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Removal of Cr (VI) Using Cross linked Poly (2-allyl-N^1, N^3-dihydroxymalonamide)

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Thesis approval

Removal of Cr (VI) Using Cross linked Poly (2-allyl-N^1, N^3-dihydroxymalonamide)

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Declaration

I certify that this thesis is submitted for the degree of master of applied and industrial technology is my own research, except where otherwise acknowledged, and that this thesis has not been submitted for a higher degree to any other university or institution.

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Data: 24 - 8 - 2024

Abstract

Polymers have many applications in humans' life, as a result more than half of the prepared chemicals in the industrial field are polymers. One of these important applications is wastewater purification. There is no doubt that Cr (VI) is one of the main pollutants because it may cause many harmful effects on human beings and the environment. The aim of this study is investigating the removal efficiency of Cr (VI) from polluted water using cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide). Batch experiments were conducted to investigate the removal efficiency of Cr (VI) from polluted water using the cross liked polymer with different parameters: pH, initial concentration, dosage weight, kinetic in addition to adsorption isotherms. It was found that the best removal efficiency of Cr (VI) when pH = 2. As the initial concentration of Cr (VI) increases the percentage removal decreases, increasing the polymer dosage the removal efficiency of Cr (VI) increasing, removal of Cr (VI) increases with increasing contact time and the process is first order as obtained from kinetic calculations. The variation concentration of Cr (VI) behavior was studied using Langmuir, Freundlich and Temkin adsorption isotherm models. R^2 of the curves of Cr (VI) removal by cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) shows that the removal process fits Langmuir, Freundlich and Temkin isotherms very well. However, the Freundlich isotherms has the largest correlation coefficient value (R^2). It was found that the maximum adsorption capacity (Q_{max}) is equal to 40.75 mg / g.

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Chapter One
Introduction

1. Chapter One

Introduction

1.1 Environmental problem

Environmental pollution is the addition of foreign and harmful substances and elements to the environment. Pollution types classified by environment in three classes: air pollution, land pollution and water pollution. All types have bad effects of wildlife, human beings and the environment.

Water pollution is the contamination of water resources causing negative impacts and leading to many problems, such as; aquatic ecosystem degradation, spreading of diseases and the reduction of ecosystem services.

1.2 Pollutants

Water polluted by a lot of contaminants including: microorganisms, fertilizers, organic wastes, toxic chemicals and others.

Water pollutants divided into six classes: pathogens, organic compounds, Inorganic contaminants, contaminants with an origin in sewage, pharmaceutical pollutants and solid wastes and plastics.

Each class of water pollutants can cause a lot of harmful effects on humanity and the environment, for instance; oxygen consumption, excessive algae growth, septic conditions, death of fish, the groundwater pollution, waterborne diseases and toxicity to humans, animals and plants.

Among of all classes, heavy metals which is type of inorganic contaminants have dangerous effects because it is toxic in very small amounts.

1.3 Occurrence and fate of heavy metals in environment

Heavy metals have bad effects on the environment causing pollution. The source of heavy metals pollutants divided into natural and anthropogenic sources. An example for natural sources is volcanic eruptions. However for anthropogenic sources, there are many examples as power plants, biomedical wastes, mining, electroplating, electronic wastes, agriculture activates, and industrial effluents from industrial activates like textile, dyes and pigment production, film and photography, leather tanning and metal finishing industries [1]. These activities produce large amounts of wastewater, and discharging it into waterbodies causes harm effects on living organisms and other species, especially heavy metals accumulation in human body by direct ways or via biological chain because they are not metabolized [2]. The main heavy metals causes' water pollution is due to Copper, Zinc, Cadmium, Lead, Mercury, Arsenic, and Chromium. Table (1.1) below describes some heavy metal pollutants and the maximum acceptable concentration in drinking water.

Table (1.1): Describes some heavy metal pollutants and the maximum acceptable concentration in drinking water [3, 4].

Element name	Maximum acceptable concentration
Copper	1.3 mg/L
Zinc	3 mg/L
Cadmium	0.005 mg/L
Lead	0 mg/L
Mercury	0.002 mg/L
Arsenic	0 g/L
Chromium	0.1 mg / L

Chromium is one of the main groundwater pollutants which is exists in Cr (VI) and Cr (III) states. Cr (III) is an essential element in humans and is much less toxic than Cr (VI), it is required in trace amounts for sugar and lipid metabolism [5 - 7]. Cr (VI) may cause bronchitis, ulceration, stomach cancer, liver and kidney damages, irritation of the skin, asthma, nasal perforation, produce effects on the gastrointestinal and immune systems and have mutagenic effects [8 - 12].

1.4 Removal methods for heavy metals

It is necessary to remove these toxic heavy metal from our environment. Various Cr (VI) and toxic metals removal methods have been reported, such as reverse osmosis, ion exchange, electro dialysis, electrochemical treatments, biological removal, membrane filtration, and adsorption [13].

Each removal method have advantages and disadvantages. For membrane filtration; when using this method 60–100% removal is possible and wide range pollutants removed. While ion exchange method advantages are: easy, effective and varieties of resins are available. But for electrochemical treatment method, advantages are: low pretreatment, economical and minimum reaction time. However, biological removal method advantages are: eco-friendly and have simple operation.

Most of these methods have a lot of disadvantages including expensive costs, low efficiency, generate sludge or secondary pollutants [13 - 16]. So that more research in the future is urgently required. However, adsorption is the most promising method of Cr (VI) removal because it is simple, fast, economical, not harming the environment, and easily applied in industrial activates [17].

1.5 Adsorption method

The adsorption depends on a using a surface of a material to make connection between the target particles and the surface by a specific forces.

Adsorption method used in removing contaminants from water. This is done through the use of specific adsorbent materials. The adsorption method is effective for eliminating various pollutants from groundwater and industrial wastewater.

Pollutants cling to the surface of materials known as adsorbents. The adsorption method classified in many classes according to the adsorbents types such as: natural materials, bio sorbents, miscellaneous sorbents, agricultural wastes, industrial wastes, nanomaterials, synthetic polymers and hydrogels. These adsorbents have a high surface area and microporous structures that allow for increased adsorption capacity.

Water purification by adsorption method offers several advantages, for example; simple and low cost method and the equipment for adsorption based purification systems relatively simple and inexpensive. Both anionic and cationic species can absorbed, removal of heavy metals is comparatively higher than other processes and minimum sludge production.

Adsorption method is energy efficient method; the adsorption process typically requires less energy than methods like reverse osmosis. It works at ambient temperature and pressure, making it energy efficient and suitable for areas with limited access to electricity.

Adsorption method is non - toxic and environmentally friendly method so that the process does not produce harmful by - products, making it a sustainable and safe method for water treatment.

Adsorption is a selective method; adsorbents can choose to selectively target specific contaminants, offering high customization for the purification process. This may be useful when certain pollutants need to be removed more effectively than others. Also Adsorption systems are scalable to meet the needs of both small and large scale water treatment, making them a versatile solution. The process is applicable in a variety of contexts, from household filters to industrial scale systems.

Adsorption systems generally require minimal maintenance, especially if the adsorbent material has a long life or can regenerated making it convenient for long term use. And many adsorbents can regenerated by heating or washing, which allows for reuse and can lower operating costs in the long run.

While the adsorption method offers several advantages for water purification, it also has its disadvantages, for instance; adsorbents limited capacity and have a finite capacity to adsorb contaminants. Once the adsorbent becomes saturated with pollutants, its effectiveness diminishes. This requires periodic replacement or regeneration of the adsorbent material.

While many adsorbents could regenerated, the regeneration process (e.g., heating or washing) could be energy intensive and may not always restore the adsorbent to its original capacity. This can lead to higher operational costs. In some cases, regeneration methods might not fully remove the adsorbed contaminants, potentially leading to the release of these substances back into the environment. Also the adjustment of pH considered o be one of the main challenges for some adsorbents.

Adsorption is more effective for organic compounds, chlorine, and heavy metals, but it may not be efficient for certain inorganic contaminants, such as some salts, or microbial pathogens.

Adsorption method is low efficiency for small molecules; so that very small or highly soluble molecules might not adsorb well onto the surface of the adsorbent material, limiting the scope of contaminants that can be removed.

