Deanship of graduate studies
Al-Quds University

Synthesis and characterization of a novel Hydroxamic acid based polymer

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M.Sc. Thesis

Jerusalem, Palestine

1444/2022

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A thesis submitted in partial fulfillment of requirements for the degree of Master of Applied and Industrial Technology /

Graduate studies / Al-Quds University.

1444/2022

Al-Quds university

Deanship of Graduate Studies

Applied and Industrial Technology program

Thesis approval

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Master thesis Submitted and Accepted, Date: 10/9/2022

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Jerusalem – Palestine

1444/2022

Dedication

I would like to dedicate this work to my first hero in this life, who was the reason to get to this stage, to the greatest man in my life, my father, Ismail, "May God have mercy on him,".

To the source of my strength and giving, the one who paved the way for me, my dear mother, Najat.

To my dear sisters and brother, to the source of hope and light in my life, I'm grateful of your support at all times.

To my fiancé, Bashar, who has been supporting me all along, the source of my continuity, who has never stopped pushing me forward, I would not have passed this stage without his support.

To all my friends and everyone who supported me and helped me.

To my supervisor, Dr. Mahmoud Alkhatib, who did not hesitate for a moment to provide assistance and the co-supervisor, Dr. Mohanad Qurei.

Declaration:

I certify that this thesis is submitted for the degree of Master graduation in applied industrial technology is my own research, except where otherwise acknowledges, and that this thesis (or any part of the same) has not been submitted for the higher degree to any other university or institute.

Signed: ……………………..

Name: Alaa Ismail Asaad.

Date: 10/9/2022

Acknowledgment

First, I thank God for everything.

Dr. Mahmoud Alkhatib, my supervisor, deserves special thanks for his expert advice, direction, and tireless efforts in completing this project.

I thank my family, friends, and everyone who supported me during my pursuit of knowledge.

Abstract:

Water-insoluble polymers based on hydroxamic acids are becoming increasingly important in their applications for their impressive chelating ability with heavy metals. In this study, N^1,N^3-dihydroxymalonamide monomer was produced by reacting diethyl malonate with hydroxylamine hydrochloride using potassium hydroxide as a base in methanol. Then this monomer, N^1,N^3-dihydroxymalonamide, was reacted with α,α′-Dibromo-p-xylene in DMF at 60 °C with stirring overnight to produce a condensation polymer. The average molecular weight of this polymer was determined by static light scattering to be 728,000 g/mol. The glass transition temperature (T_g) and the melting point (T_m) were determined using DSC, their values were 73 °C and 148 °C, respectively. The polymer was partially soluble in dichloromethane and insoluble in water and in DMF. In addition, when the polymer's ability to chelate with chromium ions Cr(VI) was investigated, it was discovered that 1.0 g of polymer could remove 21% of Cr(VI) at pH=3 .

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Abbreviations:

$CDCl_3$	**Deuterated chloroform**
DLS	**Dynamic light scattering**
DMF	**Dimethylformamide**
DMSO	**Dimethyl Sulfoxide**
DSC	**Differential Scanning Calorimetry**
FTIR	**Fourier transform infrared**
HA	**Hydroxamic acid**
HDAC	**Hydroxamic acid anticancer**
kDa	**kilodaltons**
MW	**Molecular weight**
NMR	**Nuclear magnetic resonance**
PDI	**Polydispersity index**
PHA	**Poly hydroxamic acid**
Rbf	**Round bottom flask**
SLS	**Static light scattering**
Tg	**glass transition temperature**
WHO	**World Health Organization**

Chapter one

1. Introduction

1.1 Polymer

The word "polymer" comes from the Greek words poly and meros, which signify many pieces. The intramolecular forces that keep the polymer molecule intact are covalent bonds. Assemblies of these molecules are also linked together in the bulk state by the typical intermolecular forces, such as hydrogen bonds, dipole-dipole interactions, London forces, etc. [1]

As repeat units are arranged in chains to form polymer molecules, the repeat unit is the second most significant structural component after the chain. By covalently connecting only one or two types of repeat units together, many polymer molecules can be created. As a class of compounds, these units are known as monomers and are the building blocks from which chains are created. The molecular mass of a repeat unit must be indicated as M_0. Simple repetitions within a molecule determine a polymer's degree of polymerization. The molecular weight rate between the polymer and the repeat unit, called N, that determines the degree of polymerization. [1]

$N = M/M_0$ [1]

Based on the differences in composition of the polymer comparing to that of the monomer(s), wherefrom it was derived, first categorized polymers into condensation and polymer additions. Condensation polymers are those that are created from multifunctional monomers by a variety of condensation processes in organic chemistry, while eliminating some small molecules like water. Diamine and diacids with water removal-based polyamides are examples of such a condensation polymer. [2]

There are dozens of syntheses of reactants that may be used in the assembly of numerous condensation polymers. Thus, the self-condensation of amino acids and the reactions of diamine with diacid or diacetyl chloride may be used to produce polyamides. Similarly, polyesters can be synthesized from diols by esterification with diacids or ester interchange with diesters. Some are naturally created polymers, including cellulose, starch, wool, and silk, are categorized as condensation polymers because it is possible to infer their composition from a reactant by excluding water. Consequently, it is possible to think of cellulose as a polyether produced through the dehydration of glucose. [2]

Addition polymers are composed entirely of monomers, with no small molecule loss. The repeating unit of an addition polymer is similar to the monomer, in contrast to condensation polymers, which differ in composition. The major addition polymers are those formed by polymerization of monomers containing the carbon–carbon double bond. In addition to vinyl chloride and styrene, it applies to any monomers that have a carbon-carbon double bond, among them methyl methacrylate, vinylidene chloride, and 2-butene. By changing double bonds into saturated bonds, vinyl monomers can react with one another to form polymers. [2]

Chain initiation, chain propagation, and chain termination are the three steps that form chain polymerization. Chain transfer, a fourth process that might possibly be present, could be seen as a fusion of chain termination and chain initiation. Chain polymerization processes are also defined as free-radical polymerization, cationic polymerization, or anionic polymerization because chain initiation is caused by an attack on the monomer molecule by a free radical, cation, or anion. Free radicals are reactive substances with an unpaired electron that are often created by the decomposition of an initiator, a substance that is particularly unstable. [3]

The free radical attacks one of the monomer's doubly bonded carbon atoms in the initiation process, the initial stage of the chain reaction. A bond is created between the free radical and one carbon atom when one electron from the double bond pairs with the free radical's odd electron. The other carbon atom, which is now a free radical, receives the last electron of the double bond. [4]

However, the new free radical has the potential to add more monomer units, starting a chain reaction that stands in for the propagation step. Termination is the last stage, and it can happen through a variety of processes. [4]

A homopolymer, often known as a polymer, is produced when a macromolecule is created from just one type of monomer. When referring to polymers of a structure that may be described by the repetition of a single type of repeating unit including one or more species, the term homopolymer is sometimes used more generally. A hypothetical polymer – $[AB]_n$−composed of A and B species is therefore also a homopolymer. A copolymer is a polymer made of many species of monomer. A random copolymer is a type of copolymer that has diverse mers or repeating units distributed quite randomly throughout its structure. [3]

—ABBABBBAABBAABAAABBA—

The last three types are alternating, block and graft copolymer structures. The two monomers alternate in a predictable manner across the polymer chain in an alternating copolymer. A linear polymer called a block copolymer contains one or more uninterruptedly long sequences of each monomer in the chain.

Different block copolymers can have various numbers of blocks. As a result, the terms "AB di-block," "ABA triblock," "ABAB tetra-block," and "AB multiblock copolymers" are used to refer to A_xB_y, $A_xB_yA_x$, $A_xB_yA_xB_y$, and $(A_xB_y)_n$, respectively. Along the copolymer chain, block lengths and block counts are distributed. Therefore, x, y, and n constitute average values. Contrarily, a graft copolymer is a branched copolymer with a backbone made of one kind of mer and multiple side chains made of a different type of mer. [3]

Polymers are classified based on the structure of the monomer chain are (linear polymers, branched-chain polymers, and cross-linked polymers); classification based on polymerization (addition polymerization and condensation polymerization); classification based on monomers (homomers): In this type, a single type of monomer unit is found. For instance, polyethene and heteropolymer or co-polymer: It is made of several types of monomer units, i.e, nylon-6,6), classification according to molecular forces (Elastomers, Fibers, Thermoplastics and Thermosetting polymers), chain growth polymerization (Free radical polymerization, ionic polymerization, coordination polymerization, living polymerization, ring opening polymerization and reversible deactivation polymerization). Chelating resins have been employed successfully in a variety of fields, including elimination of hazardous trace metal ions, due to their very selective absorptivity for thick metal ions. [5]

1.1.2 Condensation polymerization

This practically every condensation process may be used to create polymers. The essential component is a monomer, or pair of monomers, containing two or more condensable functional groups. Depending on the inter-unit linkage or the process at hand (esterification, amidation, etc.), these reactions produce polyesters, polyamides, polyethers, etc. In other instances, a longer term must be used, such as phenol-aldehyde or urea-aldehyde. [6]

There are two distinct categories of condensation polymers: linear polymers created from only
bifunctional reactants and non-linear polymers made from reactants, some of which are triunctional or higher. Representative linear condensation polymers include those produced from hydroxy acids, glycols, and dibasic acids; the latter are three-dimensional. The two different types of polymers have different structures and differing properties. [6]

Due to the nature of the process, bifunctional condensation inevitably produces linear products with a limited molecular weight. There will always be a small number of functional groups that have not completed the condensation process since it is impossible to force it to finish. The linear molecules' ends are indicated by them, and as a result, their length is limited. In the right solvents, linear condensation polymers can be dissolved. They frequently take the shape of crystals because of their regular structure. They are fusible unless the crystalline polymer's melting point is higher than the decomposition temperature. [6]

Synthesize a polymer with controlled molecular weight and low polydispersity, the polymerization should start from an initiator unit and proceed in a chain-growth polymerization manner without disproportionation or termination. This is the case for so called living polymerization. Numerous living polymerizations have been created and used since the discovery of anion living polymerization to synthesize well-defined polymers with regulated molecular weight and a restricted molecular weight distribution. These polymerization techniques have also made it possible to create polymers with a variety of forms, including star and block copolymers as well as graft copolymers, allowing for the creation of self-assembled supramolecular architectures with specific shapes and characteristics. However, live polymerization has typically only been used to add vinyl monomers to other polymers and exothermic ring opening polymers. [7]

1.3 Water pollution

Water is an essential natural resource that we utilize on a daily basis to build our lives. As a result, safe and clean drinking water is a global essential for health of humankind. Water contamination, on the other hand, is a big issue all over the world. It occurs when foreign elements contaminate clean water, causing it to lose its quality. Industry has the ability to pollute lakes, streams, and rivers. Contamination stems from different sources as industry to industry and plant to plant. Pollution resulted from industrial sector is on the rise, and it is a major contributor to the deterioration of water quality, posing a serious environmental threat around the world. [8]

A variety of extraction technologies are now used to eliminate impurities from natural water or wastewater. These procedures depend on the distribution of two phases, include liquid-liquid extraction, adsorption, precipitation, and others. Membrane and adsorption technologies are the most advanced, and they mostly apply polymeric materials. [9]

