nm , 319 , 435 , 560 , 603 nm due to $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$ and d-d transitions .

H-nmr (deuterated DMSO) : NH = 7.5 ppm , NH2= 4.7 ppm , CH2 group 2.72 -3.35 ppm , CH 2-1 ppm , OH 5.3 ppm

ESR giso : 2.09 -Octahedral structure with covalent character

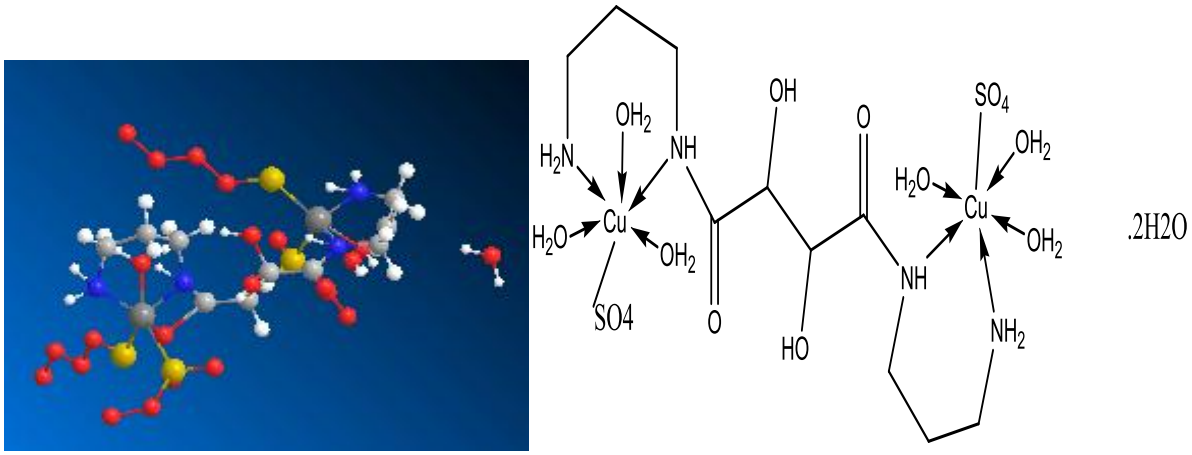


Fig (4): Molecular and 3D structure of the complex. (3)

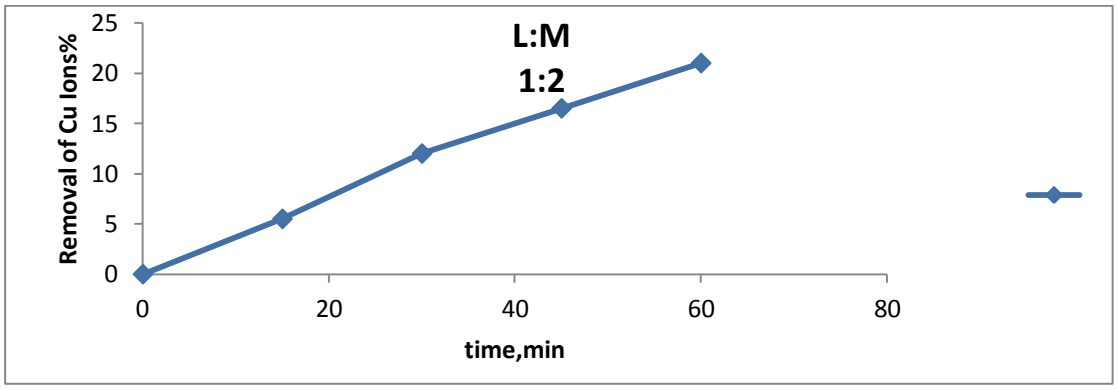


Chart (3) removal Cu ions from sewage sludge at time

At 30 C (1L : 2M)

15 min 6.5%

30 min 12 %

45 min 16%

60 min 23 %

Complex (4) (1L : 2 M) , $Cu(NO3)_2 .3H_2O$

Chemical Formula: $C_{10}H_{30}N_8O_{20}Cu_2$,M.Wt : 710,Colour : Brown ,M.P : >300 C ,Cond : 8.4 Ω^{-1} $mol^{-1}Cm^{-1}$, Elemental Analysis: Calc: C, 16.9, H, 4.22, N, 15.8, Cu,17.83 ; Found (%): C, 16.31, H, 4.0 , N, 15.56 , Cu,17.56 ,

IR Spectrum: $\upsilon(H_2O)$ = 3530 - 3320 Cm^{-1},3320-3150 Cm^{-1},υ(H-Bond)= 3600 - 3320 Cm^{-1},3310 - 2820 Cm^{-1},υ(OH) = 3470,3410 Cm^{-1},υ(C=O)= 1635,1615 Cm^{-1},υ(NH) = 3270 Cm^{-1},$\upsilon(NH_2)$ = 3376, 3350 Cm^{-1},$\upsilon(NO_3)$ = 1380,1160,1074,820 Cm^{-1},υ(OH)= 1350,1295 Cm^{-1},υ(Cu O)= 628 Cm^{-1}

μ eff 1.17 B.M

Mass spectrum:

$C_3H_{10}N_2$,m/z=74 amu , $H_6N_2O_4$, m/z=98amu , $C_5H_{11}N_2O_2$,m/z=131 amu , $C_7H_{13}N_2O_2$,m/z=189 amu , $C_{10}H_{22}N_4O_4$, m/z=262 amu , $C_{10}H_{22}N_4O_4Cu_2$, m/z=388 amu

UV-vis :296 nm , 317 , 456 , 580 , 605 nm due to $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$ and d-d transitions .

ESR **g11** = 2.14 **g**1 = 2.05 and **g**iso = 2.08

A11 = 100G **A**1 = 10 G and **A**iso = 40 G - Octahedral structure with covalent character

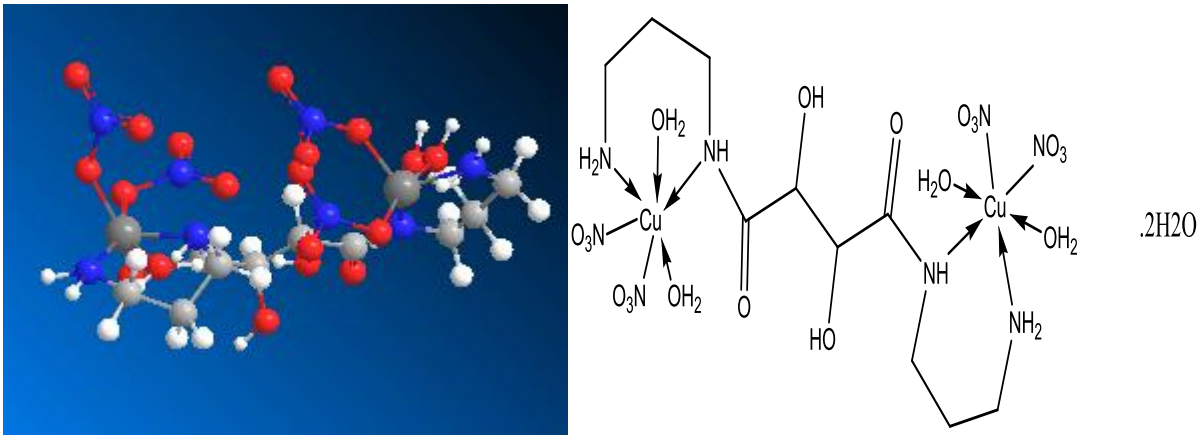


Fig (5): Molecular and 3D structure of the complex (4).