Adsorption may be slower compared to other water purification methods, particularly in systems dealing with high concentrations of pollutants. The time required for effective purification may be a limiting factor in urgent or high demand situations.

In adsorption method the release of adsorbed contaminants may takes place; if the adsorbent is not properly regenerated or replaced, the pollutants may leach back into the water. In some cases, contaminants may be released back into the water during the filtration process, especially if the adsorbent material is not managed properly [13, 18 and 19].

1.6 Polymers as an adsorbent

Adsorption using synthetic polymers is a method of water purification that involves using specially designed synthetic polymer materials to remove contaminants from water. These polymers have the ability to be engineered to selectively adsorb specific contaminants, such as heavy metals making them versatile for various water treatment applications.

Heavy metal adsorption using synthetic polymers is an advanced method of removing toxic metals from contaminated water. Heavy metals like lead (Pb), mercury (Hg), cadmium (Cd), arsenic (As), and chromium (Cr) pose serious environmental and health risks. Synthetic polymers are increasingly being used to address this challenge because of their ability to selectively bind with metal ions and effectively remove them from water.

Synthetic polymers functionalized with thiol, amine, or carboxyl groups are highly effective for adsorbing toxic heavy metals like lead, mercury, cadmium, and arsenic.

Heavy metal adsorption by synthetic polymers takes place via various mechanisms such as; ion exchange, complexation (chelation), physical adsorption (Van der Waals forces) and electrostatic attraction.

In 2020, Tran et al used the sulfonated polystyrene in heavy metal removal. The research showed that sulfonated polystyrene have the ability to adsorb cadmium, copper and zinc with the maximum adsorption capacities $Q_{max} =$ 4.04 mg / g, 4.58 mg / g and 4.09 mg / g, respectively [20].

In 2020, Dong et al reported that polystyrene microplastic particles have the ability to adsorb Arsenite molecules by non - covalent interactions and electrostatic forces. The research found that the maximum adsorption capacity (Q_{max}) = 1.12 mg / g [21].

In 2019, Godoy et al reported that microplastics can adsorb heavy metals. The research showed that polyethylene have the ability to adsorb copper, lead and zinc from solutions with maximum adsorption capacity (Q_{max}) = 0.259 mg / g, 2.36 mg / g and 0.505 mg /g, respectively. Also polyvinyl chlorides removal of heavy metals was reported, the polymer have the ability of removal of lead and zinc with maximum adsorption capacity (Q_{max}) = 1.90 mg / g and 0.634 mg /g, respectively [22].

In 2019, Fu et al reported that natural - aged microplastics capable to adsorb heavy metals. The research showed that natural - aged microplastics can adsorb lead from solutions with maximum adsorption capacity (Q_{max}) = 13.60 mg / g, the adsorption process may be affected by many factors, such as the type and concentration of metal ions, type of microplastics and particle size [23].

Our research group in Al-Quds University prepared and characterized the novel polymer; cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) by Diana Khderat, and here the cross linked polymer's an environmental application will be investigated [24].

1.7 The aim and objectives

The aim of this thesis is investigating the removal efficiency of Cr (VI) from polluted water using cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide).

To achieve the aim of this study, the following objectives should be accomplished:

1- Preparation of 2-allyl-N^1, N^3-dihydroxymalonamide monomer.
2- Preparation of the cross linked polymer.
3- Investigating the removal of Cr (VI) using the cross linked polymer with the following parameters: pH, initial concentration and polymer dosage effects.
4- Study the kinetics of Cr (VI) ions removal.
5- Study adsorption isotherm models.

Chapter Two
Literature Review

2. Chapter Two

Literature Review

2.1 Polymers: an overview

2.1.1 Polymerization

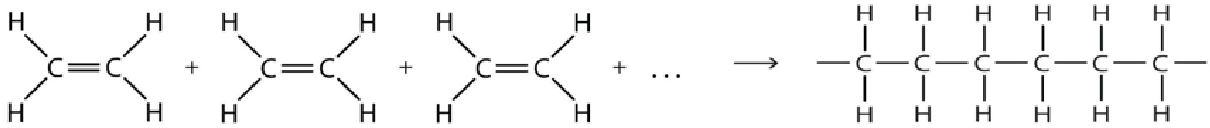
Polymerization is one of the very important chemical reactions used in the synthesis and production of a wide number of organic compounds called “Polymers”. The word “Polymer” contains two fragments; the first is “Poly”, which means many, and the second is “mer” which means part. Polymerization is a chemical reaction between large numbers of small parts called monomers which are united with each other in a repeated form to make the polymer. Polymers are divided into three classes based on their chemical structure: linear, branched chain, and cross linked polymers. Each one of the previous classes needs a different method to be synthesized. Cross linking polymerization depends on creating new covalent bonds between different linear polymers. As a result network-connected polymers will be produced. The polymerization type is chosen according to the properties that are want to be produced, which depends on the effect of branching, molecular weight and composition on the produced polymer properties, so that the linear polymers properties different than branched and cross linked polymers, also cross linked different than branched and have unique properties [25].

2.1.2 Mechanisms of polymerization

The polymerization takes place via one of the two general mechanisms which are: addition “chain reaction” polymerization mechanism and the condensation “step reaction” polymerization mechanism [26 - 28].

Addition polymerization depends on connecting the monomers with each other, and the resultant polymer will contain every atoms that exist in the monomers.

Equation (2.1) below describes that all ethene monomer atoms connected with each other and exist in the resultant product poly (ethene) without any atoms loss.

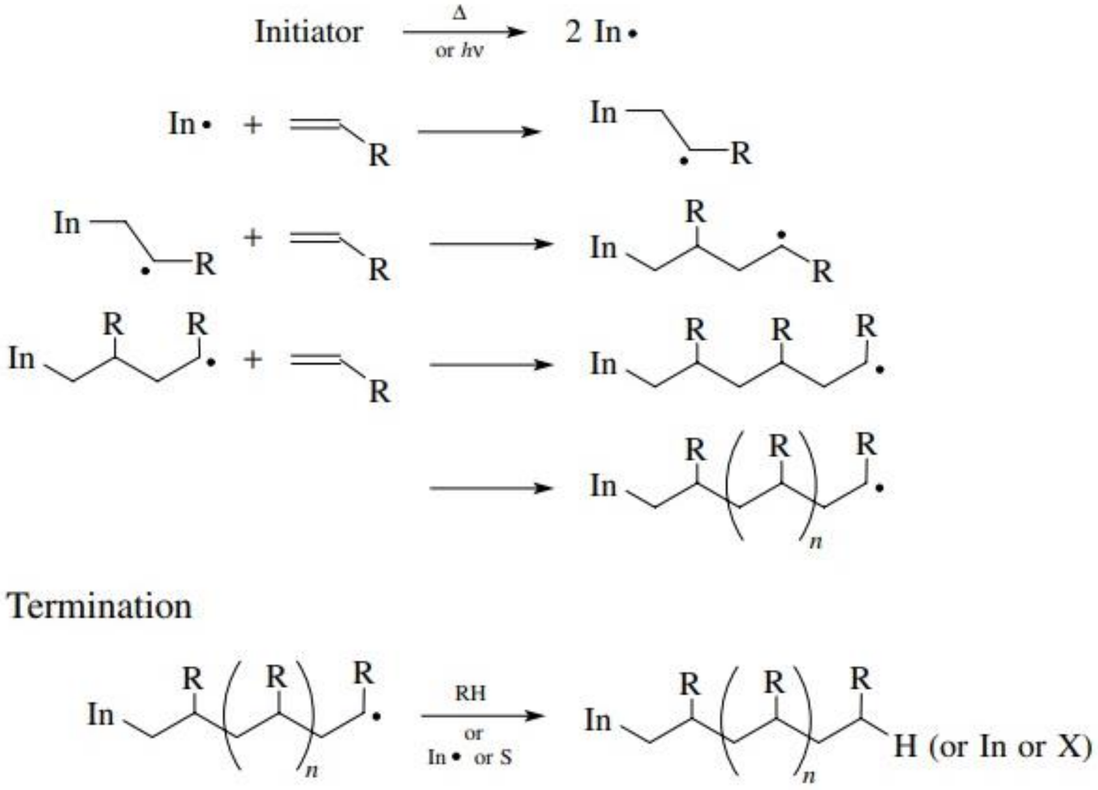


Equation (2.1): Describes the polymerization of ethene to produce polyethene [28].