Heavy metals are a major contributor to water pollution. Industrial processes such as mining, ore processing, metal polishing, cleaning, paint manufacturing and battery manufacturing continuously release heavy metals into the aquatic ecosystem. These industries generate large amounts of effluent, which have negative consequences on human life and the environment when released into bodies of water. Furthermore, heavy metals are not metabolized and accumulate in our bodies via the biological chain.[10]

Resin polymer matrices can be synthetic or biopolymer based. Thick metal pollution removal from aqueous sources necessitates the presence of ionizable groups that ease contact with an ionic species via electrostatic forces or groups with the capacity of creating a chelate that forms coordination bonds with the metal. In such cases, chelating agents such as iminodiacetic, N-methyl-D-glucamine, amidoxime, aminophosphonic acids, thiourea, and 2-picolylamine or polymers with assemblies such as sulfonic acids, carboxylic acids, ammonium, and amine are commonly used Incorporating a moiety of a different chemical type, such as an inorganic moiety, into a polymer matrix can result in a composite. [9]

The most frequent heavy metals found in industrial effluents are copper and iron (Abuh et al., 2013; Ghosh et al., 2008). At high amounts, these metals can cause serious health problems such anemia, stomach or intestinal problems and liver damage. In addition, iron at high concentrations in water serves as a food source for bacteria, resulting in foul odors and disagreeable tastes, as well as ailments such conjunctivitis, choroiditis and retinitis (Alimohammadi et al., 2007). As for the Lead (II), it is also among the most dangerous heavy metals, producing behavioral abnormalities, learning difficulties, and seizures. Anemia has been linked to low levels of lead (II), whereas high levels induce severe kidney, liver, central and peripheral neurological system, reproductive system and blood pressure abnormalities. Hence, the World Health Organization (WHO) suggests the toxic limits of 0.0015 mg L- 1. [11]

Mercury, chromium, arsenic, lead, zinc, copper, and cadmium are the major pollutants in effluents. Chromium is among the most toxic metals, posing serious health risks to the environment. The second most common heavy metal in organic contaminants in ground water is chromium. Chromium is used in a variety of industries, including alloys, paper and pulp, basic steel works foundries, petroleum refining industries, mining, dyes and pigments, textiles, leather, tanning and electroplating. [12]

In general, chromium is present in two oxidation states: Cr(VI) and Cr(VII) (III). Trivalent chromium is required in trace amounts for sugar and lipid metabolism and is much less toxic than hexavalent chromium. One such metal ion is chromium (VI), which is both carcinogenic and mutagenic.[13] Cr(VI) poisoning can cause asthma, bronchitis, ulceration and perforation, as well as liver and kidney damage. [14] Because of its association with oxygen as chromate ions, Cr(VI) is becoming the more toxic form of the metal. It is a strong oxidizing agent that is reduced to Cr(III) more quickly in an acidic environment when there is organic matter present. [13]

Whereas, at high concentrations, Cr(VI) can get over the environment's reducing capacity and thus remains as a pollutant; its concentrated in industrial waste water with a range from 0.5ppm to 270ppm. [14]

It is critical to eliminate such a hazardous metal from the environment. Toxic metals can be removed using conventional methods such as reverse osmosis, precipitation, chemical reduction, and so on. However, these methods necessitate extensive experimental setup, are costly and have low efficiency.

Adsorption has recently been disclosed to be a promising technique to remove lead. A metal chelate forming agent that interacts specially with the heavy metal ions and a carrier matrix that can be an inorganic material or polymeric microspheres that contain functional assemblies that can bind to the heavy metal ions make up specific polymer sorbents. [15]

1.4 Hydroxamic acid

1.4.1 overview

Hydroxamic acid stands for N-acyl derivatives of hydroxylamine, indicates the substance that has the hydroxyl amide structure most readily. A substance known as hydroxamic acid is shown in Figure (1.1). Although the term hydroxamic acid (HA) is frequently used to refer to all of the mono-acyl
derivatives of hydroxylamine, the tautomeric form with hydroxylimide structure is what is actually being discussed here. [16]

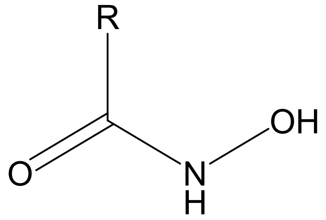


Fig. 1.1: Hydroxamic acid general structure.

Since they have the functional group that satisfies the fundamental condition of complex formation with metal ions, hydroxamic acids have been found to be significant chelating agent toward a variety of metal ions. [16]

Hydroxamic acids (HA) are classified into three categories: primary (I), secondary (III), and cyclic (IV). They can also be represented by two tautomeric forms, including keto (I) or enol (II) figure (1.2) , or by substitution of the hydrogen atom that is attached to the nitrogen atom in (I) with an aryl or alkyl group, Or you can create *N*-substituted hydroxamic acid of type (III) by substituting the hydrogen atom attached to the nitrogen atom in (I) with an aryl or alkyl group (II). Although derivatives of it are known , is most appropriately referred to as hydroxamic acids. It was expected that in the keto form Z and E-isomerism figure (1.3), the possibility of rotation around the C-N bond is restricted after studying this-trans isomerism in mono-alkyl hydroxamic acids using spectroscopy. According to NMR studies on N-substituted hydroxamic acid, the Z and E-isomeric rate is solvent dependent, with the z-isomer predominating in polar solvents. [16]

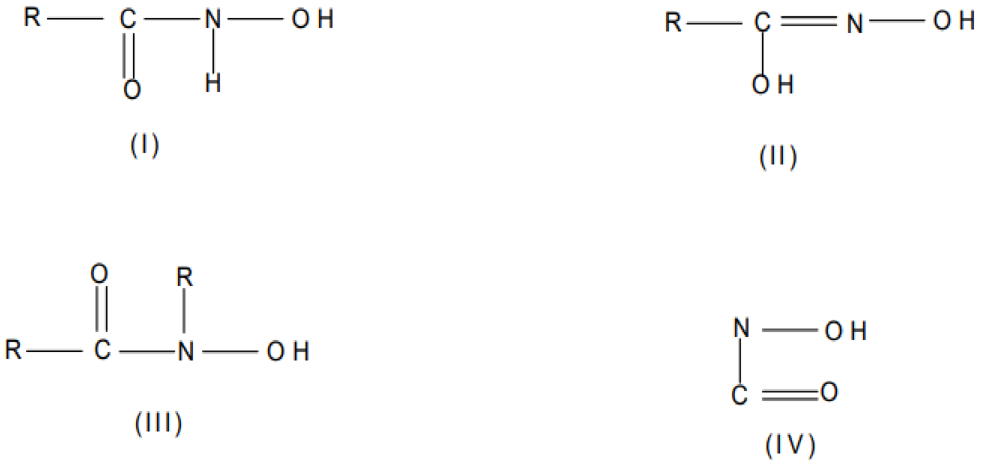


Fig .1.2: Tautomeric forms of hydroxamic acid. [16]

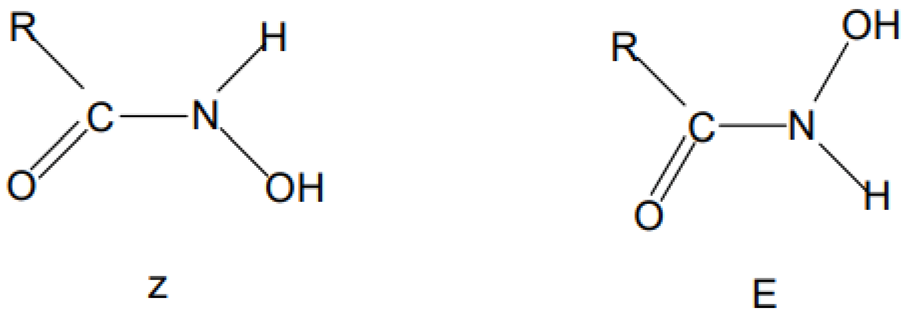


Fig .1.3: Z and E-isomeric of hydroxamic acid. [16]

Hydroxamic acids (HA) are typically synthesized using carboxylic acid and its acid chloride derivatives. Other functional groups, such as aldehyde, amine, amide, and alcohol, can be easily converted to hydroxamate. [16]

For a long time, hydroxamic acids have been known for their unique capacity to form complexes with heavy metals. Several hydroxamic acids show biological activity, including urease inhibition and anticoagulant activity. Hydroxamic acid polymers have been used as ion exchangers in sporadic reports over the last 40 years. Polymers were created by either polymerizing hydroxamic acid containing vinyl monomers or turning functional groups in polymers into hydroxamic acids. We are interested in poly (hydroxamic acids) (PHA). It occurred to that polymer with hydroxamic acid groups, like their low molecular weight analogs, might have anticoagulant or urease inhibitory activity. If this is the case, they may have a one of a kind application as a protective coating. [17]

1.4.2 Properties of hydroxamic acids

Hydroxamic acids (HA) are white crystalline solids, excluding the iodo and nitro substituted variants, which are pink and light yellow, respectively. They are slightly soluble in cold benzene and carbon tetra chloride but are soluble in alcohol, chloroform, hot benzene, diethyl ether and dioxane. They have low melting temperatures and are weak acids. Since they include a hydroxyl group, they are acidic. They are, however, more strong acids than phenols. Due to intramolecular hydrogen bonding, the acidic character was suppressed.[16]

1.5 Aim and objective

This work's aim is to prepare new polymers that can remove heavy metals.

Objectives:

The main objectives of this study are:

1. Synthesis of a novel condensation polymer based on *N^1*, *N^3*-dihydroxymalonamide and
   α, α′-Dibromo-p-xylene.
2. Characterization of the polymer by FT-IR, NMR and determination of average molecular weight by light scattering.
3. Studying the polymer's ability to remove chromium ions (Cr(VI)) from aqueous solution.

Chapter Two

Literature review

2.1 Hydroxamic Acid

Andrea et al. (2021) reported a protected hydroxylamines, such ethers or silyl ethers, are being used more often, demonstrating their considerable benefit in producing hydroxamic acids in high yields while avoiding unfavorable side effects. The most straightforward technique for producing HAs uses carboxylic acids as starting materials that have been appropriately activated as an ester or acyl chloride to perform a nucleophilic acyl substitution reaction with hydroxylamine. Contrary to the opposite reaction, the stronger nucleophilic nature of N along with the amide that results' better stability compared to the ester results in the production of nearly entirely hydroxamic acid. The transformation of many additional functional groups into hydroxamic acids, however, can be achieved in a few or many steps. [18]

Acid – base behavior: While the related amides are often orders of magnitude less acidic, the average pKs values vary from 7 to 9. Deprotonation can take place either at the amide N-H or the hydroxyl group in their unsubstituted NH-OH form. In DMSO or the gaseous state, deprotonation at the N-H group is preferred, but in water or alcohols, deprotonation at the hydroxyl group, analogous to carboxylic acids, is more prevalent. Due to certain metal ligand interactions, double negatively charged hydroxamic acids that are concurrently deprotonated at the amide and hydroxyl groups are only sometimes known.[19]

Reactivity and chemical stability. Under very acidic or basic conditions, hydroxamic acids don't show limitless stability. Under these conditions, their chemical behavior is extremely similar to that of the associated amides, carboxylic acids, or carboxylates.[19]