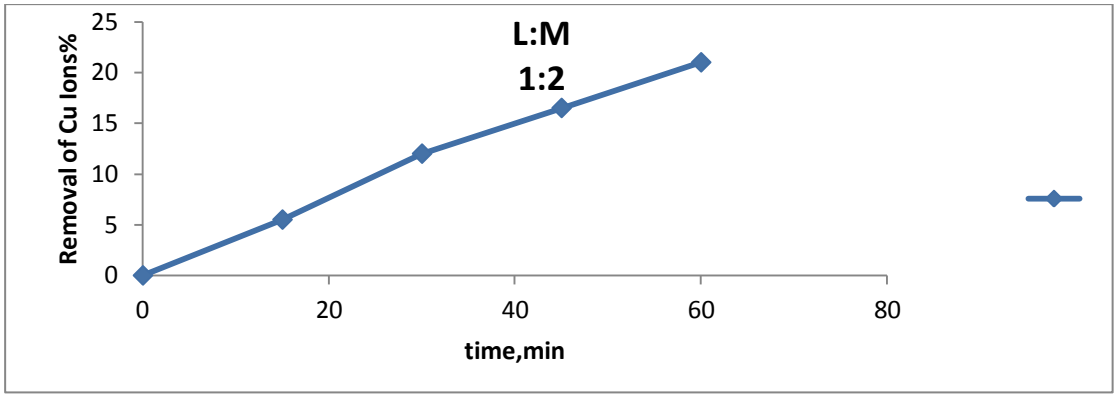


Chart (4) removal Cu ions from sewage sludge at time

At 30 C (1L : 2M)

15 min 5.5%

30 min 12 %

45 min 16.5%

60 min 21.5 %

Complex (5) (1L : 2 M), $Cu(Cl_2)$.$2H_2O$

Chemical Formula : $C_{10}H_{30}N_8O_{20}Cl_4Cu_2$,M.Wt : 604,Colour : yellow ,M.P : >300 C ,Cond : 8.5 Ω^{-1} $mol^{-1}Cm^{-1}$, Elemental Analysis: Calc: C, 19.87, H, 4.97, N, 9.3, Cu,20.96 ; Found (%): C, 19.45, H, 4.56 , N, 9.1 , Cu,20.35

IR Spectrum: υ(H2O) = 3530 - 3320 Cm^{-1},3320-3150 Cm^{-1},υ(H-Bond)= 3600 - 3320 Cm^{-1},3310 - 2820 Cm^{-1},υ(OH) = 3470,3410 Cm^{-1},υ(C=O)= 1635,1615 Cm^{-1},υ(NH) = 3270 Cm^{-1},υ(NH_2) = 3376, 3350 Cm^{-1},υ(Cl_2) = 468Cm^{-1},υ(OH)= 1350,1295 Cm^{-1},υ(Cu O)= 628 Cm^{-1}- **μ** eff 1.17 B.M

Mass spectrum:

$H_6N_2O_4$, m/z=98amu , $C_5H_{11}N_2O_2$,m/z=131 amu , $C7H_{13}N_2O_2$,m/z=189 amu , $C_{10}H_{22}N_4O_4$, m/z=262 amu $C_{10}H_{22}N_4O_4Cu_2$, m/z=388 amu $C_{10}H_{22}N_4O_4Cu_2Cl_2$, m/z=422 amu

UV-vis : 296 nm , 317 , 456 , 580 , 605 nm due to $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$ and d-d transitions .

ESR g11= 2.14 g1 = 2.05 **g**iso = 2.08 - Octahedral structure with covalent bond character

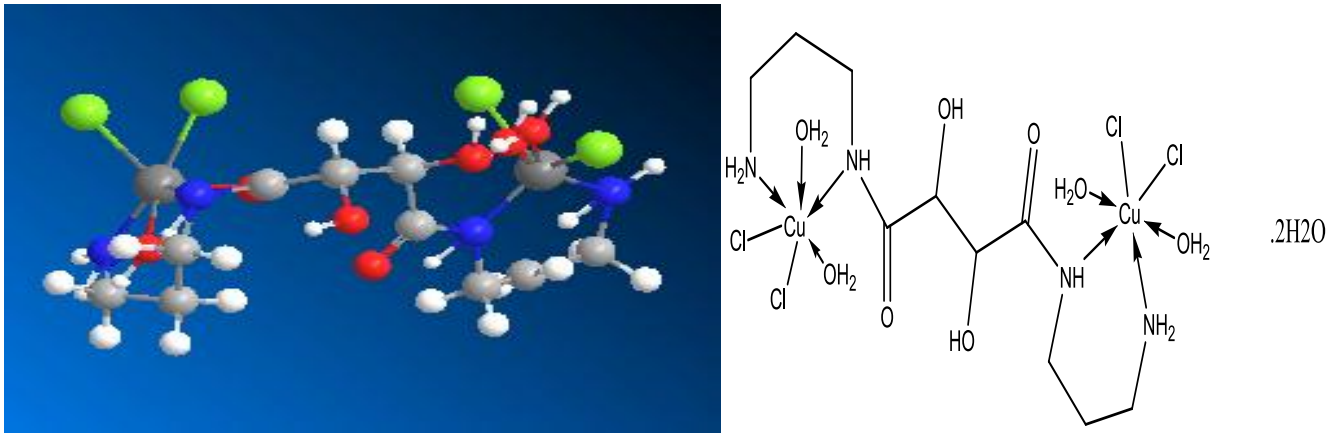


Fig (6): Molecular and 3D structure of the complex (5).

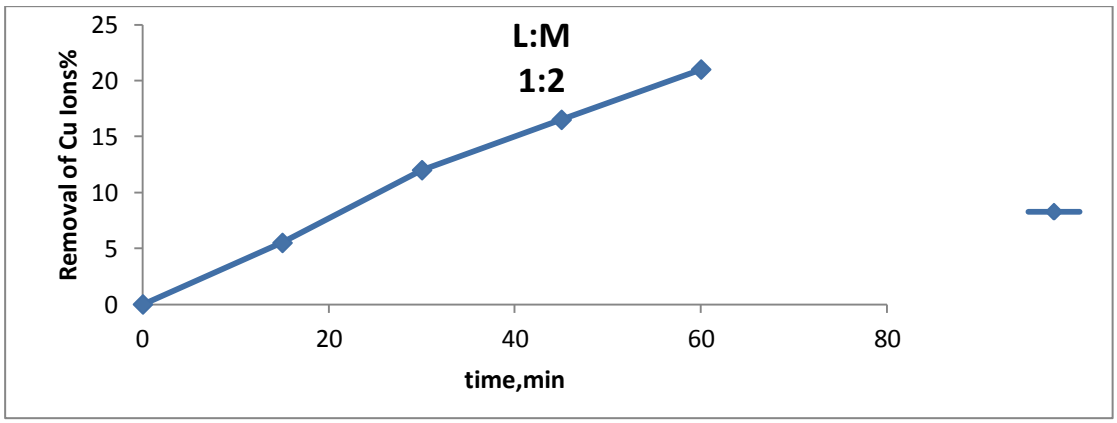


Chart (5) removal Cu ions from sewage sludge at time

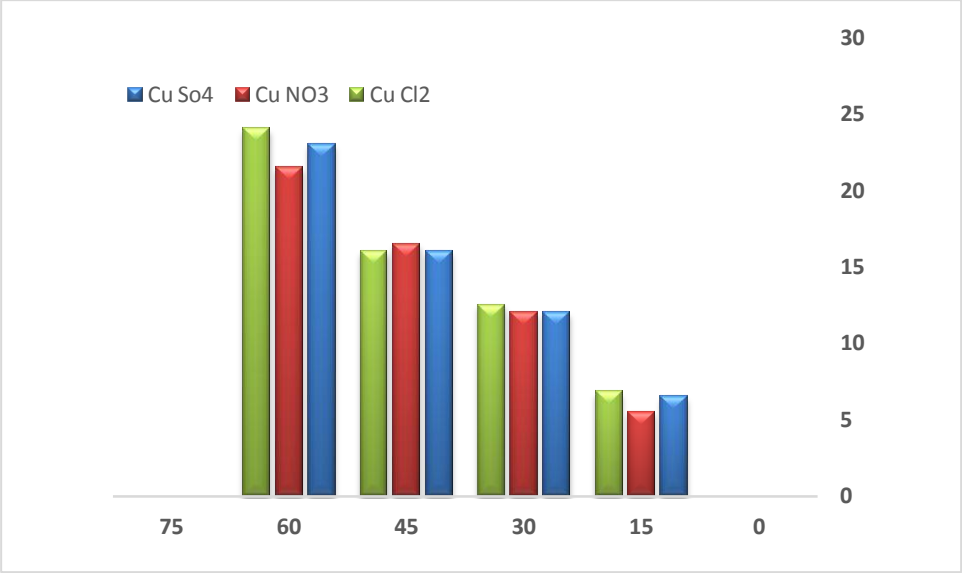
At 30 C (1L : 2M)