It is also called “chain reaction” polymerization because active matter is necessary for starting the reaction, such as radicals and ions. The polymerization process starts with “chain initiation” by the reaction with the active materials, then “chain propagation” takes place by the reaction of the product in the chain initiation with other monomers, resulting in the length of the polymer becoming larger and larger until the “chain termination” in which the active matters react with each other and the reaction will stopped.

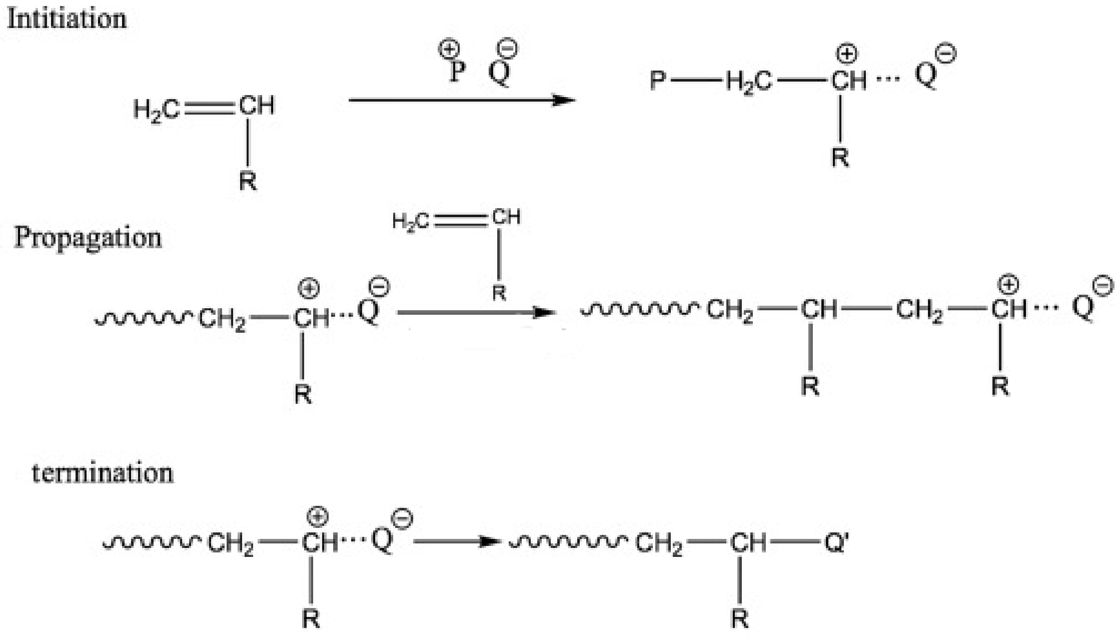
According to the active matters that initiate the polymerization reaction, chain reaction polymerization is classified into three classes; free radical polymerization, cationic polymerization, and anionic polymerization.

Free radical polymerization stars with the addition of radical matters, which are very reactive species with unpaired electrons. Equation (2.2) shows an example of a free radical polymerization mechanism [29].



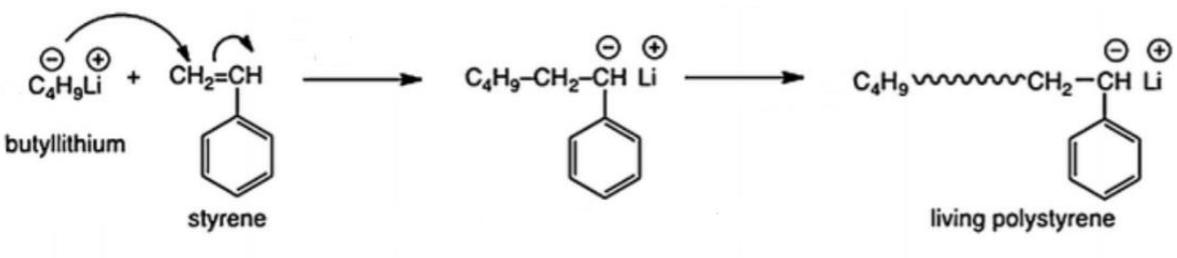
Equation (2.2): Describes the mechanism of free radical polymerization [29].

Cationic polymerization starts with the addition of a cation to react with the monomer so that a carbocation is produced and the polymerization takes place. Equation (2.3) shows an example of a cationic polymerization mechanism [30].



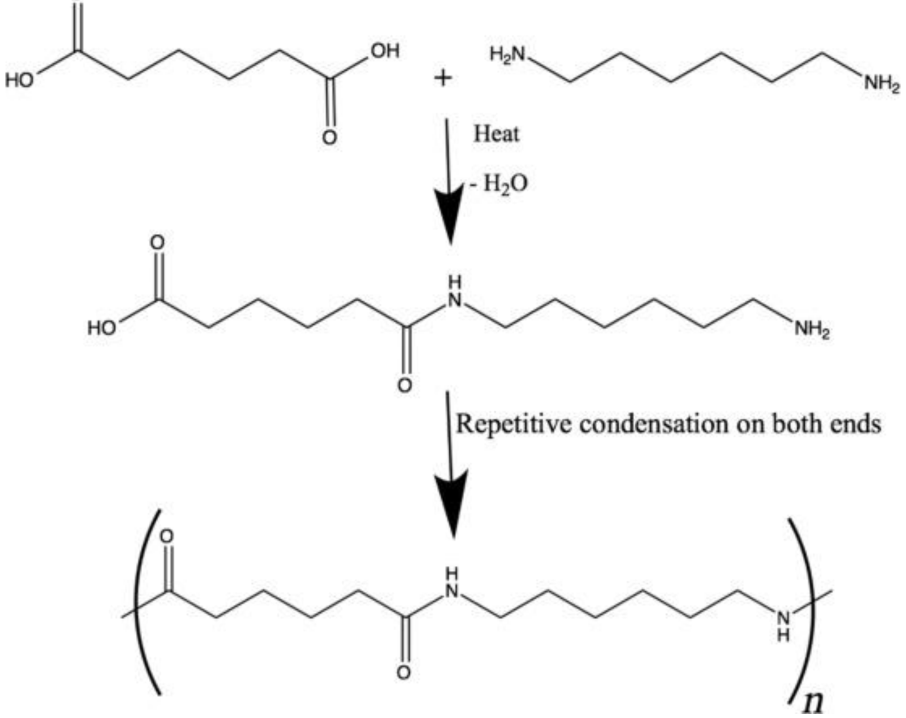
Equation (2.3): Describes the mechanism of cationic polymerization [30].

Anionic polymerization starts with the removal of hydrogen atoms from the monomer using a strong base or other compounds so that the anion will be produced then the chain initiation will begin and the polymerization takes place. Equation (2.4) shows an example of an anionic polymerization mechanism [31].



Equation (2.4): Describes the mechanism of anionic polymerization [31].

Condensation or "step reaction" polymerization mechanism often generates byproducts such as methanol or water, and there are no active matters that initiate the reaction, but the functional groups in both ends of the monomers react with each other under specific conditions to produce the polymer. Equation (2.5) shows the mechanism of adipic acid reaction with hexamethylene diamine to produce Nylon 6.6 polyamide which is an example of a condensation polymerization mechanism [28].