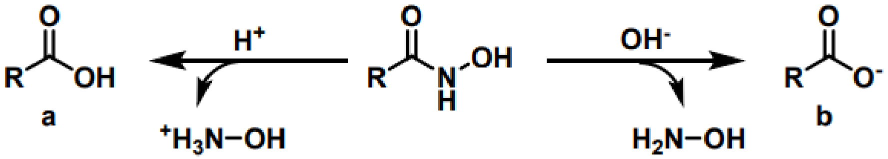


Fig. 2.1: Hydroxamic acid reaction pathway in highly acidic or basic conditions. [19]

Complexity properties. Well-known for their excellent complexation abilities are hydroxamic acids. By using O, O-coordination, hydroxamic acids often form complexes as bidentate ligands that

produce extremely stable five-membered ring structures. Compared to carboxylic acids, which only form a 4-membered ring, their stability is several orders of magnitude higher figure (2.2). Only the

single deprotonated Z tautomer, which is produced dynamically by the tautomer equilibrium, may initiate chelation.[19]

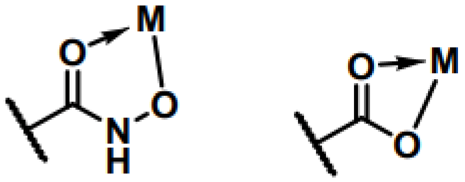


Fig .2.2 : Structure of hydroxamic acids' coordination group and carboxylic acid . **[19]**

hydroxamic acid. In nature, hydroxamic acids are essential. There are known mono, bis, and tris hydroxamic acids. They are a crucial chelator in iron metabolism because of their capacity to form extremely stable complexes with iron. They are regarded as one of the two main kinds of siderophores that can complex Fe (III), allowing microbial iron transfer. Life cannot exist without iron. It serves as the crucial element for the redox reactions that different enzymes must carry out in order to transport oxygen. Iron is extremely abundant in nature, yet it is also only present as highly water insoluble compounds, severely limiting its bioavailability. Both microorganisms and plants employ hydroxamic acids to solubilize iron and promote iron uptake in order to get around this problem. They are also present in proliferation factors, tumor inhibitors, antibiotics, antibiotic antagonists, and growth factors. Ferrichrome is a well-known example of a naturally occurring hydroxamic acid and is generated by a number of fungi belonging to the Aspergillus, Ustilago, and Penicillum families. [19]

2.2 Synthesis of di-hydroxamic acid chelating polymers and the adsorptive property for uranium in sea water.

Takahiro Hirotsu et al. (1986) reported the preparation of new chelating polymers with di-hydroxamic acid groups, as well as their uranium adsorptive ability in sea water. Chloromethylated polystyrene crosslinked with divinylbenzene was treated with diethyl malonate in *N*, *N*-dimethylformamide to produce a polymer with diethyl malonate groups, The di-hydroxamic acid polymer contained both carboxylic acid and hydroxamic acid groups, and the appearance of an IR absorption band at 1680 cm^{-1} confirmed the presence of hydroxamic acid groups.[19]

Uranium exists in sea water as the chemical species uranyl(V1) tricarbonate ion ($UO_2,(CO_3)\ )_3{}^{4-}$), which is very stable and has a concentration as low as 3 μg-U/L. As a result, ligands with high uranium selectivity are required to develop chelating polymers for uranium recovery.Chelating resins have been used successfully in a variety of applications, including the removal of harmful trace metal ions, due to their highly selective adsorptivity for heavy metal ions . [19]

By treating polymers with diethyl malonate groups with hydroxylamine, new chelating polymers with di- hydroxamic acid groups were created. In sea water, the polymers showed selective uranium
adsorptivity. The effect of polymer support morphology on uranium adsorption rate was also
investigated. [19]

2.3 Polyhydroxamic acid functionalized sorbent for effective removal of chromium from ground water and chromic acid cleaning bath.

The chemical modification of polyacrylamide resulted in the creation of a poly (hydroxamic acid) sorbent (PHA), which was then characterized by FTIR. Trivalent and hexavalent chromium ions were removed from an aqueous solution using the synthesized sorbent. In-depth research was done on how pH affected the sorption of Cr (III) and Cr (VI) in the pH ranges of 1–7 and 1–12, respectively. Beyond pH 7, Cr (III) exists as $[Cr\ (OH)_3]^0$ and forms an amorphous precipitate, making it im-possible to study its sorption behavior. The highest sorption for both Cr (VI) and Cr (III) was found to occur at pH 4. Even at lower pH levels, Cr (VI) was shown to exhibit noticeable sorption. The sur-face of the sorbent is positively charged at lower pH due to strong protonation, and since Cr (VI) occurs as the negatively charged hydrogen chromate ($HCrO_4^-$) species, its sorption is facilitated. The ($HCrO_4^-$)ultimately transforms into the divalent (CrO_4^{2-}) and then into ($Cr_2O_7^{2-}$) when the pH increases from 4 to 12. In addition, under weak acidic conditions, the protonation degree of the sorbent surface re-duces, which reduces the electrostatic attraction between more negatively charged ions and the sorbent surface. As a result, maximal sorption of Cr (VI) by PHA occurs at pH 4, but total sorption is greater at lower pH. [20]

On the other hand, PHA and Cr (III) ions exhibit electrostatic repulsion between PHA and Cr(III) ions because Cr(III) occurs in +3 oxidation states at lower pH (3) in aqueous solution. This prevents Cr (III) from sorption on sorbent surfaces at low pH levels. The surface group may be partially deprotonated at higher pH values, which enhances Cr (III) sorption. [20]

2.4 Synthesis and properties of Poly(Hydroxamic acid) from crosslinked Poly (MethaCrylate)

Haron et al. (1994) reported a describes a method for creating a polymer with hydroxamic acid groups from crosslinked polymers (methacrylate). The conversion of the starting material to the resin occurs in a single step, and the product is in the form of beads with a reasonable high hydroxamic acid capacity. The properties of this resin have been studied, and it can be used for lead-nickel separation, which is a novel application for a resin with a hydroxamic acid functional group. Whereas crosslinked poly(methacrylate) beads were used to make poly (hydroxamic acid) chelating ion-exchange resin. A suspension polymerization of methacrylate and divinyl benzene was used to create the starting polymer. The ester groups were converted into hydroxamic acid by treating them with hydroxylamine in an alkaline solution. The product's hydroxamic acid capacity was 2.71 mmol/g. The properties of this chelating ion exchange resin were investigated based on its ability to separate metal ions using a column technique. The outcome of the lead-nickel separation is given. The separation was achieved by selectively eluting nickel ions from the column with acetate buffer (pH 3.5) and lead ions with 4M nitric acid solution. This is a novel separation. [21]

Chapter three

Methodology

3.1 Chemicals

Chemicals including Diethyl malonate 99%, hydroxylamine hydrochloride 99%, potassium hydroxide 99%, α, α′-Dibromo-p-xylene 99%, Potassium dichromate ($K_2Cr_2O_7$) were purchased from chemicals-selling store (sigma -Aldrich).

Solvents: Methanol 98% (Sigma-Aldrich), Dimethylformamide (DMF) 99% (sigma Aldrich), Hydrochloric acid (HCl) 99% (sigma- Aldrich) and distilled Water (H_2O).

3.2 Instruments

Instruments used are nuclear magnetic resonance (NMR) (BRUKER 300 MHz) Genève University/Switzerland, Fourier transform infrared spectroscopy (FT-IR) (BRUKER-Tensor Ⅱ) Abu-Dies/Al-Quds University, uncle instrument which is static light scattering (SLS), ZURICH University/Switzerland, Atomic absorption spectrometer (Perkin Elmer), pH – meter (HANNA) and Differential Scanning Calorimetry (DSC) (Perkin Elmer).

3.3 Procedure

3.3.1 Preparing of di-hydroxamic acid salt of diethyl malonate:

FIESER et al. (1967) procedure for synthesis of general hydroxamic acid was

used in this
research to synthesize N^1, N^3-dihydroxymalonamide monomer [22]. 46.7 gr. of hydroxylamine hydrochloride (2.8M) was transferred to a beaker then 240 ml of methanol was added. In another beaker, 56 gr. of potassium hydroxide (7.128M) in 140 ml of methanol was prepared and boiled. The two solutions cooled to 30-40 C°, and the second solution was added to the first. After cooling in ice to ensure complete separation of potassium chloride, 26 ml of diethyl malonate was added with shaking then the mixture was filtered quickly.

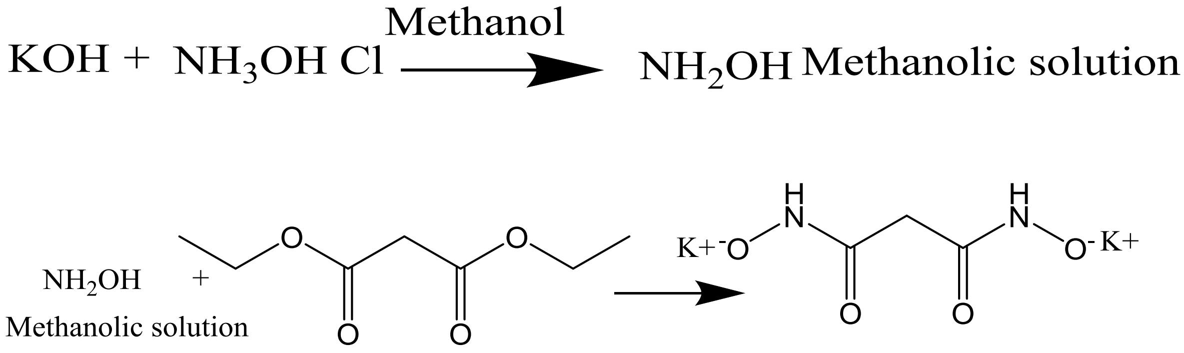


Fig.3.1: Chemical reactions of synthesis of di-hydroxamic acid salt of diethyl malonate.

3.3.2 Condensation polymerization:

5.67 g of monomer are transferred to a 50 ml round bottom flask (RBF) then 83 ml of dimethylformamide (DMF) was added to the RBF and stirred for 24 hrs. Then 5.67g α,α′-Dibromo-p-xylene was added to the reaction mixture. The round bottom flask (RBF) was placed in an oil bath at 55°C overnight. The color changed to yellow.
The percent yield of linear polymer = 61.8 %

Fig .3.2: preparing polymer by condensation polymerization.