15 min 6.5%

30 min 12.8 %

45 min 16%

60 min 24 %



Removal % of Cu ions

Chart (6) removal different Cu ions from sewage sludge at time

Complex (6) (1L : 2 M), $Mn(OAc)_2 .H_2O$

Chemical Formula: C18H40N4O15Mn2,M.Wt : 662 , Colour : yellowish Brown , M.P : >300 C ,Cond : 7.3 Ω^{-1} $mol^{-1}Cm^{-1}$, Elemental Analysis: Calc: C, 32.63, H, 6.04; N, 8.46, Mn,16.61; Found (%): C, 32.2, H, 5.85 , N, 8.12 , Mn,16.21,

IR Spectrum : υ(H2O) = 3530 - 3320 Cm^{-1},3320-3150 Cm^{-1},υ(H-Bond)= 3600 - 3320 Cm^{-1},3310 - 2820 Cm-1,υ(OH) = 3470,3410 Cm^{-1},υ(C=O)= 1635,1615 Cm^{-1},υ(NH) = 3270 Cm^{-1},υ(NH2) = 3376, 3350 Cm^{-1},υ(OAc) = 1511,1410 Cm^{-1},υ(OH)= 1350,1295 Cm^{-1},υ(Mn O)= 620 Cm^{-1}, υ(MnN)=575 Cm^{-1}

μ eff 6.3 B.M

Mass spectrum :

$C_3H_{10}N_2$,m/z=74 amu , $H_6N_2O_4$, m/z=98amu , $C_5H_{11}N_2O_2$,m/z=131 amu , $C_7H_{13}N_2O_2$,m/z=189 amu , $C_{10}H_{22}N_4O_4$, m/z=262 amu , $C_{10}H_{22}N_4O_4Mn$, m/z=316 amu and $C_{10}H_{22}N_4O_4Mn_2$, m/z=370 amu

UV-vis : 296 nm , 310, 450 , 560 , 600 nm due to $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$ and d-d transitions .

ESR giso = 2.03 - Octahedral structure with covalent bond character

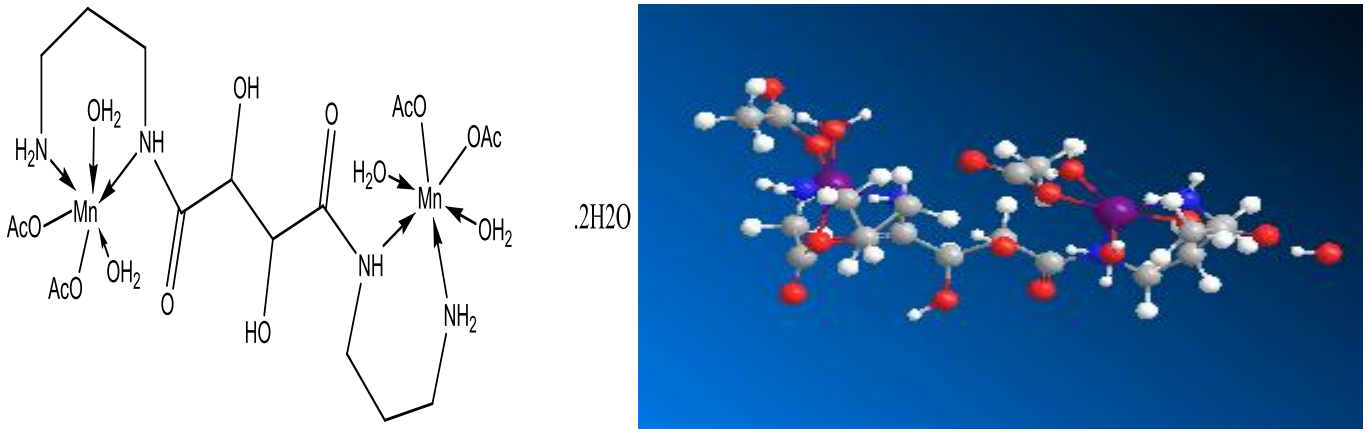


Fig (7): Molecular and 3D structure of the complex (6)

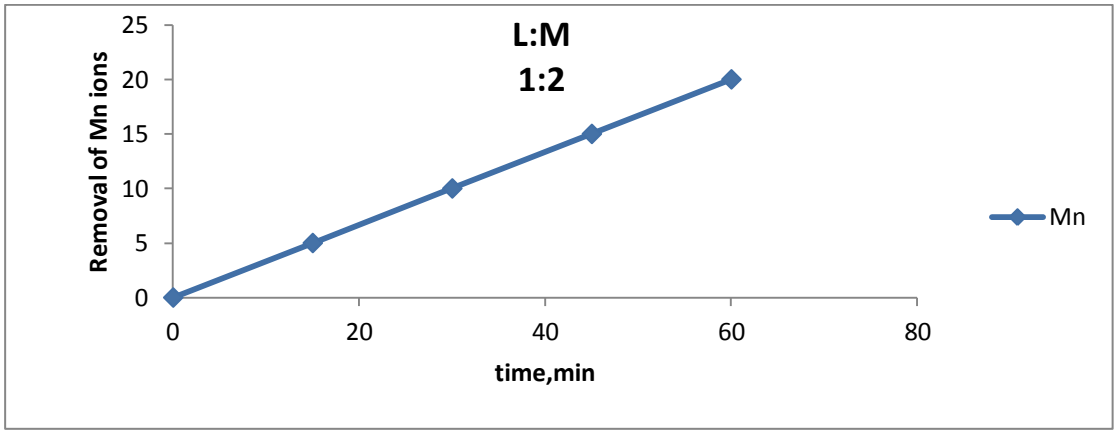


Chart (5) removal Mnions from sewage sludge at time.

At 30 C (1L : 2M)

15 min 5%

30 min 10 %

45 min 15%

60 min 20 %

Complex (7) (1L : 2 M), $Cd(OAc)_2 .4H_2O$

Chemical Formula : C18H40N4O15Cd2 ,M.Wt : 777, Colour : yellowish Brown, M.P : >300 C , Cond :6.3 $\Omega^{-1}mol^{-1}Cm^{-1}$, Elemental Analysis: Calc: C, 27.23, H, 5.14; N, 7.21, cd,28.96 ; Found (%): C, 32.2, H, 5.85, , N, 8.12 , Cd ,16.21,

IR Spect : υ(H2O) = 3530 - 3320 Cm^{-1},3320-3150 Cm^{-1},υ(H-Bond)= 3600 - 3320 Cm^{-1},3310 - 2820 Cm-1,υ(OH) = 3470,3410 Cm^{-1},υ(C=O)= 1635,1615 Cm^{-1},υ(NH) = 3270 Cm^{-1},υ(NH2) = 3376, 3350 Cm^{-1},υ(OAc) = 1511,1410 Cm^{-1},υ(OH)= 1350,1295 Cm^{-1},υ(Cd O)= 628 Cm^{-1}, υ(CdN)= 582 Cm^{-1}

μ eff Diamagnetic

Mass spectrum : H_2N_2,M/Z= 32 amu ,$C_3H_{10}N_2$,m/z=74 amu , $H_6N_2O_4$, m/z=98amu , $C_5H_{11}N_2O_2$,m/z=131 amu , $C_7H_{13}N_2O_2$,m/z=189 amu , $C_{10}H_{22}N_4O_4$, m/z=262 amu and $C_{10}H_{22}N_4O_4Cd$, m/z=374 amu and $C_{10}H_{22}N_4O_4Cd_2$, m/z=486 amu

UV-vis : 290 nm , 317 , 325 nm due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions .