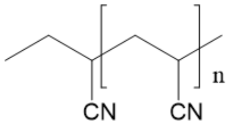
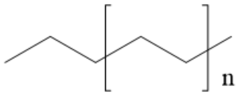
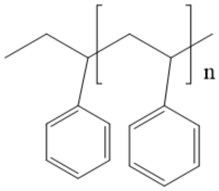
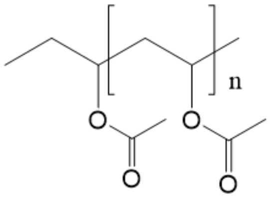
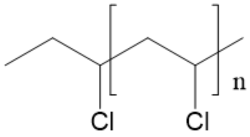
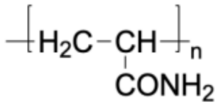
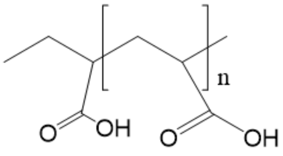
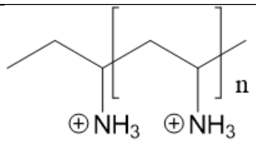


Equation (2.5): Describes the mechanism of adipic acid reaction with hexamethylene diamine to produce Nylon 6.6 polyamide [28].

2.1.3 Polymers solubility

Polymers classified according to the solubility in three classes, organic soluble polymers, water soluble and swellable polymers in either organic solvents or water [32]. Table (2.1) describes some of organic soluble polymers and water soluble polymers [33, 34].

Table (2.1): Some of polymers and there solubility [33, 34].

Polymer	Structure	Solubility (soluble in)
Poly acrylonitrile		DMF.
Polyethylene		Hexane.
Polystyrene		Aromatic solvents, esters and ketones.
Polyvinyl acetate		Organic solvents.
Polyvinyl chloride		Esters and ketones.
Polyacrylamide	 $\left[H_2C-\underset{CONH_2}{CH} \right]_n$	Nonionic synthetic polymer soluble in water.
Poly acrylic acid		Anionic synthetic polymer soluble in water.
Polyvinyl amine		Cationic synthetic polymer soluble in water.

2.1.4 Classifications of water soluble polymers

Water-soluble polymers are classified into three main classes: natural, synthetic, and semi-synthetic water-soluble polymers.

Natural water-soluble polymers, which are polymers that exist in nature, include proteins in which amino acids are connected as monomers by peptide bonds, for example, albumin, hemoglobin, and polysaccharides in which monosaccharides are attached by ether linkage, as in pullulan, Arabic gum, xanthan, and starch.

Semi-synthetic water-soluble polymers are extracted from natural sources and then chemically modified to improve their properties, such as derivatives of starch and cellulose ethers.

Synthetic water-soluble polymers are generally synthesized from petrol and its derivatives by polymerization reactions, for example, polyacrylamide, poly (acrylic acid), poly (phosphoric acid), and poly (vinyl alcohol) [24, 35].

2.1.5 Water swellable polymers and their applications

Cross linked polymers are swellable but not soluble in organic solvents or in water so that they can form “hydrogels”. Hydrogels synthesized from polymers containing the following functional groups: $–CO\ NH_2–$, $–SO_3\ H–$, $–CO\ NH–$, and $–\ OH$ [34], which gives them unique properties such as the capability to convert the rheology of aqueous mediums and the adsorption of materials from the solutions, so that they have a lot of chemical, pharmaceutical, environmental, medical, and industrial applications [26, 32, 36 - 39].

In 2023, Aljar et al reported that poly (vinyl alcohol) – chitosan / cellulose hydrogel beads have the ability to remove Pb (II), Cd (II), Zn (II), and Co (II) from aqueous solutions. The researcher found that the removal efficiency of using the adsorbent for Pb (II) was 99 %, for Cd (II) was 95 %, for Zn (II) was 92 %, and for Co (II) was 84 % [40].

Also in 2023, Mohamad Sarbani et al reported that cross linked magnetite - carboxymethyl cellulose - chitosan hydrogel beads have the ability to remove heavy metals from wastewater. The research found that the prepared hydrogel can adsorb Cr (VI) and Pb (II) with maximum adsorption capacity (Q_{max}) equal to 3.93 mg / g for Cr (VI) and 21.69 mg / g for Pb (II) as the paper described in Langmuir adsorption isotherm parameters [41].

In 2021, N. Mamidi et al. synthesized a hydrogel nanocomposite from poly (N - (4 - aminophenyl)methacrylamide)) - carbon nano-onions/ diclofenac - complex integrated chitosan which used in preparation of control release of drug systems by both pH and temperature [42].

Hydrogels have very wide applications in food chemistry, for example; food delivery systems.

In 2020, Lim et al. reported that chitosan and Ca-alginate was used in preparation of hydrogel pickering emulation which capable to deliver food because Ca-alginate not release the pickering emulation in stomach [43].

In 2020, Goncalves et al. reported that hydrogel scaffold prepared from chitosan then modified using carbon nanoscale tubes was used in dyes removal from the food [44].

Also in 2020, X. Hu et al. reported that a hydrogel poly electrolyte complex synthesized from *N*, *N*, *N* - trimethyl chitosan and salecan polysaccharides, then used as sustained release delivery system to transferee polyphenols of green tea [45].

Another food chemistry work in 2020, Huang et al. reported that the presence of carcinogenic matter "Rhodamine B" in food was detected by solid phase hydrogel extraction, providing a fast method for food safety monitoring [46].

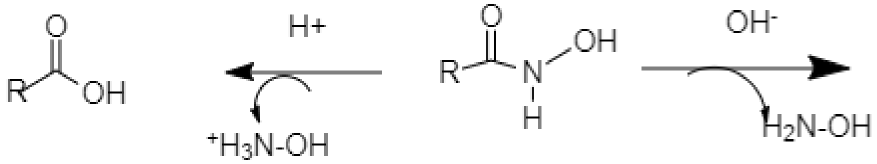
Hydrogels used in sensors as one of its application, for example temperature and humidity sensors. In 2020, Li et al. reported that poly vinyl alcohol hydrogel was modified with silver nanofibers and used as temperature sensors [47].

Also in 2020, Jang et al. reported that chitosan used in preparation of hydrogel metal filters that used as sensors of humidity connected with photoelectrical cells [48].

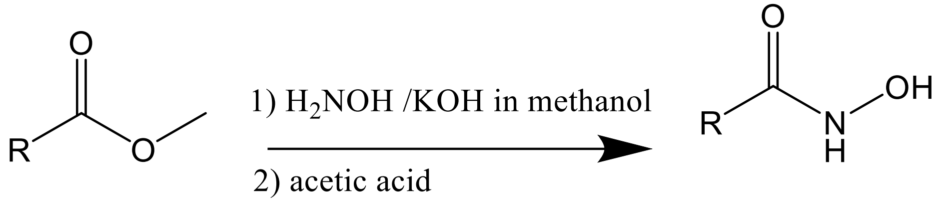
2.1.6 Hydroxamic acid derivative polymers

Hydroxamic acids are an unstable, explosive, and mutagenic crystalline solids that discovered in 1869 by Heinrich Lossen, usually available as hydrochloride salt forms, and the free hydroxylamine is prepared in situ from that salts under basic medium [49]. Hydroxamic acids have same behavior as carboxylic acids, or amides under both base and acid conditions as shown in equation (2.6) below [50]. They are a product of the reaction of hydroxylamine (or its derivatives) with ester, amide and nitriles. Equation (2.7) below shows that hydroxamic acids prepared by one step reaction, using ester derivative as starting material in basic aqueous medium. Polymers carrying hydroxamic acids first reported in 1942, then, introducing hydroxamic acids into polymers methods was developed [49, 51 - 54].

Hydroxamic acid based polymers prepared either by converting the ester monomer into hydroxamic acid, then polymerize it, the disadvantage of this method is partially hydrolysis of the hdroxamic acid group, but it is simple and easier method. Or by polymerization of the ester monomer then derivitize it into hydroxamic acid [24, 54].



Equation (2.6): Describes hydroxamic acids behavior in acidic or basic solutions (not stable) [50].



Equation (2.7): Reaction describes how hydroxamic acids generally synthesized [54].

Hydroxamic acid derivative of polymers have various pharmaceutical, biological, industrial and environmental applications. In 2023, Duan et al. synthesized poly (2 – acrylamide – pentane di hydroxamic acid) by combination of 2 – acrylamide – pentane di hydroxamic acid with the carbonyl functional group of D113 resin. The result product was used in the removal of Cu (II) and Ni (II) from wastewater [55].