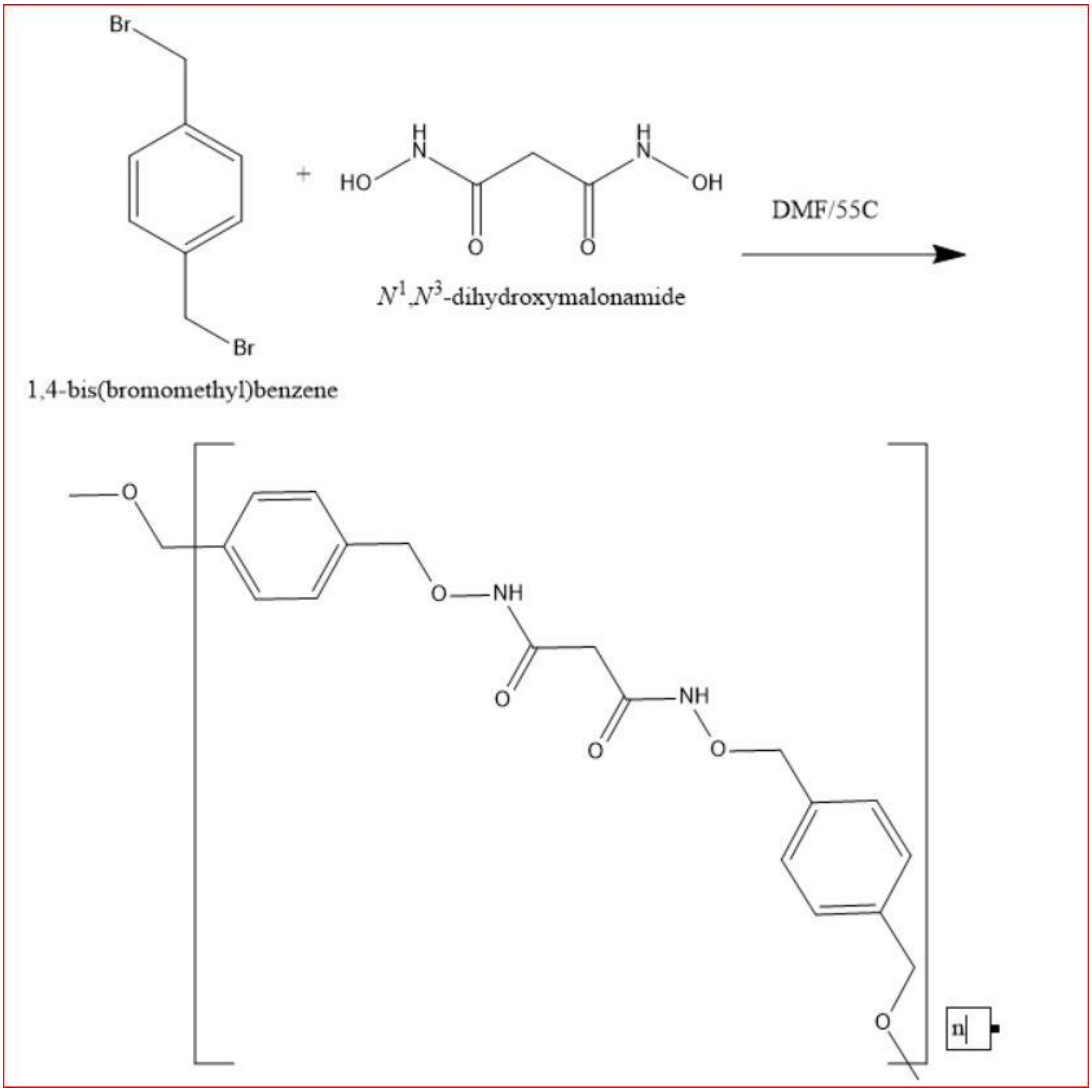


Fig. 3.3: *N*^{1}, *N*^{3}-dihydroxymalonamide then was reacted with α,α′-Dibromo-p-xylene to produce a new polymer by condensation polymerization. [23]

3.3.4 Preparation of Cr(VI) stock solutions

A 100ppm chromium (VI) stock solution was prepared by dissolving the appropriate amount of $K_2Cr_2O_7$ (0.373 g) in ionized distilled water in a 100 ml volumetric flask. The solutions were prepared in various concentrations (10 ppm, 5 ppm, 2.5 ppm, and 1.25 ppm) in a 50 ml volumetric flask by diluting an appropriate volume of stock solution in distilled ionized water. An absorption curve was constructed for each solution using atomic absorption spectrometer and a calibration curve was read.

3.3.5 Removal of Cr(VI) at different pH

A new sample was prepared at a concentration of 10 ppm in a 100 ml volumetric flask and the pH was adjusted to 3 and 5. The solutions were stirred for two weeks at 25°C, and then filtered. The concentration of Cr(VI) ions was determined by atomic absorption spectrometer.

Chapter four

Result and Discussion

The spectra of both materials were used to confirm the conversion of diethyl malonate to *N*^{1}, *N*^{3}-dihydroxymalonamide.

4.1 Analysis of FT-IR spectra:

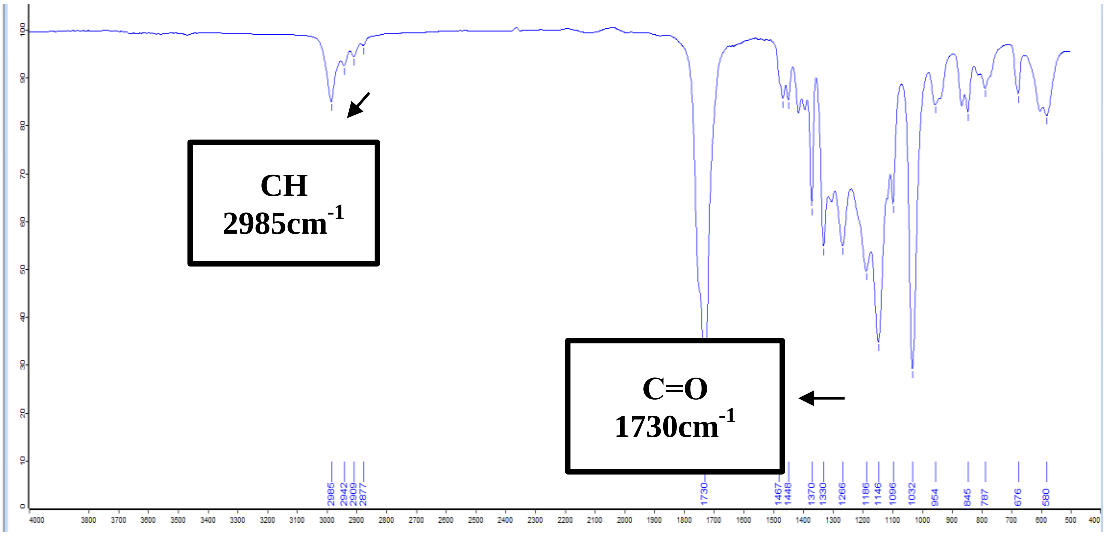


Fig .4.1: FT-IR spectrum for diethyl malonate.

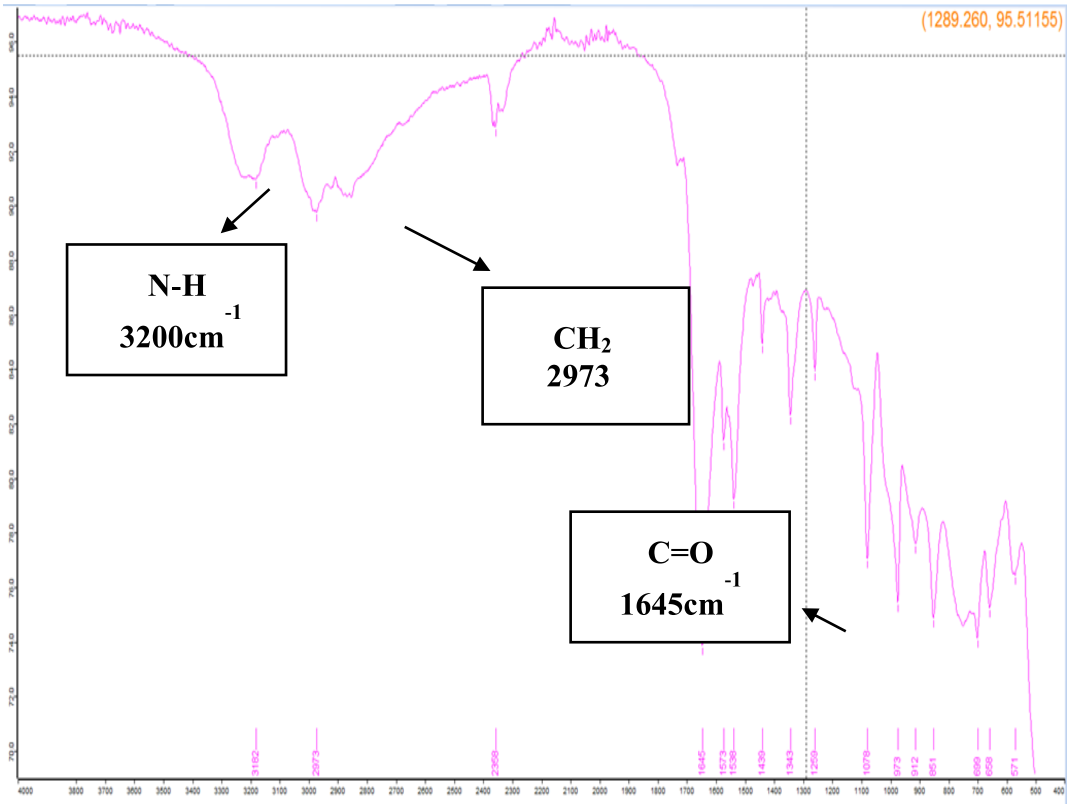


Fig .4.2: FT-IR spectrum for *N*^{1}, *N*^{3}-dihydroxymalonamide.

The FTIR spectra of the diethyl malonate are presented in Figure (4.1). The C=O band at around 1730cm^{-1}. In addition, the peak is 2985 cm^{-1} for C–H stretching. While the FTIR spectra of the *N*^{1}, *N*^{3}-dihydroxymalonamide in Figure (4.2) showed the NH stretching band was around 3200 cm^{-1}, and the C=O shifted from 1730cm^{-1} to 1645 cm^{-1}, the existence of the hydroxamic acid functional group was confirmed.

Table.4.1: The frequencies that have been detected confirm the presence of the hydroxamic acid functional group

Bond	**Frequency (cm^{-1})**
C=O	1645
C−H	2973
N−H	3200

The FTIR spectra of the polymer is shown in Figure (4.3). We observed the presence of NH and C = O in the FT-IR spectrum for the polymer. It is apparent from these results that the FT-IR spectrum indicates a successful polymerization process and that 1650 cm^{-1} refers to the C=O. The N-H stretching band was around 3300 cm^{-1} and the band at around 2900 cm^{-1} refers to stretching C-H.

Table 4.2: Frequencies detected in the respective polymer functional groups

Bond	**Frequency (cm^{-1})**
C=O	1650
N−H	3300
C−H	2900

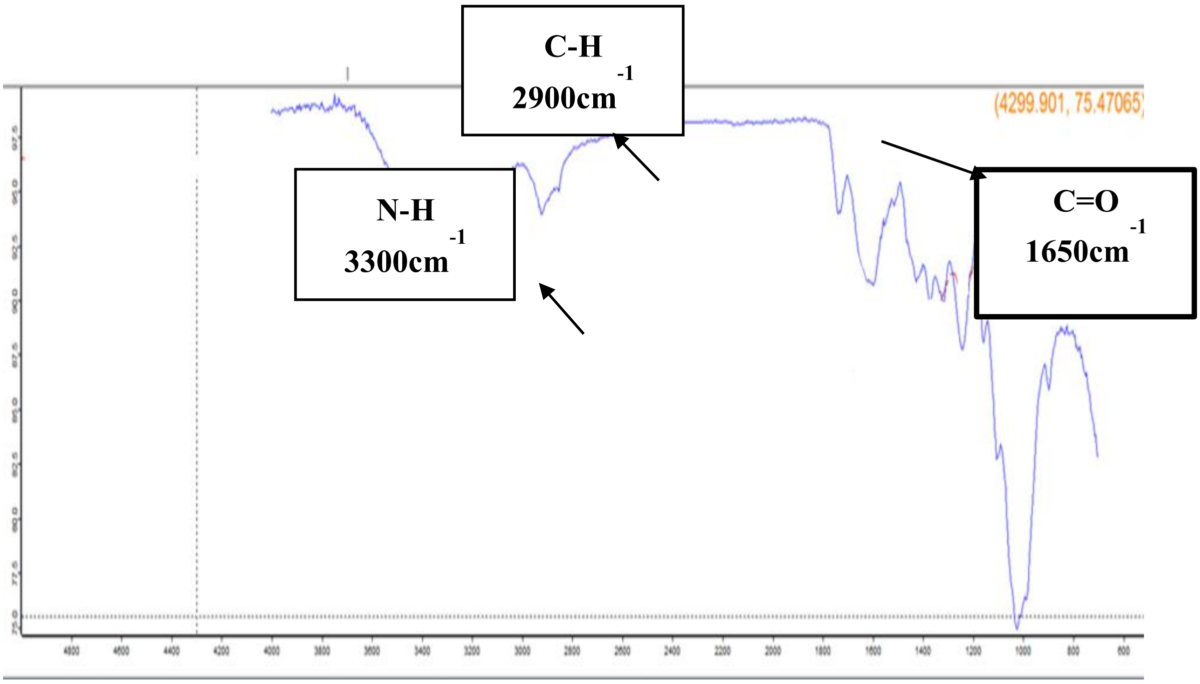


Fig. 4.3: FT-IR spectrum for polymer.

