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Synthesis, Characterization and Antibacterial Activity of Ni(Ii), Co(Ii) eith EDTA Complexes

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ABSTRACT

In this work, we study metal (II) coordination compounds of EDTA that were synthesized and characterized using physicochemical properties, where the percentage yields, colors, and melting points (M.P) of the coordination compounds are presented. Ultraviolet Spectroscopy (UV-Vis) and infrared spectroscopy have been used as spectroscopic techniques. These complexes are well known for their biological (antibacterial) activity. We demonstrated that the Co(II) complex showed higher activity against *S. aureus* as a gram-positive bacterium than the Ni(II) complex and EDTA ligand.

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Keywords- *preparation of complexes, infrared and UV-Vis spectroscopy techniques, antibacterial activities.*

INTRODUCTION

Complexone is a term introduced by Schwarzen Bach in 1945 for a series of organic ligands that usually contain at least one iminodiacetic acid group [– $N(CH_2COOH)_2$] or two amino acetic acid groups ($NHCH_2COOH$), which form stable complexes with almost all cations. The high stability constants of the complexes formed by these ligands are due to the presence of basic secondary or tertiary amino groups, the large negative charge of the ligand anions and the formation of stable five-membered chelate rings with metal ions. In general, functionalized polymers for metal-ion complications can be prepared either by deriving a basic polymer (precursor) with the desired ligand or by polymerizing the corresponding ligand derivative [1,2,3,4]. Among the functionalized polymers, those containing EDTA have attracted much attention. EDTA with amino carboxylic acid groups can be introduced into or grafted onto the backbone of polymer chains. Polymers bearing such groups form stable complexes with various heavy metal ions [5,6,7,8,9]. Therefore, we attempted to prepare new complex polymeric materials that could be used for wastewater treatment. Cobalt and EDTA form an extremely stable and soluble 1:1 complex [9].

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(EDTA) functionalized polyacrylonitrile was dispersed in aqueous solutions of various metal ions, such as Ni^{2+} and Co^{2+}, and their metal contents were measured using the UV-Vis spectroscopy technique.

Chemical compounds that inhibit or kill bacterial growth are produced through a series of chemical reactions [10,11]. Antibiotic treatment began in Germany in the late nineteenth century [12,13], and in 1980, the discovery of antibiotics declined because of their high-cost manufacturing. In other words, most microbes can resist most antibiotics [10,11,14]. Bacterial infections are the cause of death in a high percentage of people worldwide. Therefore, we need to know more about these bacteria and the extent of their resistance to available antibiotics. Therefore, there is an increasing need for new antibiotics [15].

Escherichia coli is a gram-negative, facultative aerobic, enveloped bacillus. These bacteria occur naturally in the intestines of humans and animals; however, some can cause diseases, such as urinary tract infections, domestic infections, neonatal meningitis, chest infections, septicemia, and enteritis. *Staphylococcus aureus*, also known as *Staphylococcus aureus*, is a gram-positive, non-motile, non-spore-free, facultative anaerobic bacterium. These cocci are arranged in clusters and cause purulent infections such as endocarditis, osteomyelitis, and toxic shock syndrome. It is one of the most common causes of acquired pneumonia, especially in hospitals, and causes septicemia and surgical wound infections [16].

MATERIALS AND METHODS

General

All the reagents and solvents used were of analytical quality. Melting points (M.P.) were measured using open capillary tubes on a Gallen-Kamp (variable heating) melting point apparatus. UV-Vis spectra were measured using a Genesis 10 UV-Vis spectrophotometer.

Synthesis of the Complexes

Dissolve 0.4 gm (0.01 mol) of NaOH in 10 ml water and then add 3.8 gm (0.01 mol) of Na2H2EDTA.2H2O. The solution was gently heated until the solid dissolved and a clear solution was obtained. Dissolve (2.527 gm, 2.462 gm (0.009 mol)) metal salts Co(II) and Ni(II), respectively, in 5ml water, which is then added to the EDTA solution with swirling. The water was boiled carefully until most of the powder precipitated.

The solution was cooled, and the precipitate was collected by suction filtration. The product was thoroughly washed with ice water until it was free of Ni(II) and Co(II) ions. The product was washed twice with ethanol and dried using filter paper.

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Preparation of the extracts

Take (1.72gm,1.12gm) of $[Co(EDTA)_2]^{2-}$ and $[Ni(EDTA)_2]^{2-}$ powder in (20 ml) of distilled water at 25 rpm in a shaking incubator for 24 h at room temperature. The solution was filtered through Whatman No. 1 sterile filter paper and the extracts were evaporated by drying in clock bottles in the air at room temperature [17,18]. The extracts were dissolved in distilled water (2.5 ml distilled water), placed in sealed glass tubes for storage, and stored in a refrigerator at 4 °C until use [19].

Test organisms

Escherichia coli gram-negative and *Staphylococcus aurous* gram-positive. The bacterial strains were obtained from the Hospital of El-Marj-Libya.

Determination of antibacterial activity

The following microorganisms were tested: gram-negative- *Escherichia coli* and gram-positive *Staphylococcus aureus*, which were cultivated and stored in Nutrient Agar (NA). Muller-Hinton Agar Medium was used for the antibacterial test. The agar diffusion method was used to assess the antimicrobial activity of the extracts by equipping the bacterial suspension by taking from 3-5 colonies of bacteria and putting in 3-4ml normal saline then taking from the suspension 100 µl and putting in all agar plates with a Sterile cotton swab containing bacterial cultures incubated at 37 °C for 24 h. The extracts were added directly to the agar plates using the droplet method (100µL) [20,21]. The prepared extracts were then added to the wells at a standard concentration (100µL). All the plates were incubated at 37 °C for 24 h.
The zone of inhibition on the plates was then measured. All tests were performed in triplicate. Clear zones greater than 7 mm were considered positive results because the cork borer had a diameter of 7 mm [20,22].