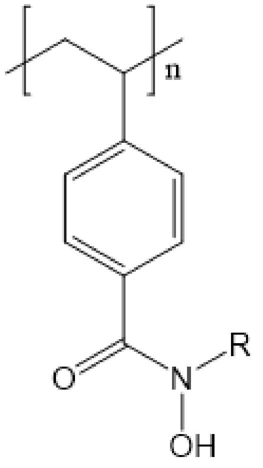
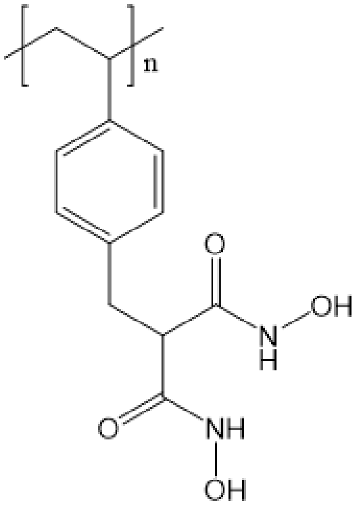
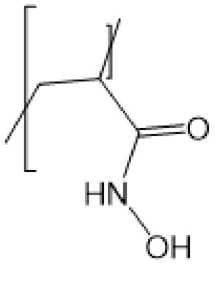
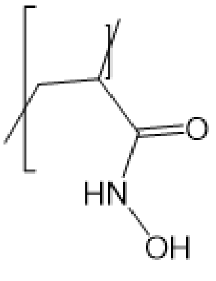
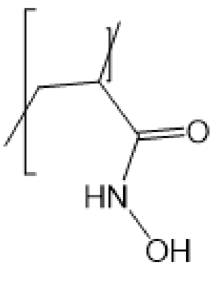
Also in 2023, Charvátová et al. synthesized N - [2-(Hydroxyamino)-2-oxo-ethyl]-2-methyl-1, 2-propandiyl-2-amide from the reaction between copolymer P (HPMA-co-MMA) and hydroxylamine solution. The result product was used in drug coating [56].

Another work in 2023, Li et al. synthesized matrix enhanced polymer inclusion membrane using octylhydroxamic acid and di (2-ethylhexyl) phosphoric acid. The result product have the ability to be used in water treatment [57].

In 2021, Fawal et al. reported that poly (hydroxamic acid) was prepared from poly (methyl methacrylate), which put on cellophane membranes, then hydroxylamine was attached with poly (methyl methacrylate) at 40°C [58].

Also in 2021, Cao et al. synthesized poly (6-acryloylamino-hexyl hydroxamic acid) by substitution reaction between polyacrylicresin followed by the substitution reaction with 6-aminohexyl hydroxamic acid. The product is suitable to be used in chemical adsorption of rare earth ions. Hydroxamic acid derivative of polymers have wide field of applications as described above, some other examples shown below in table (2.2) [59 - 61].

Table (2.2): Some of industrial and environmental applications of hydroxamic acid derivative of polymers [60, 61].

Polymer structure	Application
 R: H, C_6H_2	Used in toxic earth metals removal by resin of poly (hydroxamic acid).
	Uranium removal from water sources
  	Used in packaging products like drugs that contains lipids, because its ability of reducing lipid oxidation.

2.2 Heavy metals and chromium as pollutants

Heavy metals are naturally occurring elements with a high atomic weight and density, which can become significant pollutants in the environment when released in excess. They pose serious health risks, particularly in emergency situations where there is sudden and uncontrolled exposure to these substances, such as through industrial accidents, spills, or disasters. Heavy metals toxicity can cause long-term environmental degradation and severe health problems for both humans and wildlife.

Heavy metals are persistent in the environment and do not biodegrade. Once they enter soil, water, or air, they can remain toxic for a long time. Many of these metals bio - accumulate in organisms, meaning they build up in tissues over time and increase in concentration as they move up the food chain.

Many heavy metals are highly toxic even in trace amounts, and exposure can lead to acute or chronic health issues such as neurological disorders, organ damage, cancer, and developmental issues. Also it can lead to large scale health crises.

Unlike organic pollutants, heavy metals do not break down into less harmful substances. For example, lead and mercury, once introduced into the environment, can remain there for centuries. This persistence makes cleanup and recovery efforts more challenging and costly [1, 2].

Chromium (Cr) is a heavy metal that can pose significant environmental and public health risks. It is widely used in industrial processes such as electroplating, tanning, pigment production, and alloy manufacturing. However, its release into the environment can lead to hazardous contamination of air, water, and soil. In these cases, chromium can exist in Cr (III) and Cr (VI) oxidation states.

Cr (III) is the trivalent form, which is relatively less toxic and is considered an essential trace element for human health, playing a role in glucose metabolism. However, it can still be harmful at high concentrations.

Cr (VI) is the hexavalent form, it is highly toxic, carcinogenic, and more mobile in the environment. It can easily migrate through water and soil, contaminating broader areas. It is considered one of the most hazardous substances.

Chromium is released into the environment through various causes such as: industrial accidents, waste disposal sites, transportation accidents and mining operations.

Cr (VI) have many health risks like carcinogenic effects; it is classified as a Group 1 carcinogen. Chronic exposure can lead to lung cancer, while ingestion of contaminated water or food can increase the risk of cancers of the stomach, liver, and other organs.

Chromium is respiratory and skin irritation; short term exposure to chromium compounds can cause respiratory problems, such as coughing, wheezing, and shortness of breath. Prolonged exposure can also cause skin rashes, ulcers, and other dermatological issues.

Chromium causes organ damage; long term exposure to high concentrations of Cr (VI) can result in severe damage to internal organs, particularly the liver and kidneys. It can also lead to neurological effects such as memory loss and muscle weakness [5 - 12].

2.3 Removal of Cr (VI) methods

It is necessary to remove Cr (VI) which is very toxic mater as described in previous chapter from our environment. Various Cr (VI) and toxic metals removal methods have been reported, such as electrocoagulation, membrane filtration, reverse osmosis, biological removal, ion exchange, and adsorption [14].

Electrocoagulation is electrochemical treatments of wastewater in which Cr (VI) converted to Cr (III) via electro reduction process using specific metals as cathodes and anodes, so that oxidation and reduction reactions takes place and Cr (VI) removed. Electrocoagulation method have some advantages such as: effective for low to moderate concentrations of Cr (VI), no need for chemicals, simultaneous removal of multiple contaminants and simple design. Also it is have disadvantages such as: electrodes degrade over time and need replacement, which can add to operational costs, energy consumption, sludge production and effectiveness may be reduced in waters with high salinity or high organic content. Electrocoagulation can achieve removal efficiencies of 80 – 99 % for Cr (VI) in well - controlled systems. In 2022, Lu et al. demonstrated that 84 % to 96 % of Cr (VI) was removed using electrodes produced from aluminum metal at pH from 3 to 6 [62].

Membrane filtration also used in water purification, which is a method depends on a membrane that allow water to pass through it, but rejects other materials according to the size of the pores on the membrane. Membrane filtration method have some advantages such as: high removal efficiency, continuous operation and no chemical use. Also it is have disadvantages such as: high operational costs, membrane fouling and have low water recovery rates. Membrane filtration can achieve removal efficiencies over 95 %, and in some cases, it may approach 99 % for Cr (VI), especially with reverse osmosis. In 2022, 99.5 % of Cr (VI) was removed by Jo et al. using IP assembled poly (acrylol hydrazide) (PAH-TFC) as membrane filter for aqueous mediums that can reject Cr (VI), the result at pH 3 [63].

Biological removal is one of the methods that is used in water purification, and it is depending on the usage of microorganisms such as bacteria or fungi. Biological removal method have some advantages such as: environmentally friendly, low operating costs and selective reduction. Also it is have disadvantages such as: slow process, limited to specific conditions and microbial inoculum management costs. Biological removal can achieve removal efficiencies of 80 – 95 %.

In 2020, An et al. described that Cr (VI) was removed by using aerobic bacteria *Pseudomonas aeruginosa* G 12 by the reduction of Cr (VI) to Cr (III), and the percentage removal of Cr (VI) was found to be 93% at 10ppm initial concentration [64]. Also in 2020 Chatterjee et al. described that 99% of Cr (VI) was removed by adsorption and reduction to Cr (III) using *Aspergillus nigr* BSC-1 fungi when pH was 3, and temperature was about 40°C [65].