The dimer was hydrolyzed to confirm the presence of hydroxamic acid by the presence of an amine group, then it was refluxed with 6 molars of HCl for 6 hours, the solvent from the reflux process was evaporated, causing recrystallization with methanol, and the sample was analyzed by FT-IR.

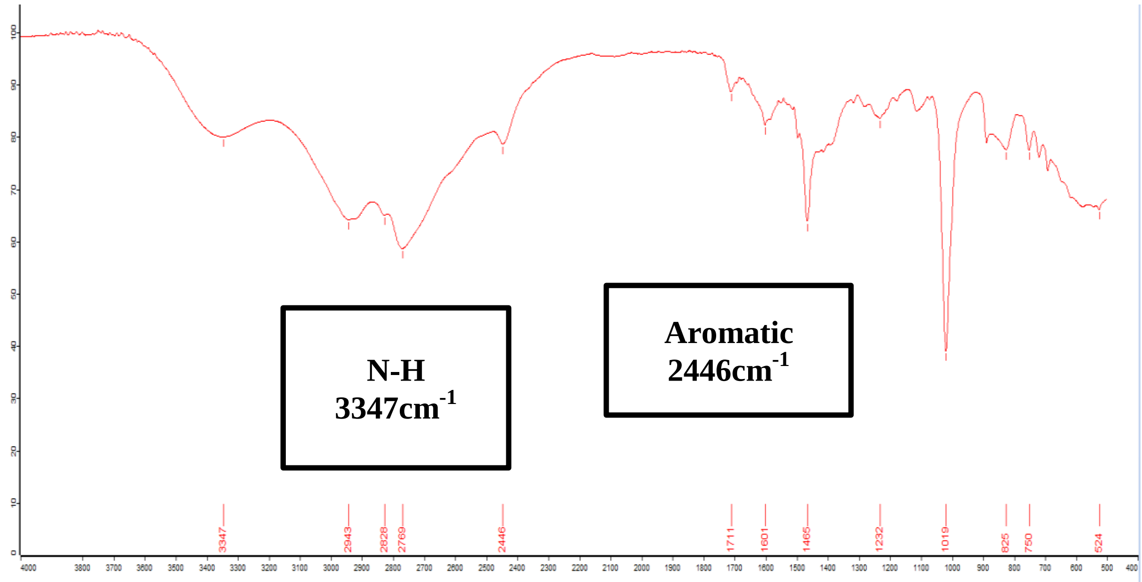


Fig. 4.4: FT-IR spectrum for hydrolyze product.

The FTIR spectra of the hydrolyzed product are shown in Figure (4.4). The N-H stretching band was around 3347cm^{-1} and the band at around 2446 cm^{-1} refers to stretching the aromatic ring. It is apparent from these results that the FT-IR spectrum indicates a successful polymerization process and the presence of hydroxamic acid.

4.2 NMR analysis:

NMR spectroscopy is used to characterize polymers and gives detailed structural information for polymer development. Polymer NMR: monomer kinds, levels, distribution (block or random), sequence branching, sequence, polymer techniques, molecular weight (MW), and end groups are all evaluated using polymer NMR. The structure and properties of the co-monomer are also discussed [27].

Figure (4.5) shows the 1H NMR spectrum for the monomer. We observed that the monomer was freely dissolved in D_2O. As a result, C-H is clearly seen in 1H NMR and the hydrogen shifted to (4.72 ppm), so the signal at (4.72 ppm) refers to C-H. In the case of hydrogen in N-H, it did not appear in the D_2O, we also found out that in N–H, it was not clearly seen in the ^{1}H NMR and in the part of the peak at (2.8 ppm) returned to the impurities.

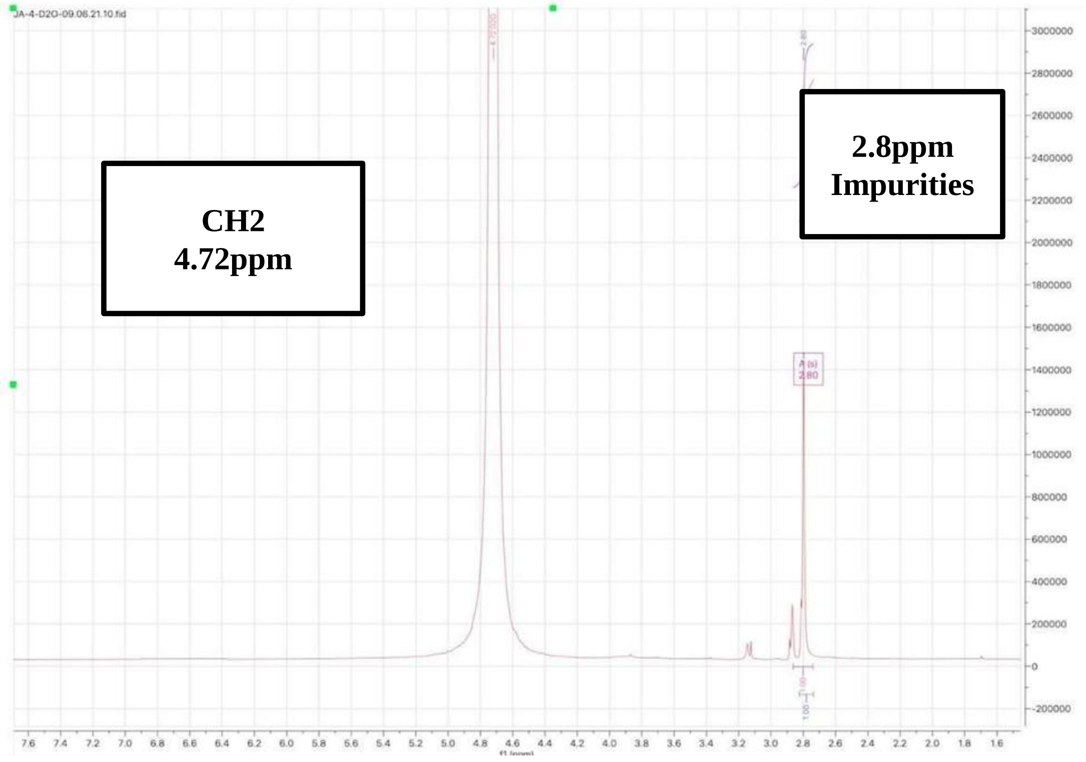


Fig. 4.5: ^{1}H NMR for *N*^{1}, *N*^{3}-dihydroxymalonamide.

The 13-C NMR spectrum of the *N*^{1}, *N*^{3}-dihydroxymalonamide that was analyzed by D_2O as a solvent is shown in Figure (4.6). (CH) is clearly seen in 13-C NMR appeared in (37.35 -37.56 ppm), signals at (164.25-166.61 ppm) refer to C=O, and we also found that the peak at 175 ppm and 179.48 ppm returns to the impurities.