H-nmr (deuterated DMSO) : NH = 7.3 ppm , NH_2= 4.5 ppm , CH_2 group 2.70 -3.15 ppm , CH 2.2 ppm , OH 5.1 ppm

ESR giso = 2.03 - Octahedral structure

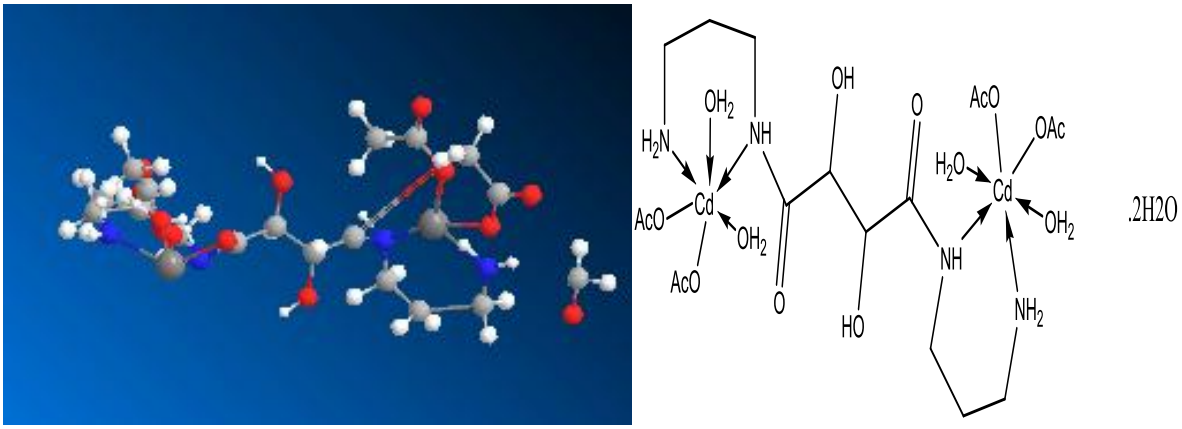


Fig (8): Molecular and 3D Structure of the complex (7)

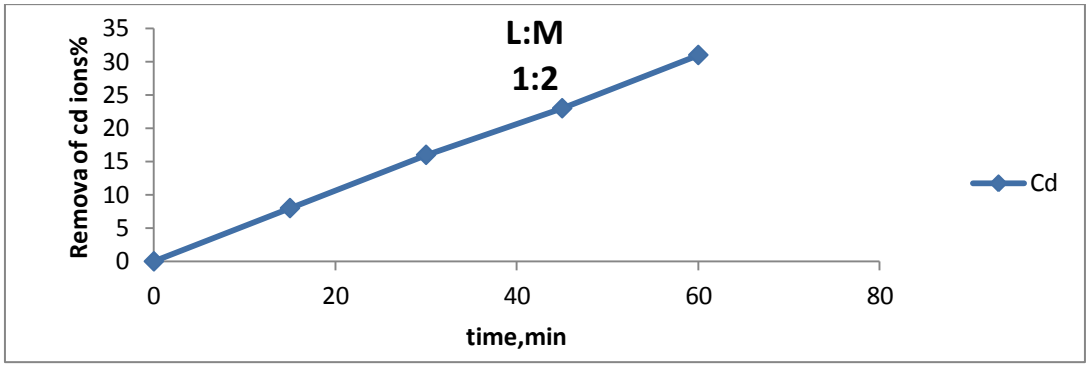


Chart (8) removal Cd ions from sewage sludge at time:

At 30 C (1L : 2M)

15 min	8%
30 min	16 %
45 min	23%
60 min	31%

Complex (8) (1L : 2 M),$Zn(OAc)_2 .2H_2O$

Chemical Formula : C18H40N4O15Zn2 , M.Wt : 777 , Color : yellowish Brown , M.P : >300 C , Cond : 7.2 $\Omega^{-1}mol^{-1}$ Cm^{-1}, Elemental Analysis: Calc: C, 27.73, H, 5.14; N, 7.21, Zn,28.96 ; Found (%): C, 32.2, H, 5.85 , N, 8.12 , Zn,16.21,

IR Spect : υ(H2O) = 3560 - 3320 Cm^{-1},3320-3150 Cm^{-1},υ(H-Bond)= 3600 - 3320 Cm^{-1},3310 - 2820 Cm^{-1},υ(OH) = 3450,3420 Cm^{-1},υ(C=O)= 1635,1615 Cm^{-1},υ(NH) = 3270 Cm^{-1},υ(NH2) = 3376, 3350 Cm^{-1},υ(OAc) = 1511,1410 Cm^{-1},υ(OH)= 1340,1295 Cm^{-1},υ(Cu O)= 628 Cm^{-1}, υ(ZnN)= 585 Cm^{-1}

μ eff Diamagnetic

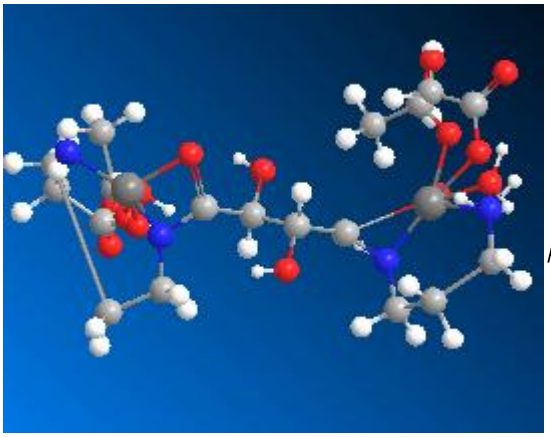
Mass spectrum :

H_2N_2,M/Z= 32 amu ,$C_3H_{10}N_2$,m/z=74 amu , $H_6N_2O_4$, m/z=98amu , $C_5H_{11}N_2O_2$,m/z=131 amu , $C_7H_{13}N_2O_2$,m/z=189 amu , $C_{10}H_{22}N_4O_4$, m/z=262 amu, $C_{10}H_{22}N_4O_4Zn$, m/z=327 amu and $C_{10}H_{22}N_4O_4Zn_2$, m/z=392 amu

UV-vis : 296 nm , 318 , 418 nm due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions .

H-nmr (deuterated DMSO) : NH = 7.2 ppm , NH2= 4.5 ppm , CH2 group 2.70 -3.37 ppm , CH 2.0 ppm , OH 5.5 ppm

Diamagnetic



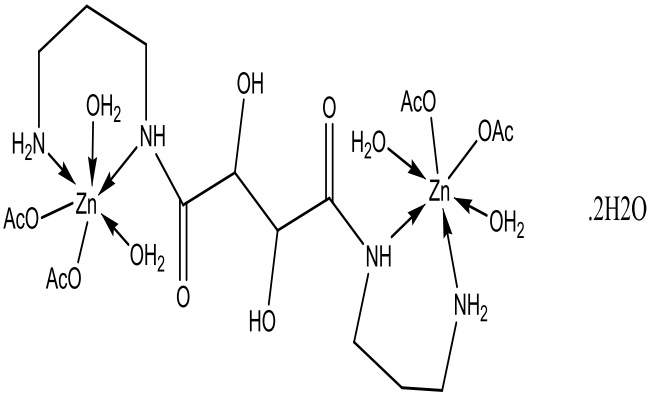


Fig (9): Molecular and 3D structure of the complex (8)

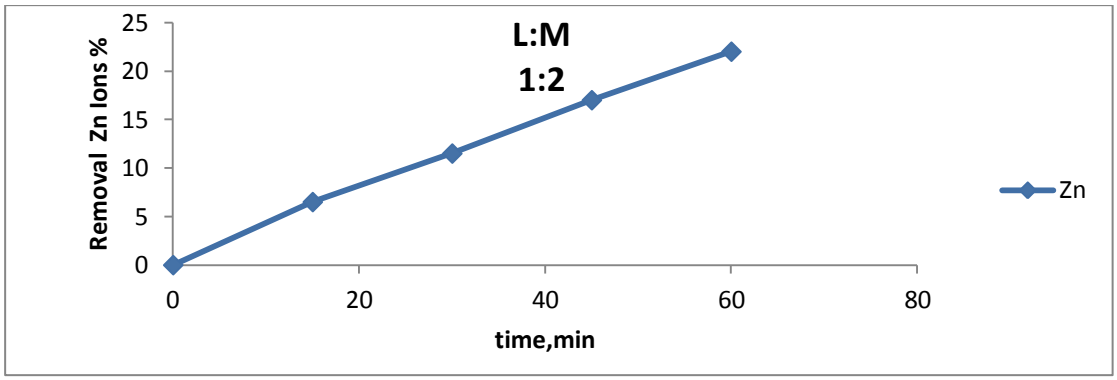


Chart (9) removal Zn ions from sewage sludge at time:

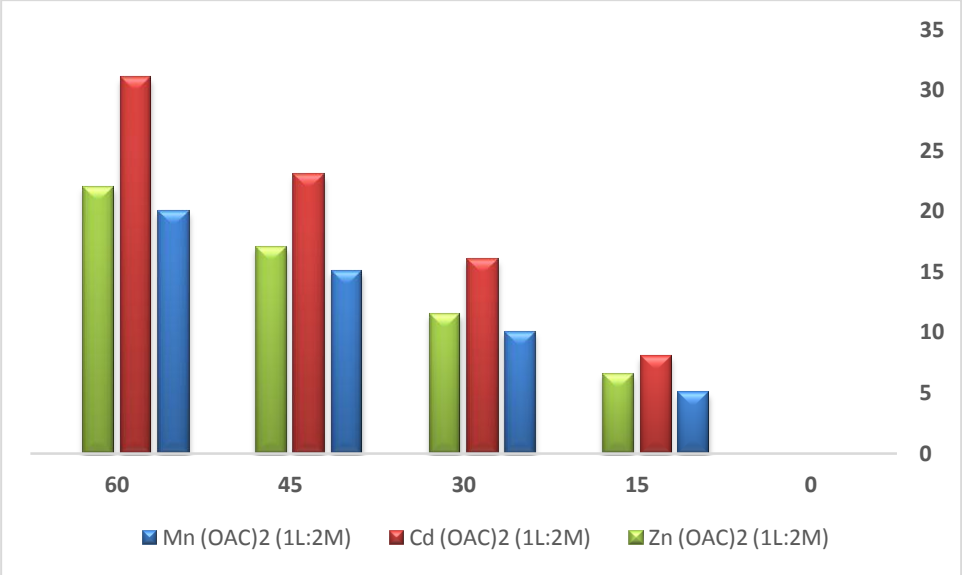
At 30 C (1L : 2M)

15 min 6.5%

30 min 11.5 %

45 min 17%

60 min 22 %



Removal % of Metals ions

Chart (10): removal Mn, Cd and Zn ions from sewage sludge at time

Complex (9) (1L : 2 M), $Fe(SO4)_2 .3H_2O$

Chemical Formula: $C_{10}H_{32}N_4O_{17}S_2Fe_2$, M.Wt : 656 , Color : yellowish Brown , M.P : >300 C , Cond : 8.3 Ω^{-1} mol^{-1} Cm^{-1}, Elemental Analysis: Calc: C, 18.29; H, 4.88, N, 8.54, Fe,17.01; Found (%): C, 17.87, H, 4.52, , N, 8.23 , Fe,16.88,

IR Spectrum : $\upsilon(H_2O)$ = 3530 - 3310 Cm^{-1},3320-3150 Cm^{-1},υ(H-Bond)= 3600 - 3320 Cm^{-1},3310 - 2820 Cm^{-1},υ(OH) = 3470,3410 Cm^{-1},υ(C=O)= 1635,1615 Cm^{-1},υ(NH) = 3270 Cm^{-1},υ(NH2) = 3376, 3350 Cm^{-1},$\upsilon(SO_4)$ = 1252,1167,1082,750 Cm^{-1},υ(OH)= 1350,1295 Cm^{-1},υ(Cu O)= 625 Cm^{-1}

μ eff 5.82 B.M

Mass spectrum:H_2N_2,M/Z= 32 amu ,$C_3H_{10}N_2$,m/z=74 amu , $H_6N_2O_4$, m/z=98amu , $C_5H_{11}N_2O_2$,m/z=131 amu , $C_7H_{13}N_2O_2$,m/z=189 amu , $C_{10}H_{22}N_4O_4S_2$, m/z=334 amu and $C_{10}H_{22}N_4O_4S_2$, m/z=444 amu

UV-vis : 290 nm , 318 , 445 , 572 , nm due to $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$ and Charge transfer transitions .

Octahedral structure with covalent bond character

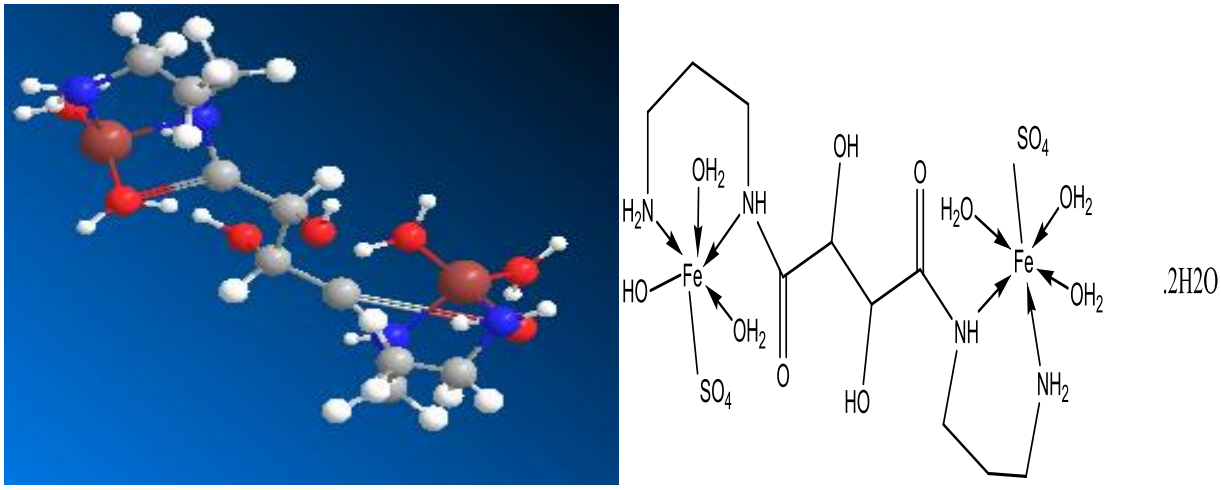


Fig (11): Molecular and 3D structure of the complex (9)

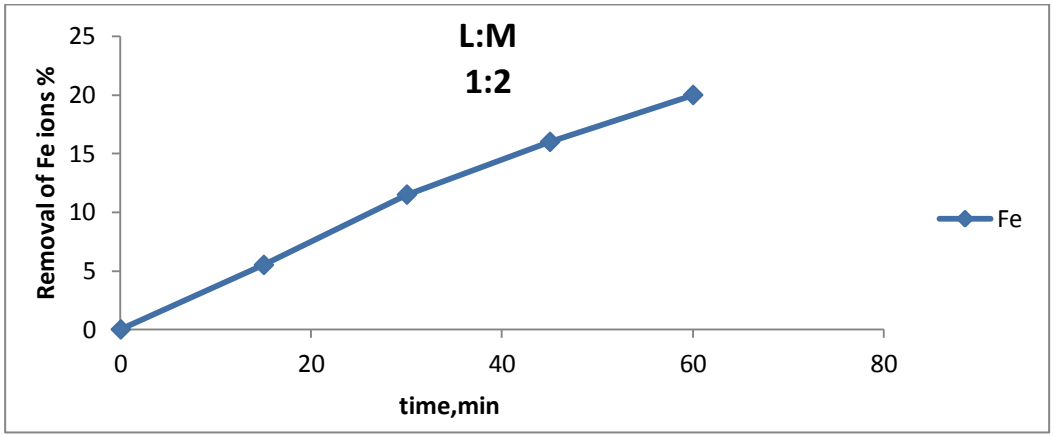


Chart (10) removal Fe ions from sewage sludge at time

At 30 C (2L : 5M)

15 min 5.5%

30 min 11.5 %

45 min 15%

60 min 20%

Complex (10) (1L : 2 M),$Cr(SO_4)_2 .H_2O$

Chemical Formula : $C_{10}H_{32}N_4O_{17}S_2Cr_2$, M.Wt : 648 , Colour : yellowish Brown , M.P : >300 C , Cond : 7.5 Ω^{-1} mol^{-1} Cm^{-1}, Elemental Analysis: Calc: C, 18.52, H, 4.94, N, 8.64, Cr,16.05; Found (%): C, 18.10, H, 4.72, N, 8.35 , Cr,15.75 .