One of the methods used in Cr (VI) removal is ion exchange, which is depends on the replacement of harmful ions with other nontoxic ions. Ion exchange method have some advantages such as: high selectivity, fast process and re-generable. Also it is have disadvantages such as: its resins have a finite capacity, requires chemicals for regeneration adding to the operational costs and water may need to be pre-treated to remove suspended solids, which can clog the resins. Ion exchange method can achieve removal efficiencies more than 90 % of Cr (VI). In 2018, Zang et al. described that Cr (VI) was removed by the replacement with Cl ions in poly - epichlorohydrin - dimethylamine with styrene macro porous weak basic anion exchange resin D - 301, and it was found that at room temperature and pH 2 the maximum adsorption capacity was 194 mg / g [66].

2.4 Removal of Cr (VI) using adsorption method

Most of Cr (VI) removal methods have a lot of disadvantages including expensive costs, low efficiency, generate sludge or secondary pollutants and fouling in membrane technology [13 - 16]. However, adsorption is the most promising method of Cr (VI) removal because it is simple, fast, economical, not harming the environment, and it is easily applicable in industrial activates [17]. The adsorption depends on a using a surface of a material to make connection between the target particles and the surface by a specific forces.

In 2024, M. Wang et al. reported that Cr (VI) was removed from a solution using poly ethylenimine modified hydro char with nanoscale Fe particles attached on its surface, and Cr (VI) removal maximum adsorption capacity found to be 532.35mg / g [67].

In 2023, S. Jayakumar et al. reported that the use of a mixture that prepared from mixing three components was used in Cr (VI) removal, the three components is nylon6, chitosan, and also poly urethane, in a ratio 1:2:1, the mixture stirred in formic acid for 1 hour, then the result mixture product was dried. The product used in heavy metals removal from wastewater. It was found that the maximum adsorption capacity of Cr (VI) removal from a solution was 250mg / g where pH=5 [68].

In 2022, Neolaka et al. reported that zeolite that was obtained from natural resources and activated to remove all impurities and attached with Fe_3O_4 to become magnetic and easy removed, this zeolite was used as adsorbent for Cr (VI) removal and adsorption from aqueous mediums. The adsorbent was prepared by co precipitation technique. The maximum adsorption capacity of the Cr (VI) by the treated zeolite was found to be 2.850mg / g at pH 2, at room temperature, the time of contact between solution and the treated zeolite adsorbent was 50 min, and the mass of the adsorbent is 0.2g [69].

In 2021, Chakraborty et al. reported that sawdust was used as adsorbent for Cr (VI) removal from aqueous solutions and the removal percentage was enhanced by a modification process using sulfuric acid and formaldehyde then heated for 4 hours at 100 °C then washed and dried. It was found that the maximum adsorption capacity was 8.84 mg / g at pH 2.0, when the initial concentration used of chromium is 10 mg / L and modified sawdust dose is 4 g / L [70].

In 2020, Qiao et al. reported that *Enteromorpha prolifera* algae was used in preparation of bio chars that have beads like shape and attached with iron particles on its surface, which used in heavy metals removal by adsorption. Also the prepared adsorbents can float in aqueous solutions, so that it easy removed after the adsorption of the heavy metals. It was found that the maximum adsorption capacity of the adsorbent that produced using algae was 11.13 mg / g, when the pH was 4, and the time of contact between solution and the adsorbent was 900 min [71].

2.5 Polymers as Cr (VI) adsorbents

Cr (VI) adsorption using synthetic polymers is an important method for removing Cr (VI), which is a toxic and carcinogenic metal commonly found in industrial wastewater. Synthetic polymers due to their adsorption abilities have emerged as effective materials for chromium removal from contaminated water.

In 2022, Bobik et al. reported that poly (sodium acrylate) modified magnetite nanoparticles can used for the removal of Cr (VI) from aqueous solutions. The research found that the maximum adsorption capacity (Q_{max}) for the adsorption of Cr (VI) using the adsorbent is equal to 39.99 mg / g [72].

In 2021, Yu et al reported that poly (*m* - aminobenzene sulfonate) with cellulose nanofibrils can used in Cr (VI) adsorption. The research showed that prepared adsorbent have the ability to adsorb Cr (VI) from water solutions, and it fits Langmuir isotherm with maximum adsorption capacity (Q_{max}) = 5.263 mg / g. [73]

In 2019, Godoy et al reported that microplastics can adsorb Cr (VI). The research showed that polyethylene have the ability to adsorb Cr (VI) from solutions with maximum adsorption capacity (Q_{max}) = 4.7 mg / g. Also polyvinyl chlorides removal of heavy metals was reported, the polymer have the ability of removal Cr (VI) from solutions with maximum adsorption capacity (Q_{max}) = 2.44 mg / g [22].

In 2019, Debnath et al reported that micrometer sized polystyrene particles which modified with electromagnetic polyaniline have the ability to remove Cr (VI). The research showed that the prepared adsorbent have the ability to adsorb Cr (VI) from water solutions, also it was found that the process fits Freundlich isotherm model and the maximum adsorption capacity (Q_{max}) = 20.289 mg / g. [74]

In 2018, Zon et al reported that low density polyethylene microbeads have the ability to adsorb Cr (VI) from solutions with maximum adsorption capacity (Q_{max}) = 1.9171 mg / g after 180 hours. The research found that the process fits Langmuir adsorption isotherm with correlation coefficient (R^2) = 0.9707. [75]

In 2018, Yang et al reported that polypyrrole with corncob biochar can used for Cr (VI) adsorption. The research showed that the prepared adsorbent have the ability to adsorb Cr (VI) from water solutions, also it was found that the process fits Langmuir isotherm model and the maximum adsorption capacity (Q_{max}) = 19.23 mg / g. [76]

Chapter Three
Methodology

3. Chapter Three

Methodology

3.1 Chemicals and reagents

Acetone (C_3H_6O), diethyl allyl malonate monomer ($C_{10}H_{16}O_4$, 99%), 1,5-diphenylcarbazide ($C_{13}H_{14}N_4O$, 98%), Iron(III) chloride ($FeCl_3$), hydrochloric acid (HCl, 37%), hydroxylamine hydrochloride ($HONH_2.HCl$, 99%), potassium dichromate ($K_2Cr_2O_7$), potassium persulfate ($K_2S_2O_8$, 99%) , potassium hydroxide (KOH, 85%) , methanol (CH_3OH, 98%) , *N, N′*-Methylene bis (acrylamide) ($C_7H_{10}N_2O_2$, 99%) , sodium hydroxide (NaOH , 99%) , and sulfuric acid (H_2SO_4 , 95%) . All chemicals and reagents were purchased from Sigma Aldrich.

3.2 Instruments

Edge pH meter (Hanna instruments, Italy), UV- Visible Spectrophotometer “UV- 1601” (SHIMADZU, Japan), MK 200 D Shaker (Yamato, Japan), and Atomic absorption spectrometry (AAS) (AA - 6200 Shimadzu, Japan).

3.3 Methods

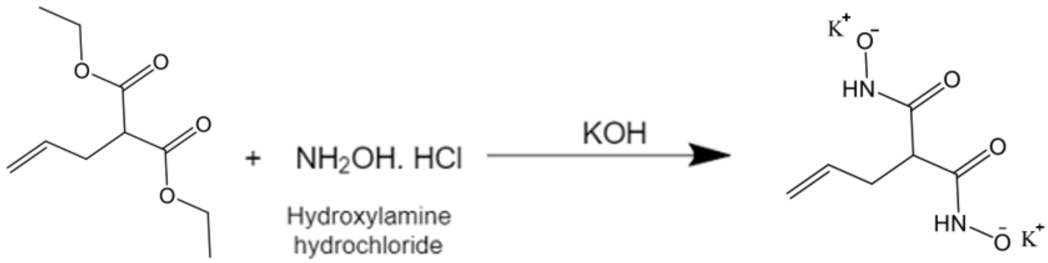
3.3.1 2-allyl-N^1, N^3-dihydroxymalonamide monomer synthesis

2-allyl-*N^1, N^3*-dihydroxymalonamide monomer was synthesized with the following procedure as described in equation (3.1) below [77]:

1) 7g of hydroxylamine hydrochloride was dissolved with heating in 50 ml methanol (first solution).
2) 8.5g potassium hydroxide dissolved with heating in 25ml methanol (second solution).
3) Both solutions were reached boiling points, then were cooled to 30-40 °C and potassium hydroxide solution (second solution) was transferred to the first solution with shaking to form hydroxylamine solution.
4) The mixture was cooled in ice bath to precipitate all KCl molecules, then KCl removed

by filtration.