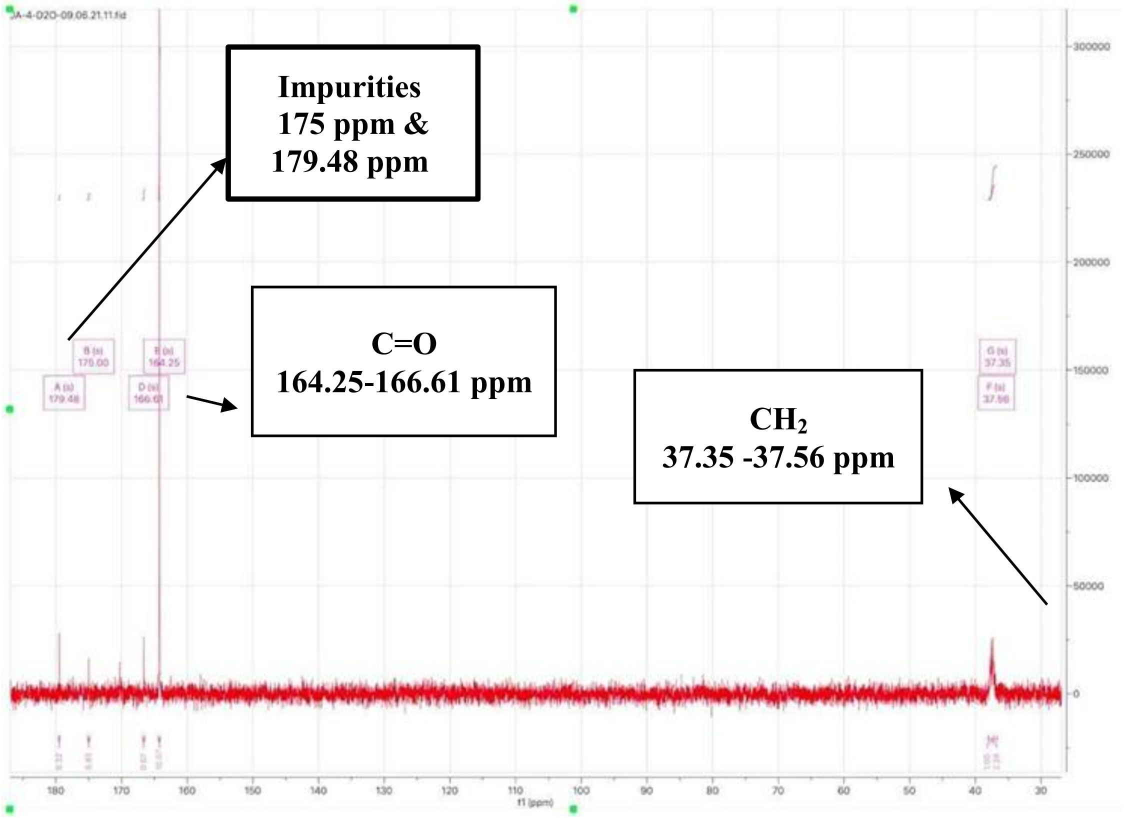
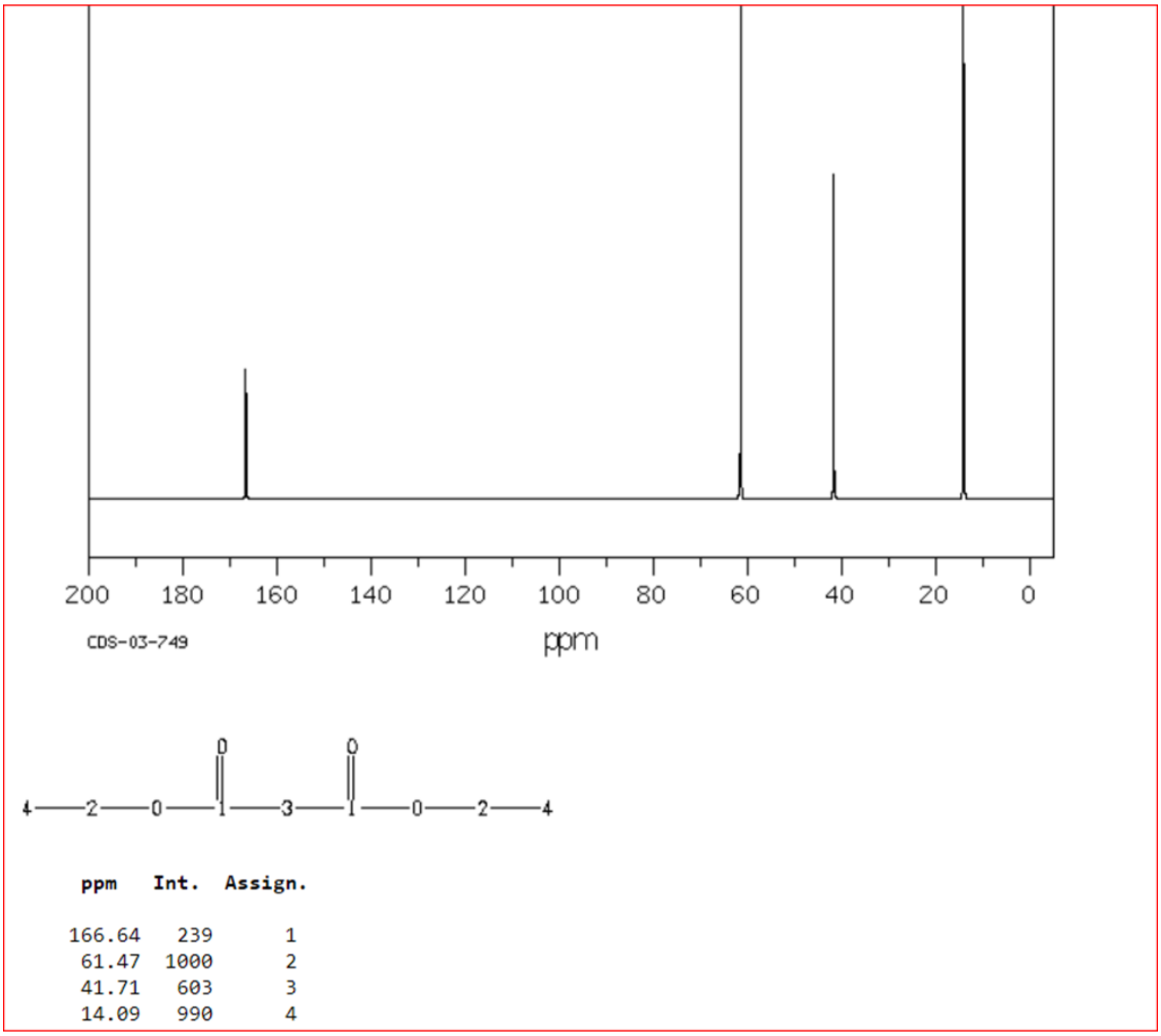


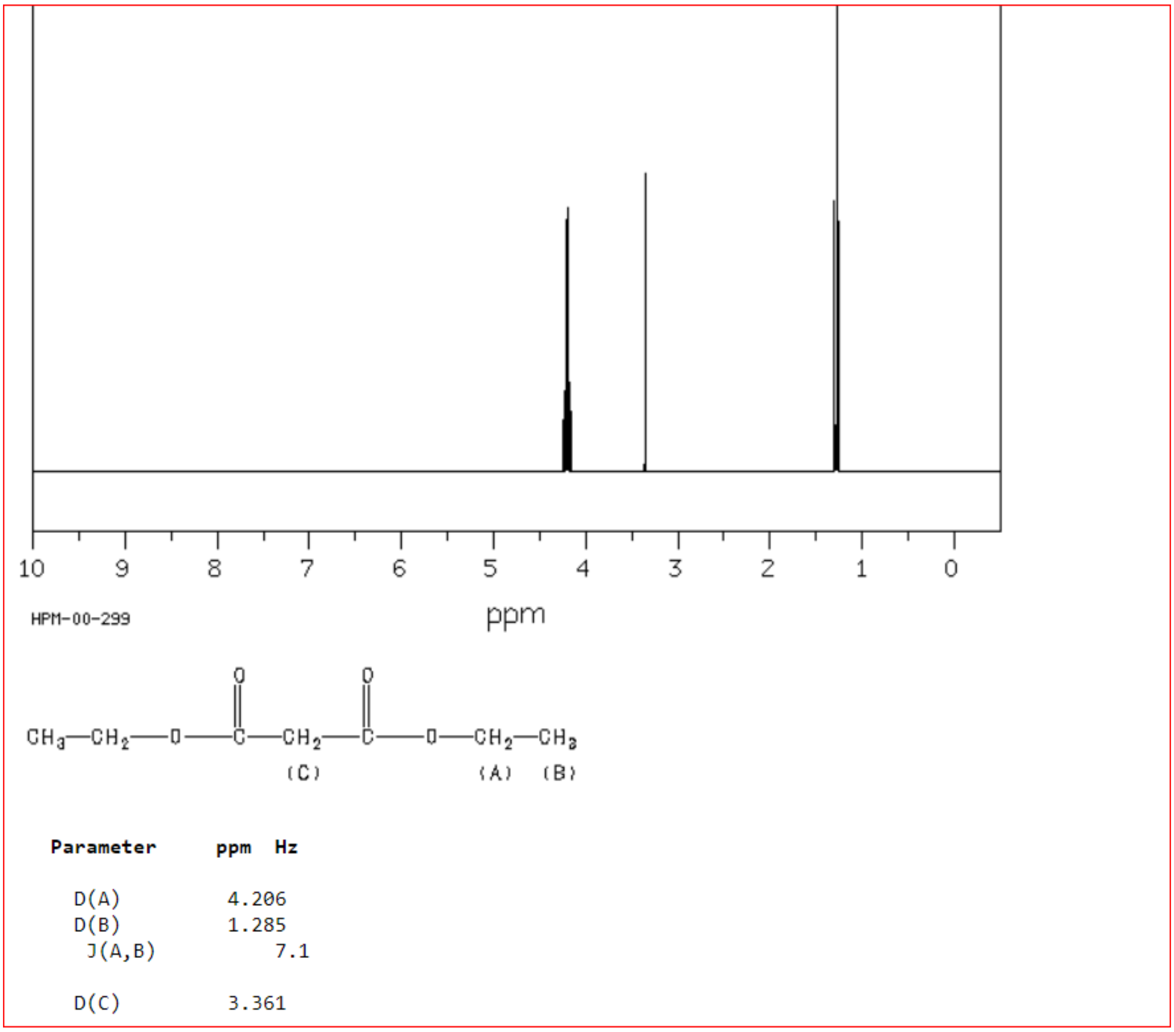
Fig. 4.6: 13 - C NMR for *N*^{1}, *N*^{3}-dihydroxymalonamide.

The NMR spectrum of the diethyl malonate that was compared with the NMR spectrum of *N*^{1}, *N*^{3}-dihydroxymalonamide The 13-C NMR spectrum of the diethyl malonate that was analyzed by deuteated chloroform ($CDCl_3$) shows that C=O is clearly seen in the 13-C NMR at 166.64 ppm (figure 4.7), CH_2 showed up between C=O at 41.71 ppm, and the ethyl group (CH_2 CH_3) appeared at 61.47 ppm and 14.09 ppm, respectively. The ethyl group (CH_2CH_3) was observed at 4.206 ppm and 1.285 ppm in 1H NMR of the diethyl malonate figure (4.8), the CH_2 appearing between C=O at 3.361 ppm, the hydroxamic acid was found and confirmed to be present by CH at 4.72 ppm in 1H NMR, shown in figure (4.5), while it appeared in 13-C NMR, figure 4.6, at 37.35–37.56 ppm with the ethyl group disappearing.



ppm	Int.	Assign.
166.64	239	1
61.47	1000	2
41.71	603	3
14.09	990	4

Fig .4.7: 13-C NMR of diethyl malonate. [24]



Parameter	ppm	Hz
D(A)	4.206	
D(B)	1.285	
J(A,B)		7.1
D(C)	3.361	

Fig .4.8: 1H NMR of diethyl malonate. [24]

The 1H NMR spectra of polymer figure (4.9) is under investigation. The polymer was slightly soluble in deuterated chloroform ($CDCl_3$). It is observed in 1H NMR clearly because it doesn't require a high concentration. It contrasts with the 13-C NMR, which appears to be noisy because it is slightly soluble and requires high concentration to be clearly seen. As a result, the CH shows the characteristics of the aromatic hydrogens in the region of 4.73 ppm. Taking into consideration the failure of the polymerization process to be when CH protons appear in the 4.5 ppm region clearly.

The CH was found in the range of 3.11 ppm, positioned between two carboxyl groups. Because all the hydrogens in the ring are equivalent and returned in the form of a single peak, the signal at 7.20 ppm refers to aromatic hydrogen. The peaks at 2.75 ppm, 7.40 ppm, and 9.18 ppm return to the

impurities. This is attributed to the presence of impurities in the molecule since it is not in its pure form.

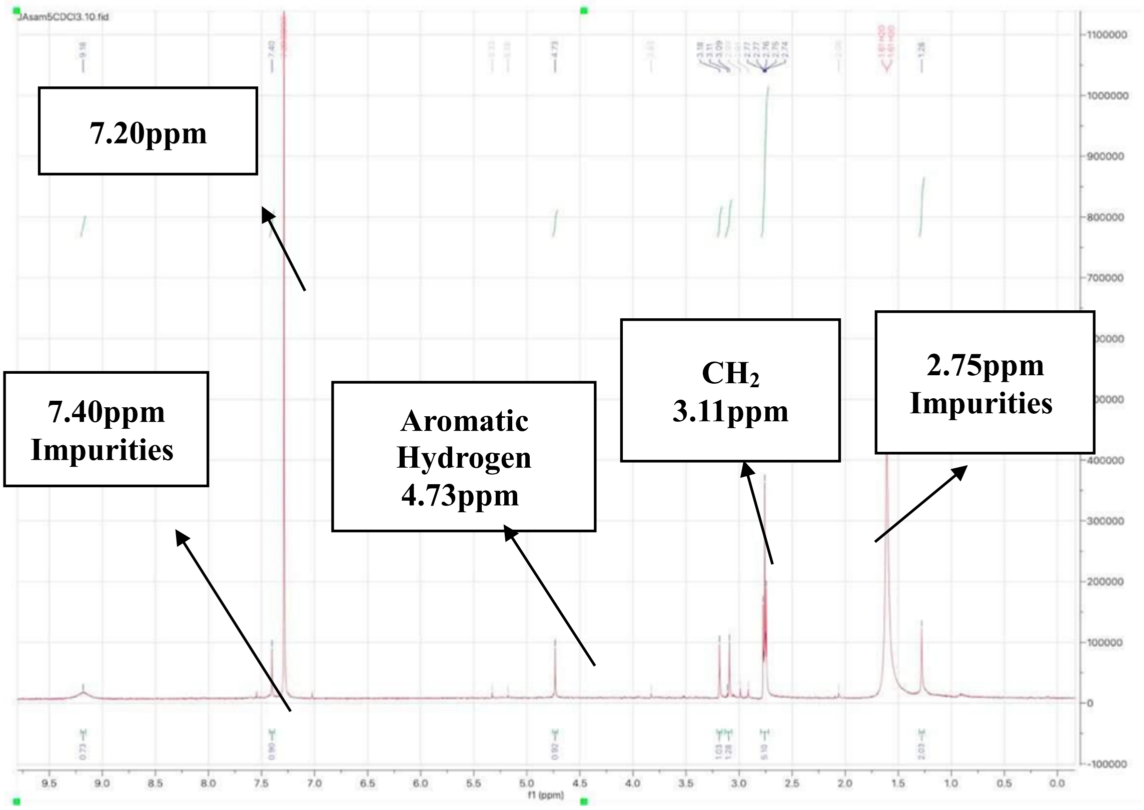


Fig. 4.9: 1H NMR for polymer.