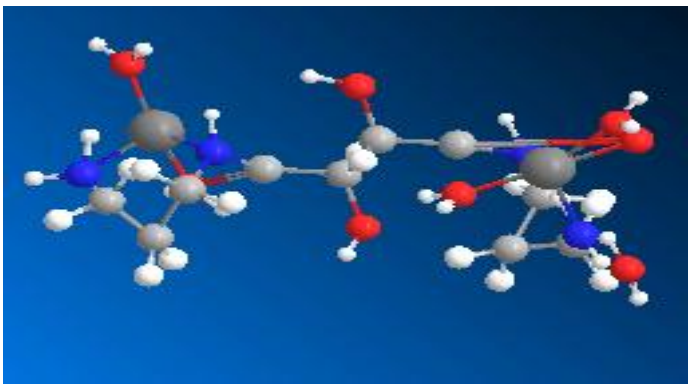
IR Spectrum : $\upsilon(H_2O)$ = 3530 - 3320 Cm^{-1},3320-3150 Cm^{-1},υ(H-Bond)= 3600 - 3320 Cm^{-1},3310 - 2820 Cm^{-1},υ(OH) = 3470,3410 Cm^{-1},υ(C=O)= 1635,1615 Cm^{-1},υ(NH) = 3270 Cm^{-1},υ(NH2) = 3376, 3350 Cm^{-1},$\upsilon(SO_4)$ = 1285, 1180,1041,740Cm^{-1},υ(OH)= 1350,1295 Cm^{-1},υ(Cu O)= 628 Cm^{-1}.

μ eff 3.68 B.M

Mass spectrum: H_2N_2,M/Z= 32 amu ,$C_3H_{10}N_2$,m/z=74 amu ,$H_6N_2O_4$, m/z=98amu , $C_5H_{11}N_2O_2$,m/z=131 amu , $C_7H_{13}N_2O_2$,m/z=189 amu , $C_{10}H_{22}N_4O_4$, m/z=262 amu and $C_{10}H_{22}N_4O_4Cr_2$, m/z=364 amu

UV-vis : 295 nm , 315 , 438 , 550 , nm due to $\pi\rightarrow\pi^*$, $n\rightarrow\pi^*$ and Charge transfer transitions .

Octahedral structure



.2H2O

Fig (11): Molecular and 3D structure of the complex (10)

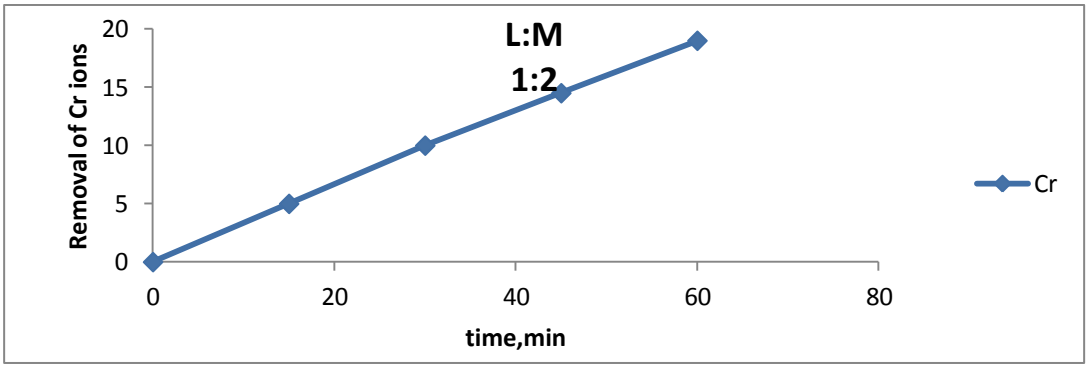


Chart (12) removal Cr ions from sewage sludge at time

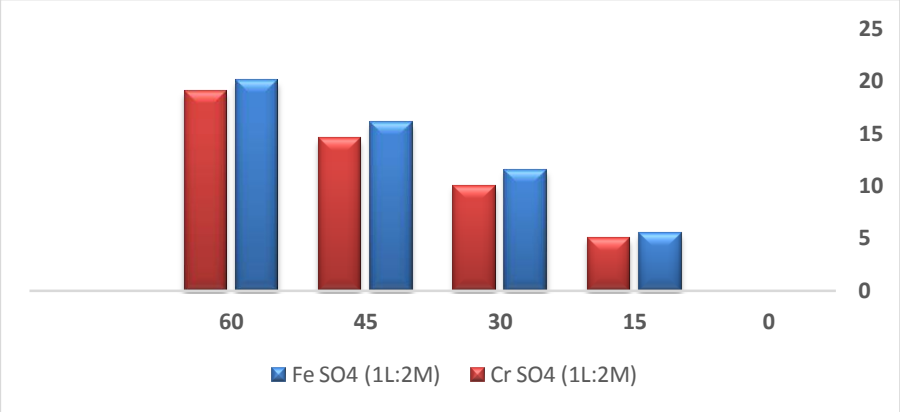
At 30 C (1L : 2M)

15 min 5 %

30 min 10 %

45 min 14.5 %

60 min 19 %



Removal % of Metals ions

Chart (13) removal Zn ions from sewage sludge at time.

Complex (11) (1L : 2 M), $Pb(OAc)_2 .2H_2O$

Chemical Formula: $C_{18}H_{40}N_4O_{15}Pb_2$, M.Wt : 967 , Color : yellowish Brown , M.P : >300 C , Cond : 8.3 Ω-1 mol^{-1} Cm^{-1}, Elemental Analysis: Calc: C, 22.33, H, 4.14 ,N, 5.79, Pb,42.85; Found (%): C, 21.98, H, 3.85, N, 5.38 , Pb,42.45,

IR Spectrum: υ(H_2O) = 3530 - 3320 Cm^{-1},3320-3150 Cm^{-1},υ(H-Bond)= 3600 - 3320 Cm^{-1},3310 - 2820 Cm-1,υ(OH) = 3470,3410 Cm^{-1},υ(C=O)= 1635,1615 Cm^{-1},υ(NH) = 3270 Cm-1,υ(NH2) = 3376, 3350 Cm-1,υ(OAc) = 1511,1410 Cm-1,υ(OH)= 1350,1295 Cm^{-1},υ(Cu O)= 628 Cm^{-1}, υ(CuN)= 585 Cm^{-1}

μ eff Diamagnetic

Mass spectrum:H_2N_2,M/Z= 32 amu ,$C_3H_{10}N_2$,m/z=74 amu , $H_6N_2O_4$, m/z=98amu , $C_5H_{11}N_2O_2$,m/z=131 amu , $C_7H_{13}N_2O_2$,m/z=189 amu , $C_{10}H_{22}N_4O_4$, m/z=262 amu and $C_{10}H_{22}N_4O_4$, m/z=469 amu

UV-vis : 297 nm , 316 , 422 , nm due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions . - **Diamagnetic**