5) After hydroxylamine solution filtration, 5g of diethyl allyl malonate was added with shaking, and the monomer collected after 48 hrs.
6) To confirm that hydroxamic acid was produced; a small amount of monomer was reacted with 1 ml $FeCl_3$ solution in a test tube, if there is hydroxamic acid; ferric hydroxamate as burgundy or magenta colored complex will be formed [78].

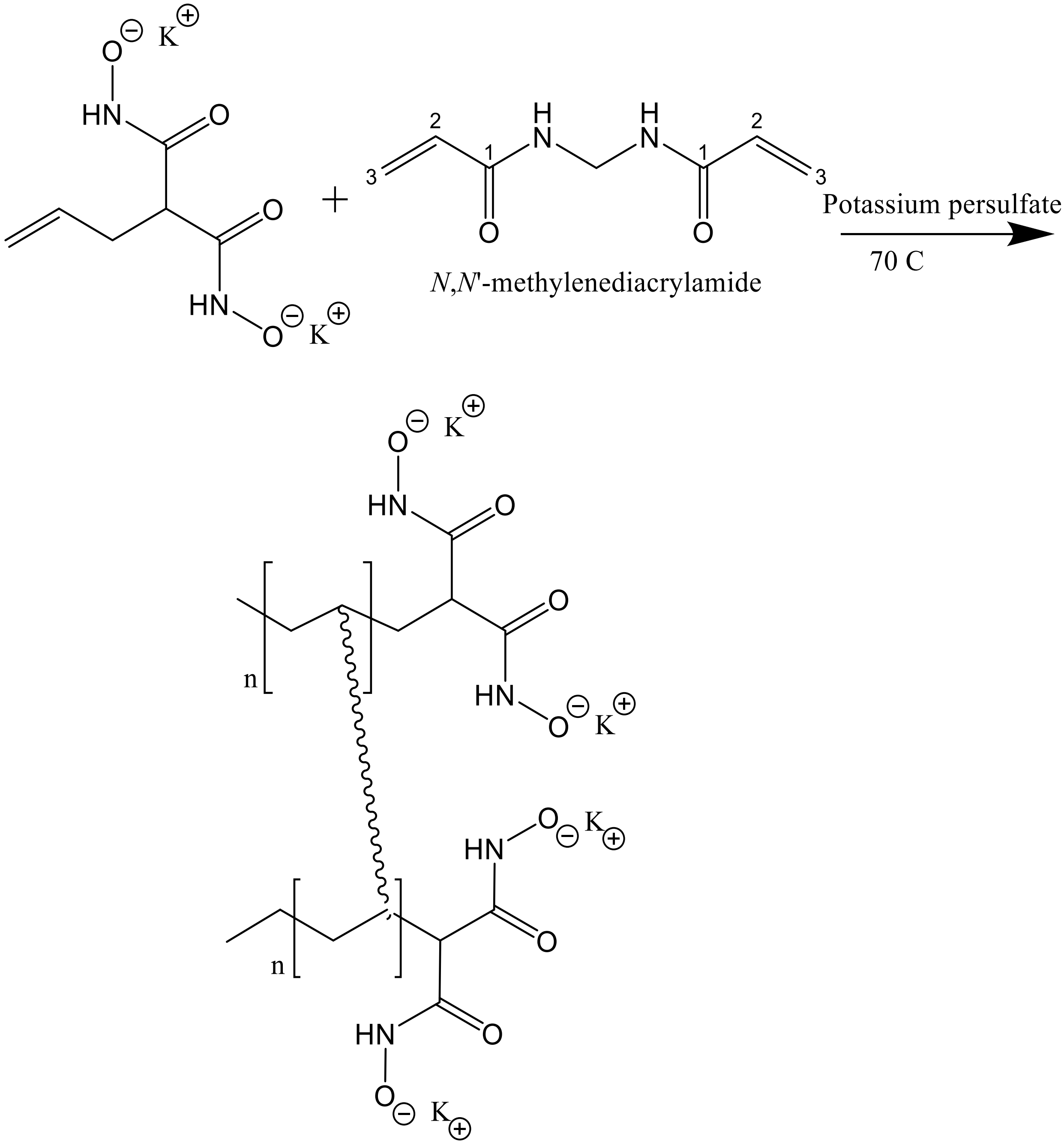


Equation (3.1): 2-allyl-*N*^{1}, *N*^{3}-dihydroxymalonamide monomer synthesis by reacting diethyl ally malonate with hydroxylamine hydrochloride in basic medium.

3.3.2 Preparation of cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide)

The following procedure were applied for preparation of cross linked poly (2-allyl-*N*^{1}, *N*^{3}-dihydroxymalonamide) by free radical polymerization as described in equation (3.2) below:

1) 18.8 g of 2-allyl-*N*^{1}, *N*^{3}-dihydroxymalonamide and 8.96 g *N,N'*-methylen bis(acrylamide) which is cross linking agent dissolved in 200 ml distilled water in round bottom flask, which stoppered with septum.
2) The reaction takes place in the presence of inert gas (nitrogen).
3) Round bottom flask with the compounds inside was heated at 70°C for 30 min, 0.108g potassium persulfate was added as initiator. After one day polymer was collected.



Equation (3.2): Cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) synthesis reaction [24].

3.3.3 Stock standards Cr (VI) preparation and dilutions for batch experiments

Cr (VI) stock solution (500 ppm) was prepared by dissolving 141.4 mg $K_2Cr_2O_7$ in distilled water and diluted to 100ml.

200, 100, 50, 25, 12.5, 6.25 ppm Cr (VI) solutions were prepared from Cr (VI) stock solution, and the calibration curve was obtained after absorbance of Cr (VI) solutions was measured and plotted with the solutions concentrations, the absorbance measurement method described in section (3.3.4).

The removal of Cr (VI) was confirmed by a simple test, 0.1g of the polymer mixed with 25ml of diluted Cr solutions and shacked for 2hrs. Samples before and after shaking were taken and filtered with PVDF-L syringe filter with 0.22 µm pore size.

3.3.3.1 Variation pH

The effect of pH variation was studied by repeating the test at different pH values (2, 4, 6, and 8), while other factors were kept constant. 0.1 g of the polymer was weighed in 4 Erlenmeyer flasks. 50 ml of 10 ppm Cr (VI) solution were added to 4 beakers and the pH was adjusted by using diluted HCl and NaOH, then transferred to the Erlenmeyer flasks and were shacked at 140 rpm for 2 hrs. at room temperature. Samples before and after shaking were taken and filtered with PVDF-L syringe filter with 0.22 µm pore size.

3.3.3.2 Variation of initial concentration of Cr (VI)

Initial concentration of Cr (VI) effect was studied by the preparation of different concentrations of Cr (VI) solutions; 100, 50, 20, and 10 ppm, 50 ml were taken from each one, and the pH was adjusted to 2, then the different solutions were added to four Erlenmeyer flasks containing 0.1 g of the polymer, these solutions were shacked at 140 rpm for 2 hrs. at room temperature. Samples before and after shaking were taken and filtered with PVDF - L syringe filter with 0.22 µm pore size.

3.3.3.3 Variation of polymer dose

Polymer dosage effect was studied by weighing 0.1, 0.2, 0.3, and 0.4 g of the polymer, 50ml of 10 ppm Cr (VI) solution was added to each one after adjustment of pH to 2, the four Erlenmeyer flasks were put on the shaker for 2 hrs. at room temperature and the speed was 140 rpm. Samples before and after shaking were taken and filtered with PVDF - L syringe filter with 0.22 µm pore size.