4.3 Characterization by Light scattering:

The average molecular weight of each sample was assessed by light scattering Figure (4.10) at 20 °C. The molecular weight of the polymer after condensation polymerization (which took 24 hours) was 728,000 kDa. The cryoscopic osmometric method was applied to determine the average molecular weight of these polymers. The polymer's sticky and viscous appearance is due to its high melting point. Because the chains become more intertwined as they become longer, it becomes more difficult to make them flow. as shown in the table (4.3).

Table 4.3: characteristic of polymer by SLS and DSL

Peak of interest Est. MW (kDa)	728000
Temperature (C°)	28.22
PDI	0.231

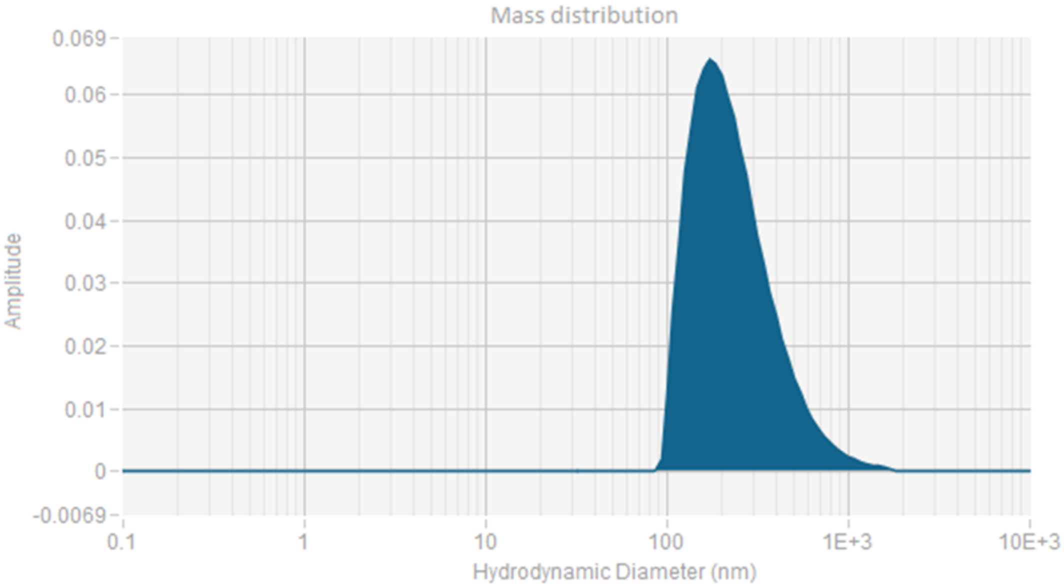


Fig. 4.10: Mass distribution of polymer by condensation polymerization.

The distribution of molecular weight is a key characteristic of a polymer since it has such a large impact on the polymer's properties, and there are several ways of determining molecular weight: Currently, membrane osmometry, viscometry and light scattering and spectroscopy are available. Static light scattering is used to calculate the polymer's molecular weight (SLS). It is the most accurate method for estimating a polymer's molecular weight since photons are absorbed and re-emitted or scattered in all directions when a macromolecule is irradiated by an incoming photon beam. [25]

Light scattering indicated that the polydispersity index (PDI) for the polymer was 0.231. (Table 4.3). The International Standard Organizations set PDI < 0.05 for monodisperse samples and > 0.7 for broad size values, such as polydisperse samples (ISOs).

Since no one, according to our knowledge, prepares our polymer, we can compare it with the molecular weight of polyamide, which is very close to its chemical structure. The average molecular weight of polyamide is 61.100 g/mol. A larger molecular weight and a doubled chain length are the results of longer cumulative build times and build times for one build process. In addition, the time of reaction reaches 26 hours for poly(amide). [30] The average molecular weight of our polymer is 728000g/mol. The most important factor that determines the high molecular weight type of functional group is that the aminooxy group is a very reactive nucleophilic group and the dibromide is a very good
electrophile. The average molecular weight depends on the concentration and time of reaction.

N^1, N^3-dihydroxymalonamide was prepared by reacting diethyl malonate with a hydroxylamine in methanol. Which was highly soluble in water. This compound has the advantage of being a good nucleophile and is capable of forming complexes with a variety of metal cations. The monomer was then reacted with dimethylformamide (DMF) to produce the polymer, which was insoluble in water but very soluble in Dimethyl Sulfoxide (DMSO).

4.4 Differential Scanning Calorimetry (DSC) Analysis

The DSC curves of the monomer in Figure (4.11) (N^1, N^3-dihydroxymalonamide) were prepared by reacting diethyl malonate with hydroxylamine in methanol. The polymer exhibited a melting point (Tm) of 164.74 °C and the delta H of fusion was 74.5705 J/g in figure 4.10, whereas the polymer exhibited a melting point of 148.00 °C and the delta H of fusion was 0.1958 J/g. There is a glass transition temperature (Tg) of 72.96 °C in figure (4.12). Furthermore, because of the intermolecular interactions on the structure, the N^1, N^3-dihydroxymalonamide has a higher melting point than the polymer.

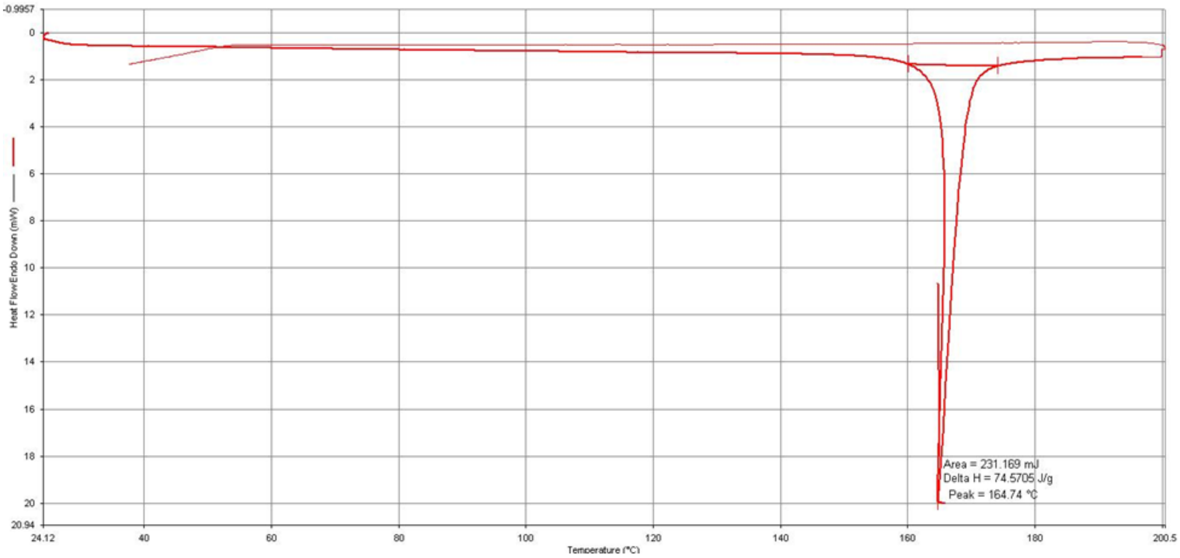


Fig. 4.11: DSC curve of N^1, N^3-dihydroxymalonamide.

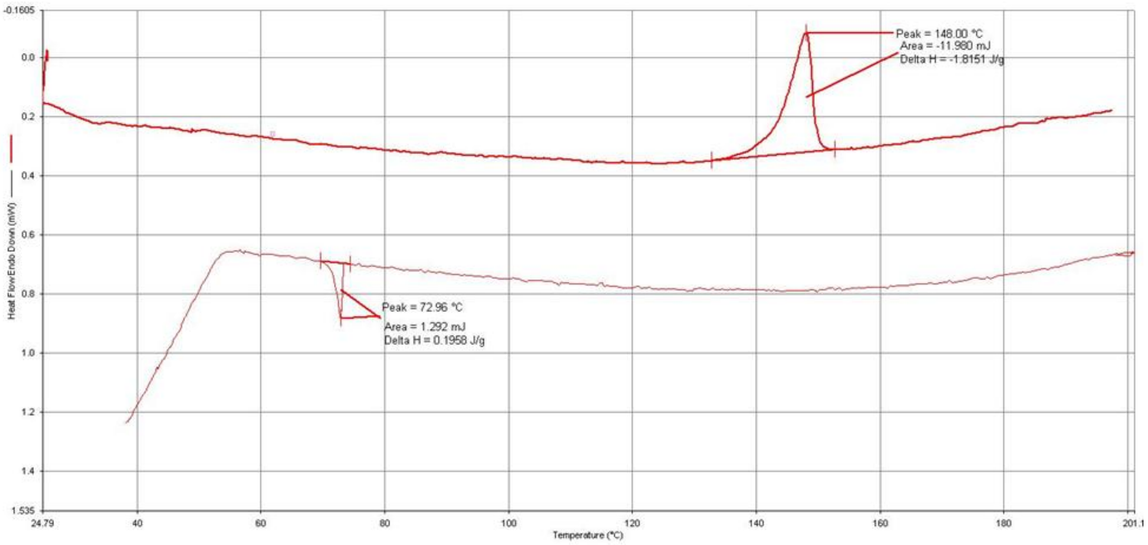


Fig. 4.12: DSC curve of polymer.

4.5 Removal of Cr(VI) by polymer at different pH

4.5.1 Calibration curve of Cr(VI)

After adding the polymer to Cr (VI) solutions of different concentrations, the absorbance of each

solution was measured. The obtained absorbance values were plotted against Cr(VI) concentration (ppm) to create a calibration curve. Figure (4.13). The curve was linear, and the correlation coefficient

(R^2 = 0.98) indicated that the parameters had a strong positive linear relationship. The linear equation of the curve and the absorbance by atomic absorption were used to calculate the Cr(VI)

concentrations.