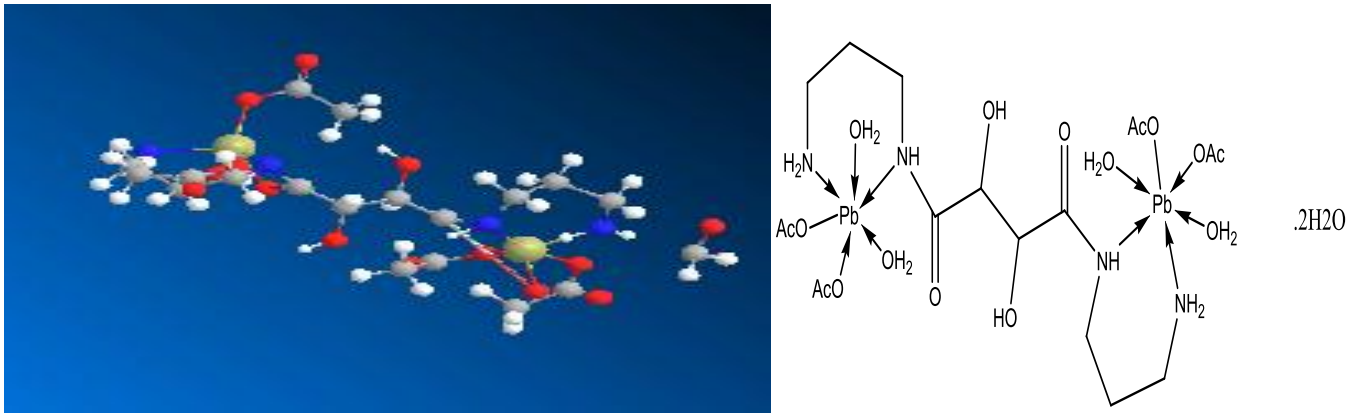


Fig (12): Molecular and 3D structure of the complex (11)

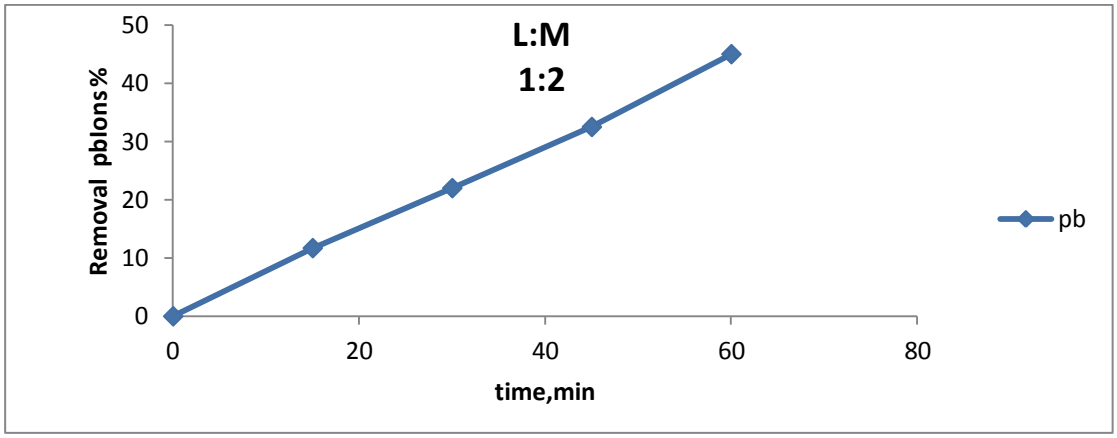


Chart (14) removal Zn ions from sewage sludge at time

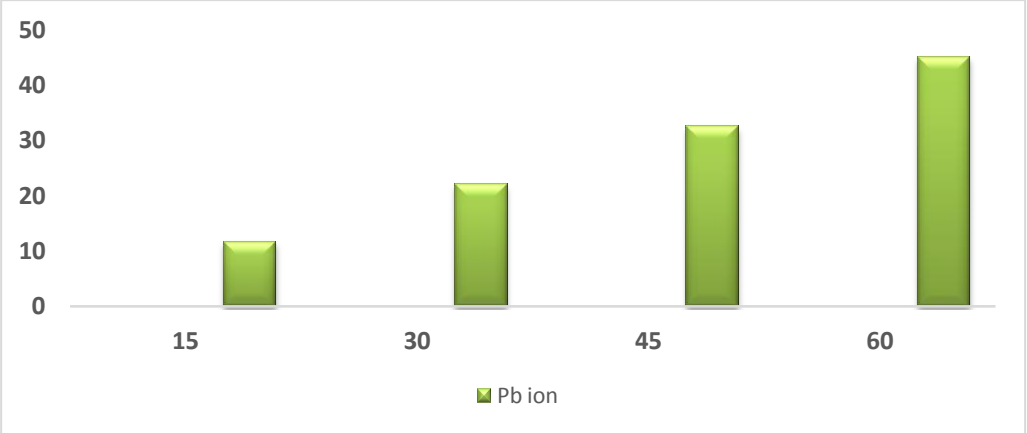
At 30 C (1L : 2M)

15 min 11.7 %

30 min 22 %

45 min 32.5

60 min 45 %



Removal % of Metals ions

3. RESULTSAND DISCUSSION: -

3.1 Measuring of the capacity of the organic ligand (metal removal efficiency):

The amount of metal adsorbed per gramme of biosorbent (qe) can be estimated in mg/g as follows:

$(C_o - C_e)\ V/\ m = Qe$

Where C_o denotes the initial concentration of metal ions (mg/L), Ce denotes the equilibrium concentration of metal ions (mg/L), V denotes the volume of solution (L), and m is the mass of biosorbent (g). The proportion of metal removal can alternatively be represented by the percentage of metal removal, as shown below.

Metal removal (%) = $100(C_o – C_e) / C_o$

When the organic ligand was treated with metal ions in a (2M:1L) molar ratio, the removal efficiency was 65% after 30 minutes and 100% after 60 minutes, while at a (2M:1L) molar ratio, the removal efficiency of metal ions was 52% after 30 minutes (at $25C^o$) and 100% after 60 minutes, as shown in table (1):

Post-treatment at RHT 1hrs and 3hrs in absence of the ligand:

Table (1):- Performance results in treating 5Lsemisolid at a total HRT of 1hrs(at dose 1.0 gL^{-1} from the ligand).

COD	TSS	TDS	pH	BOD	Tem	E.X.P
1532	1050	3500	8.22	952	25C	Raw
65	34	340	7.91	45	25C	Run

Parameters		Unit	Raw	Run	Efficiency
Heavy metals removal	Cu(II)	mgL^{-1}	4.25	0.64	85%
	Cr(II)	mgL^{-1}	5.24	0.35	93%
	Fe(II)	mgL^{-1}	5.01	1.15	77%

	pb(II)	mgL^{-1}	2.23	0.45	80%
	Zn(II)	mgL^{-1}	1.87	0.32	82%
Fecal Coliform		unit/l00ml	$5.4x10^6$	$3 x10^3$	99.9%

At, **Volume** = 5L Semisolid sludge
(RT) Retention Time = 1 hours
Wt of Lig(1)=1.0gm

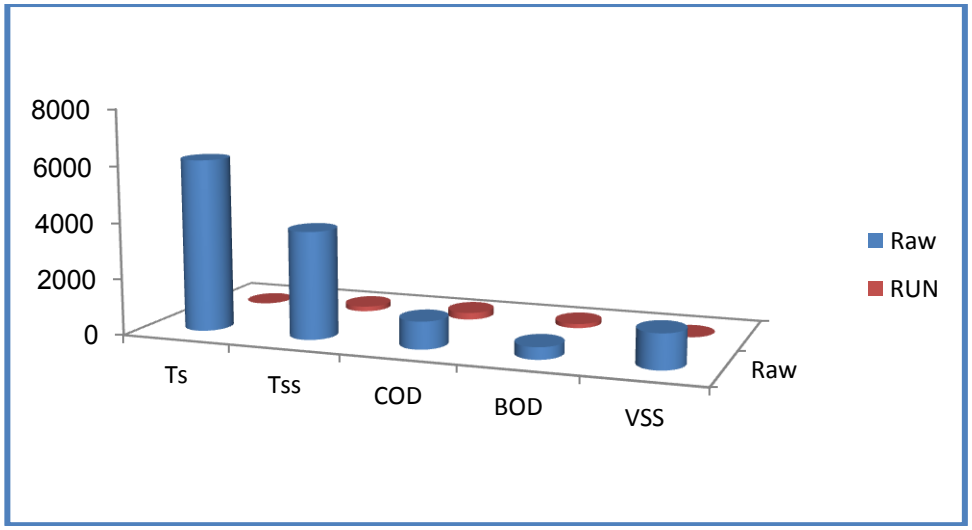


Chart (15): Performance results at a total HRT of 1 hrs.(at dose 1.0 gL^{-1}).