RESULTS AND DISCUSSION

Physicochemical Properties

The colors, percentage yields, melting points (M.P), and temperatures of decomposition (d) of the coordination compounds are presented in **Table 1.** The complexes showed a wide range of colors, consistent with those of similar coordination compounds.

Table 1. shows some physical properties of prepared complexes

complexes	***Color***	***MP(C^0)***	***Wt of product(g m)***	***%Yield***
$[Co(EDTA)]^{2-}$	Pink	349.6	2.7243	80
$[Ni(EDTA)]^{2-}$	Blue	349.3	2.1744	75

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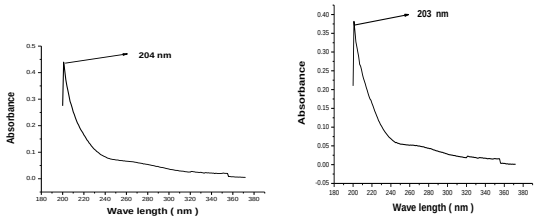
Electronic Spectra and UV visible spectra

The electronic spectral data for the complexes are shown in **Figure 1(a,b)** of the complexes prepared. They showed that the EDTA band corresponding to n→π* changes to a higher wavelength in the Co(II) and Ni(II) complexes, indicating a combination of H2N:→M and COO:→M LMCT transitions.

Transitions of -NH_2 and – COO -. Shifts in these bands and the observed *d-d* transitions of the compounds. The Co(II) complex spectrum showed bands at 204 nm, and weak band transitions were observed at 360 nm and assigned to $^2T_{1g}\rightarrow{}^2E_g$ [23]. The spectrum for Ni(II) in this complex showed a band at 201 nm, which was assigned to T_{1g} [24].



a **b**

Figure 1(a,b)UV\Vis absorption spectra complexes(Ni(II),Co(II)) with EDTA

Magnetic Susceptibility (BM)

The magnetic susceptibilities of the complexes are presented in **Table 2.** The Co(II) complex exhibited a magnetic moment of (d^7) with paramagnetic indicating the low-spin distorted tetrahedral geometry of the complex. The Ni(II) complex exhibited a magnetic moment of (d^8), indicating the low-spin nature of the complex and it has octahedral geometry.

Table 2. shows the magnetic susceptibility values of the complexes:

Complexes	*Magnetic Susceptibility(BM)*
[Co(EDTA)]$^{2-}$	1.41
[Ni(EDTA)]$^{2-}$	2.82

Infrared Spectra

Figures 2(a,b) show the Infrared spectra results, which compare their vibration frequencies to those of EDTA with a metal ion.

The Infrared spectra for EDTA indicate a broad band at 3323.22cm^{-1} - 2941.02cm^{-1} and a medium band of 1575.38cm^{-1} corresponding to -OH, -NH_2, and –COOH stretching [25].

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The new band at 898cm^{-1} - 816cm^{-1} and 781cm^{-1} -712cm^{-1} were included in the [M-N] and [M-O] bond stretching band frequencies, respectively, and served as further evidence of coordination via the nitrogen and oxygen atoms of the ligand. While Ni(II) complex at 3272cm^{-1}, 3003cm^{-1} and Co(II) complex at 3125cm^{-1}, 2871cm^{-1}.

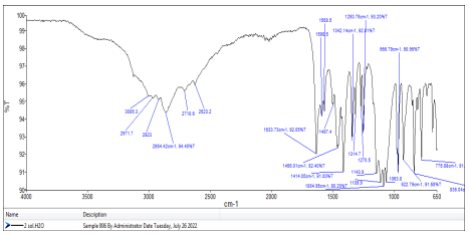

Figure 2(a).IR Spectra of [Ni(EDTA)]$^{2-}$

Figure 2(b).IR Spectra of [Co(EDTA)]$^{2-}$

Antibacterial activity

The EDTA ligand and its metal complexes Co(II) and Ni(II) were screened for susceptible organisms (*S. aureus* and *E. coli* as gram-positive and gram-negative bacteria).

The EDTA ligand and the complexes tested showed low activity against *S. aureus*, a gram-positive bacterium. On the other hand, the ligand shows lower activity against *E. Coli* as a gram-negative bacterium, but the Co(II) complex shows high activity and the Ni(II) complex shows moderate activity against S. aureus as a gram-positive bacterium **Table 3.**

Table 3. shows the antibacterial activities of the ligands and their Co(II) and Ni(II) complexes.

Compound	*inhibition zone diameter (mm) E. Coli*	*Inhibition zone diameter (mm) S. aureus*
EDTA	9.43	10.23
$[Co(EDTA)_2]^{2-}$	21.43	23.39
$[Ni(EDTA)_2]^{2-}$	9.88	10.56

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CONCLUSION

The results of this study indicated that (ethylene diamine tetra acetic acid) is one of the best and most commonly used chelating agents that use both nitrogen and oxygen atoms, which is a Hexa dentate. This was confirmed by the characterization of IR and magnetic moment data and from the UV spectra, and we found that the geometry was octahedral and yet exhibited increased antibacterial activity. We found that the greatest effect was due to the bond between the metal and ligand and not by the ligand alone. The most affected bacteria were *Staphylococcus aurous*-gram-positive because the structure of the cell wall of gram-positive bacteria is less complex than the structure of the cell wall of gram-negative bacteria.

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