Table (4.4.1): Calibration Curve of Cr(VI)

Concentration (ppm)	**Absorbance**
20	**0.1465**
10	**0.094**
5	**0.0597**
2.5	**0.0373**

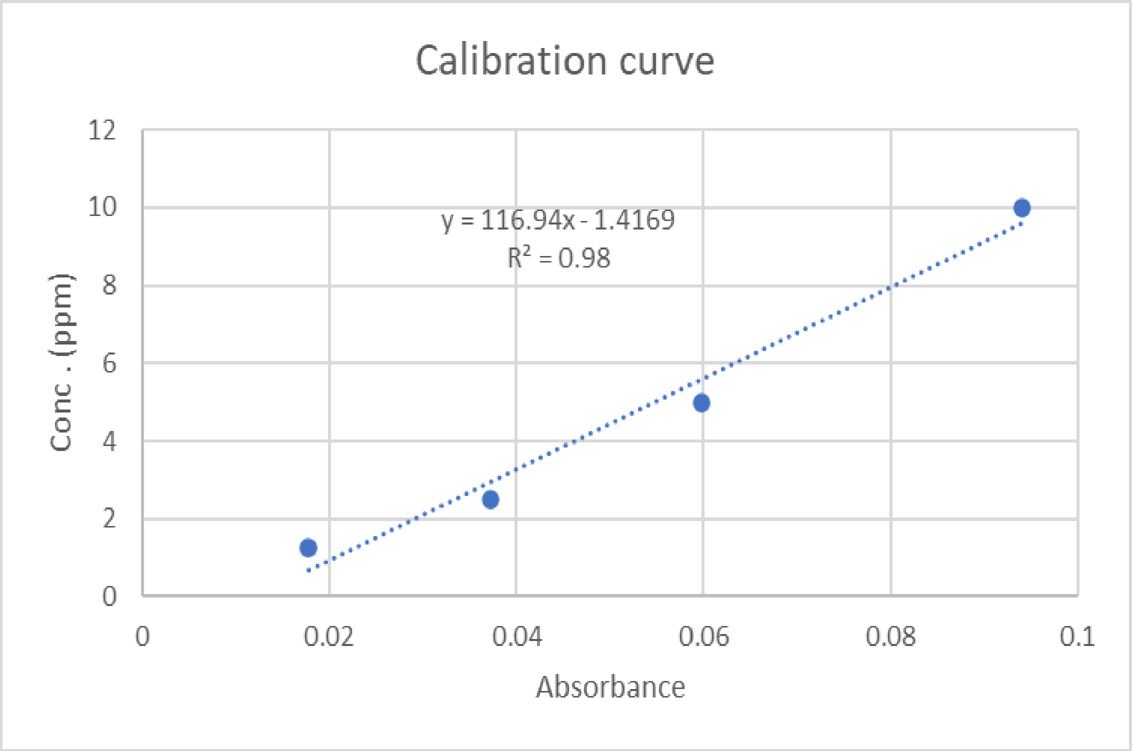


Figure 4.13: Calibration curve for the determination of Cr(VI).

4.5.2 Effect of pH

Table (4.4.2): The result of absorbance with different concentrations at pH 5

Concentration (ppm)	**Absorbance**
9.6066	0.0857
9.2787	0.0837
9.8852	0.0874
Average = 9.5902 ppm Standard deviation = 0.3036	

Table (4.4.3): The result of absorbance with different concentrations at pH 3

Concentration (ppm)	**Absorbance**
7.4918	0.0728
7.9180	0.0754
8.2459	0.0774
Average = 7.8852 ppm Standard deviation = 0.3781	

4.5.2.a) The determination concentration of Cr (VI) at pH 5

After determining Cr(VI) at pH 3 with an initial concentration of 10 ppm, After taking 25 ml of the solution, the final concentration (at equilibrium) was 9.5902 ppm after 10 days. When we subtract the initial concentration (10 ppm) from the final concentration (9.5902 ppm) in order to calculate the

Cr(VI) concentration eliminated, the result is 0.4098 ppm in a 100 ml solution as the removed

concentration, and the result is 0.1024 mg in a 25 ml solution. Therefore, 0.1024 mg of Cr(VI) was eliminated when 0.25 g of polymer was introduced. Thus, 0.0409 mg of Cr(VI) will be removed by 1 g of polymer.

4.5.2.b) The determination concentration of Cr (VI) at pH 3

After determining Cr(VI) at pH 5 with an initial concentration of 10 ppm, After taking 25 ml of the solution, the final concentration (at equilibrium) was 7.8852 ppm after 10 days. When we subtract the initial concentration (10 ppm) from the final concentration (7.8852ppm) in order to calculate the Cr(VI) concentration eliminated, the result is 2.115 ppm in a 100 ml solution as the removed concentration, and the result is 0.5287 mg in a 25 ml solution. Therefore, 0.5287 mg of Cr(VI) was eliminated when 0.25 g of polymer was introduced. Thus, 0.2115 mg of Cr(VI) will be removed by 1 g of polymer.

The highest chromium ion adsorption capacity was 0.2115 mg at pH 3.0. The adsorption capacities of Cr (VI) decreased from 3.0 to 5.0 with increasing starting pH values, however. At pH 3.0, the amine and hydroxyl groups on the adsorbent surface became protonated with increasing H+ to (NH_3^+, OH^+), forming a positively charged surface. These positive charges have an efficient adsorption capacity because they strongly electrostatically attract the anionic species of Cr (VI) in the solution. The deprotonation of functional groups, which results in a negatively charged surface, resulted in a decrease in adsorption capacity when the pH was greater than 3.0. This causes competition and electrostatic repulsions between the OH groups and chromium, which makes it difficult to adsorb or have a bent surface. In this experiment, an ideal pH of 3.0 was selected.

After preparing hydroxamic acid from an ester in a basic medium, it is in the form of ions which are deprotonated (O-), so it cannot perform complexation in a basic medium, so it cannot give color complexes because the metals can precipitate in the form of hydroxide and not act as chelators. Thus, it was converted to an acidic medium to give the color complex. This was proven by adding $FeCl_3$ and giving a gray color. In addition to the possibility of having an OH group, it is possible to chelate these with the metals.

Chapter Five

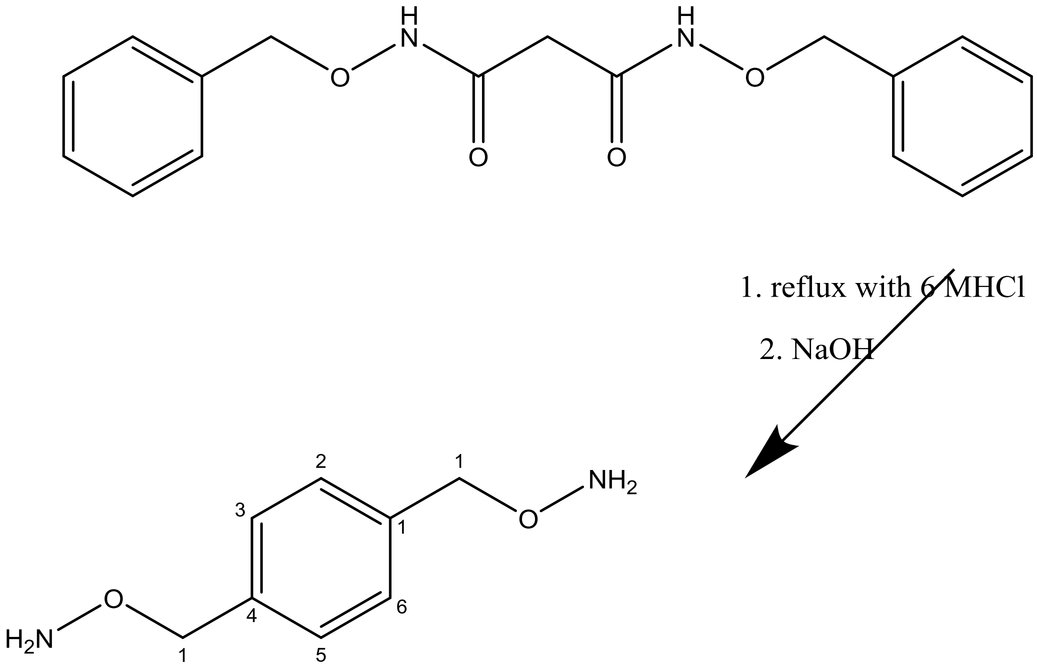
Conclusion and references

4. Conclusion

Hydroxamic acids have a wide range of applications in chemistry, biology, and medicine because of their major roles as chelating agents, enzyme inhibitors, anticancer agents, antibacterial agents, and many more. The reaction of diethyl malonate with hydroxylamine hydrochloride, using potassium hydroxide as a base in methanol, produced N^1, N^3-dihydroxymalonamide monomer. The polymer was produced by condensation polymerization of N^1, N^3-dihydroxymalonamide, and α, α′-Dibromo-p-xylene in DMF at 60 °C with stirring overnight. The average molecular weight was determined by static light scattering to be 728,000 g/mol. The glass transition temperature (Tg) was 73 °C, and the melting point (Tm) was 148 °C. The synthesized hydroxamic acid was characterized using FT-IR and NMR. Furthermore, to confirm the successful polymerization process, it was refluxed with 6 molars of HCl for 6 hours and the sample was analyzed by FT-IR, where the N-H stretching band appeared to be around 3347cm^{-1} due to the presence of an amine group by hydrolysis of dimer. Studying the polymer's ability to chelating chromium ions Cr(VI) The polymer is not optimal for chelating chromium ions Cr(VI) .

Future work

In future, more detailed studies can be conducted for the purpose of removing other metal cations than Cr with our polymer.



O,*O*'-(1,4-phenylenebis(methylene))bis(hydroxylamine)

According to Khomutov et al . can be hydrolyzed in strong acidic medium. We can conclude that this polymer is a biodegradable. We can also prepare O,O′-(1,4-phenylenebis(methylene))bis(hydroxylamine) ligand , which is potentially an excellent ligand for metal cations and also can be used for ,synthesis of other new polymers, for example when condensed with dialdehydes to form poly oxime polymers. This can be a future work concerning our polymer in this work.[26]

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تحضير ومعرفة خصائص بوليمر جديد قائم على حمض الهايدروكساميك .

إعداد: الاء إسماعيل خليل اسعد
المشرف الرئيسي: الدكتور محمود الخطيب
المشرف الثانوي: الدكتور مهند قريع

الملخص

أصبحت البوليمرات الغير قابلة للذوبان في الماء القائمة على (أحماض الهيدروكساميك) مهمة في تطبيقاتها لقدرتها الرائعة على ازالة مع المعادن الثقيلة. تم تحضير مونومر من تفاعل مالونات ثنائي الايثيل مع هيدروكلوريد هيدروكسيل امين باستخدام هيدروكسيد البوتاسيوم كقاعدة في الميثانول . تم تحضير بوليمر في ثنائي ميثل فورماميد بواسطة عملية تكثيف البلمرة وتم تحديد متوسط الوزن الجزيئي للبوليمر عن طريق تشتت الضوء وتم العثور على وزن جزيئي مرتفع 728.000 جم / مول .وكانت درجة حرارة الانصهار لهذا البوليمر 148 درجة مئوية . كان البوليمر قابل للذوبان بشكل جزئي في ثنائي كلورو الميثان وغير قابل للذوبان في الماء وفي ثنائي ميثيل فورماميد بالاضافة الى دراسة قدرة البوليمرعلى ازالة الكروم السداسي من المحلول المائي وكان 1.00 غرام من البوليمر قادر على ازالة 21% من الكروم السداسي .