Heavy metals removal efficiency:

Heavy metal particles should be absorbed and captured on the surface of the ligand surface area. Particles to be digested should be captured first, and digestion and biodegradation processes should then take place on land. The available data suggested that the ligand performed well in terms of heavy metal removal efficiency. The results also showed that when treated with the ligand, the residual values of fecal coliform decreased from 3.4x106 to 3.4x103 (99.9%).

Table (2): Performance results in treating 5L semisolid at a total HRT of 3hrs (at dose 1.0 gL^{-1} from the ligand).

COD	TSS	TDS	pH	BOD	Tem	E.X.P
1532	1050	3500	8.22	952	29C	Raw
59	25	250	7.23	39	28C	Run

Parameters		Unit	Raw	Run	Efficiency
Heavy metals removal	Cu (II)	mgL^{-1}	4.25	0.61	86%
	Cr (II)	mgL^{-1}	5.24	0.29	95%
	Fe (II)	mgL^{-1}	5.01	0.15	77%

	pb (II)	mgL^{-1}	2.23	0.41	82%
	Zn (II)	mgL^{-1}	1.87	0.21	89%
Fecal Coliform		unit/l00ml	$5.4x10^6$	$3 x10^3$	99.9%

At, **Volume** = 5L Semisolid sludge

(RT) Retention Time = 3 hours

Wt of Ligand (1)=1.0 gm

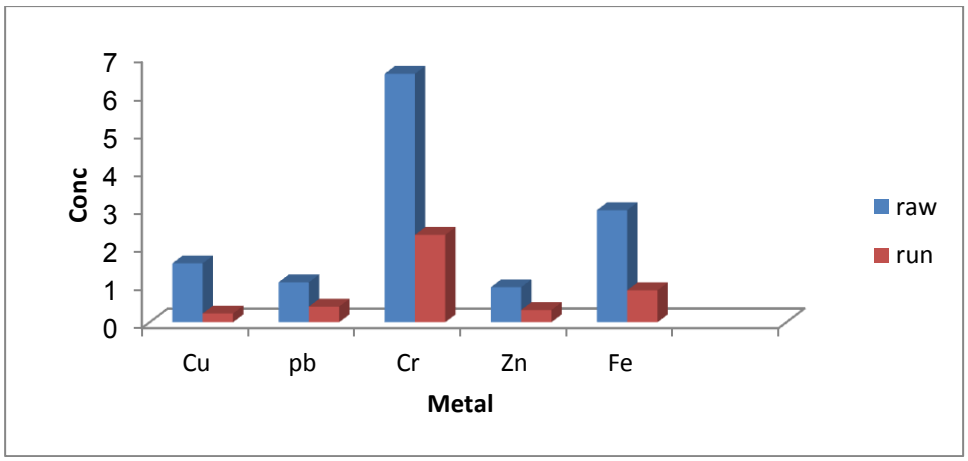


chart (16) Variation in heavy metals concentrations along raw and treated wastewater.

Heavy metals removal efficiency:

The results showed that concentration of heavy metal decreases as HRT 3hrs at dose 1.0 mgL^{-1} The Cu amount decrease from 4.25 to 0.61 mgL^{-1} (86.0%).The Cr amount decrease from 5.24 to 0.29 mgL^{-1} (95.0%). the Fe amount decrease from 5.01 to 1.15 mgL^{-1} (77.0%). Pb amount decrease from 2.23 to 0.41 mgL^{-1} (82.0%). The Zn amount decrease from 1.87 to 0.21 mgL^{-1} (89.0%). Heavy metal particles should be absorbed and captured on the surface of the Ligand surface area. Particles to be digested it should be captured first and the digestion and the biodegradation processes will be then occurred in the land. Available data indicated good performance of the Ligand regarded to heavy metals removal efficiency. Also, the results also showed that, the residual values of fecal coliform when treated with the ligand, the amount decreases from 3.4x106 to 3.4x103 (99.9%).

Post-treatment at RHT 3hrs in presence of the organic ligand (at dose 3.0 gL^{-1} from the ligand):

Table (3): Performance results of treating municipal wastewater at a total HRT of 3hrs(at dose 3.0 gL^{-1} from the ligand).

COD	TSS	TDS	pH	BOD	Tem C^0	E.X.P
1532	1050	3500	8.22	952	25	Raw
55	30	95	7.89	35	25	Run

Parameters		Unit	Raw	Run	Efficiency
Heavy metals removal	Cu (II)	mgL^{-1}	4.25	0.31	92%
	Cr (II)	mgL^{-1}	5.24	0.10	97%
	Fe (II)	mgL^{-1}	5.01	0.45	97%
	pb (II)	mgL^{-1}	2.23	0.21	90%
	Zn (II)	mgL^{-1}	1.87	0.04	98%
Fecal Coliform		unit/l00ml	$5.4x10^6$	$3 x10^3$	99.9%

At, **Volume** = 5L Semisolid sludge
(RT) Retention Time = 3 hours
Wt of Lig(1)= 3 gm

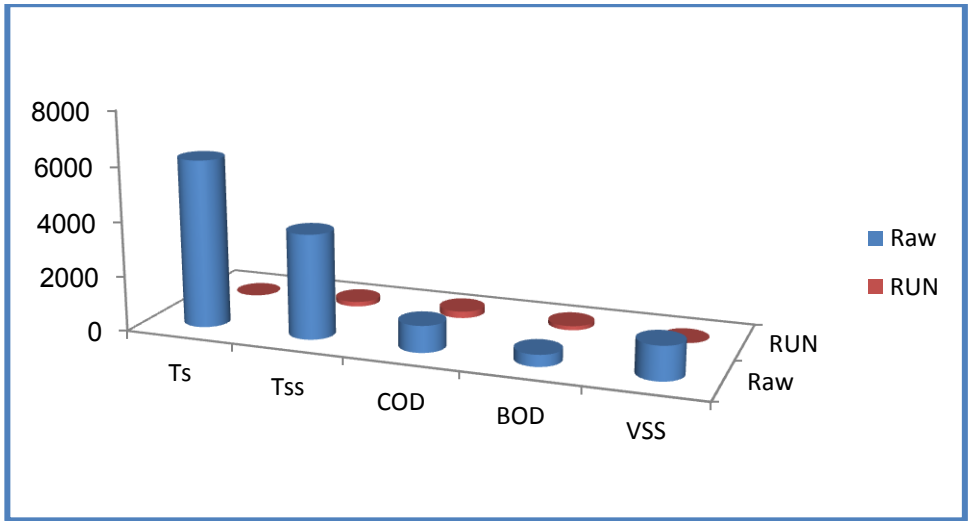


Chart (17): Performance results at a total HRT of 3 hrs (at dose 3.0 gL^{-1}).

Heavy metals removal efficiency:

The results show that concentration of heavy metal decreases as HRT 3hrs at dose 1.0 mg.L^{-1} The Cu amount decrease from 4.25 to 0.0.31 mgL^{-1} (92.0%).The Cr amount decrease from 5.24 to 0.10 mgL^{-1} (97.0%). the Fe amount decrease from 5.01 to 0.45 mgL^{-1} (97.0%). Pb amount decrease from 2.23 to 0.21 mgL^{-1} (90.0%). The Zn amount decrease from 1.87 to 0.04 mgL^{-1} (98.0%)

Conclusion:

The removal efficiency of heavy metals Cu (II),Pb(II),Zn(II),Cr(II) and Fe(II) were increased by increasing the concentration of ligand and the temperature of medium. The concentration of ligand was increased by passing time reach at 60 min and the temperature reached to 50ºC caused increasing in removal efficiency until reached to 100% in Cu(II),Pb(II),Zn(II),Cr(II) and Fe(II).We observed that, when these metals treated with ligand, their amount were decreased as they captured firstly in surface of ligand and then digestion and biodegradation process occurred in the land and the available data indicated good performance of ligand regarded to heavy metals removal efficiency. The result also, showed that, residual values of fecal coliform when treated by ligand with different concentration the amount of heavy metals decrease to 99.9%.

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Conflicts of Interest

The authors declare no conflict of interest.

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