3.3.3.4 Kinetic study

For kinetic study; 50 ml of 10 ppm Cr (VI) solution was added to 0.1 g polymer after pH adjustment to 2, the mixture was put on shaker at 140 rpm for 5min, at room temperature. Samples before shaking, after 10, 30, 90, and 300 seconds were taken and filtered with PVDF - L syringe filter with 0.22 µm pore size.

3.3.4 Cr (VI) ions concentration determination and related calculations

The Cr (VI) and total Cr concentrations were determined using the following methods:

From each filtrated samples and standards, 1ml was taken and its pH adjusted to 1 using H_2SO_4, it was then diluted to 100 ml, and to this solutions 2 ml of 1, 5-diphenylcarbazide solution - which is prepared by dissolving 250 mg of 1,5-diphenylcarbazide in 50ml acetone - was added. After leaving the solution for 15min, UV - Visible Spectrophotometer was used to measure the absorbance at 540 nm in order to determine the concentration of Cr (VI). Total Cr measurements were obtained by Atomic absorption spectrometry.

The concentration of total Cr and Cr (VI) remain in the solution were calculated by:

1. The concentration of total Cr remain in the solution after the experiment was calculated using calibration curve and absorbance that obtained from Atomic absorption spectrometry.

2. The concentration of Cr (VI) remain in the solution after the experiment was calculated using calibration curve and absorbance that obtained from UV.

The concentration of Cr (III) remain in the solution after the experiment was calculated by subtracting Cr (VI) remain in the solution concentration from total Cr concentration as described below in equation (3.3).

$$[\text{Cr (III)}] = [\text{total Cr}] - [\text{Cr (VI)}]$$

Equation (3.3): Calculation of Cr (III) remain in the solution concentration.

The concentration of Cr (VI) that was adsorbed through the experiment was calculated by subtracting total Cr concentration from initial Cr concentration as described below in equation (3.4).

$$[\text{Cr (VI)}] \text{ adsorbed} = [\text{total Cr}] - [\text{Cr}_i] \text{ initial concentration}$$

Equation (3.4): Calculation of adsorbed Cr (VI) concentration.

Percentage of Cr (VI) remain in the solution, percentage of Cr (VI) reduction to Cr (III) and percentage of Cr ions removal, all were calculated by:

1. Cr (VI) remain in the solution percentage was calculated through division the concentration of Cr (VI) remain in the solution (calculated in point 2 above) by the initial concentration and multiply by 100 as described below in equation (3.5).

   $$\%Cr\ (VI)\ remain = [Cr\ (VI)]\ remain\ in\ the\ solution\ /\ [Cr_i] * 100$$

   Equation (3.5): Calculation of Cr (VI) remain in the solution percentage.

2. Cr (III) remain in the solution percentage (reduction percentage) was calculated through division the concentration of Cr (III) remain in the solution (calculated in equation (3.3) above) by the initial concentration and multiply by 100 as described below in equation (3.6).

   $$\%Cr\ (III)\ remain = [Cr\ (III)]\ remain\ in\ the\ solution\ /\ [Cr_i] * 100$$

   Equation (3.6): Calculation of Cr (III) remain in the solution percentage.

3. Cr (VI) that was adsorbed through the experiment percentage (adsorption percentage) was calculated through division the concentration of Cr (VI) that was adsorbed through the experiment (calculated in equation (3.4)) by the initial concentration and multiply by 100 as described below in equation (3.7).

   $$\%\ adsorption\ Cr\ (VI) = [Cr\ (VI)]\ adsorbed /\ [Cr_i] * 100$$

   Equation (3.7): Calculation of adsorbed Cr (VI) percentage.

4. Removal of Cr (VI) percentage was calculated by adding reduction percentage with adsorption percentage as described below in equation (3.8).

   $$\%\ removal\ Cr\ (VI) = \%\ reduction\ Cr\ (VI) + \%\ adsorption\ Cr\ (VI)$$

   Equation (3.8): Calculation of Cr (VI) removal percentage.

The addition of the results in point 1 (% Cr (VI) remain) with point 2 (reduction percentage) and point 3 (adsorption percentage) must equal 100%. Then removal of Cr (VI) percentage (point 4), reduction percentage (point 2) and adsorption percentage (point 3) plotted against pH, initial concentration, dose or time.

Also adsorption capacity (q_e) in mg / g was calculated through subtracting the concentration in mg / L at equilibrium (C_e) from initial concentration (C_i), then the result multiplied by the solution volume (V) in L then divided by the mass of the adsorbent (m) in g as described below in equation (3. 9).

$$q_e = \frac{(C_i - C_e) \times V}{m}$$

Equation (3.9): Adsorption capacity calculation [79, 80].

Then adsorption capacity of Cr (VI) using the cross linked polymer plotted against pH, initial concentration, dose or time [79, 80].

3.3.5 Study the kinetics of Cr (VI) ions removal

Initial rate law method was used in the determination of the order of the reaction, equations (3.10 and 3.11) was used in the calculations [81].

$$\frac{d[A]}{dt} = -k[A]$$

Equation (3.10): The rate (R) of the reaction law.

From the initial rate law method; if concentration plotted against the time of the reaction then the first derivative was taken; equation of the rate of the reaction was obtained. Then, by taking natural logarithm to both sides of equation (3.10) the equation (3.11) will be obtained. If natural logarithm (ln) of the rate plotted against the natural logarithm of the concentration; the order of the reaction was obtained from the slope.

$$\ln R = \ln k + x \ln [A]$$

Equation (3.11): Natural logarithm of rate of the reaction.

Kinetic calculations:

1. The concentration (ppm) of Cr (VI) remain in the solution at each period of time was calculated using calibration curve and absorbance that obtained from UV.
2. To convert the concentration to molarity [Cr], concentration in ppm was divided by the molar mass (52 g/mol), and the result obtained will be in millimole (mM).
3. The concentration (mM) plotted against time, and first derivative was taken.
4. First derivative equation was used in rate (R) determination in mM/s.
5. Natural logarithm (ln) of [Cr] and R was taken.
6. The ln R was plotted against ln [Cr], then the order was determined from the slope.

3.3.6 Study adsorption isotherm models

Cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) adsorption of Cr (VI) behavior was studied using Langmuir, Freundlich and Temkin isotherm models. The behavior studied using the variation concentration of Cr (VI) experiment's results.

Adsorption isotherm models gives a mathematical idea (by curve) about the relationship between the amounts of the matter that will be adsorbed on the surface of polymer and the amounts that will still in the solution at the equilibrium at known temperature. The three models described below in the equations (3.12, 3.13 and 3.14) [18, 82].

$$\frac{C_e}{q_e} = \frac{1}{b\,Q_{max}} + \frac{C_e}{Q_{max}}$$

Equation (3.12): Langmuir isotherm model.

$$\log q_e = \log k_f + \frac{1}{n}\log C_e$$

Equation (3.13): Freundlich isotherm model.

$$q_e = B_T \ln k_T + B_T \ln C_e$$

Equation (3.14): Temkin isotherm model.

Where; Ce: concentration of Cr (VI) at equilibrium and the unit is (mg/l), qe: mass of Cr (VI) removed after reaching the equilibrium per mass of the polymer and the unit is (mg/g), Q_{max}: maximum qe when monolayer fully covered, b: Langmuir isotherm model constant, k_f: Freundlich isotherm models capacity of absorption constant, n: Freundlich isotherm models intensity of absorption constant, n^{-1} symbol of factor of the heterogeneity, B_T: heat of sorption constant, and k_T: Temkin isotherm model constant.

Also non- linear forms of Langmuir, Freundlich and Temkin isotherm models was used to calculate adsorption isotherm parameters, the non- linear models described below in the equations (3.15, 3.16 and 3.17) [79, 82].

$$q_e = Q_{max}\, b\, C_e / (1 + b\, C_e)$$

Equation (3.15): Non- linear Langmuir isotherm model.

$$q_e = k_f\, C_e^{\,1/n}$$

Equation (3.16): Non- linear Freundlich isotherm model.

$$q_e = B_T \ln (k_T C_e)$$

Equation (3.17): Non- linear Temkin isotherm model.

The constants in the non- linear adsorption isotherms models as described above in the linear equations.

Chapter Four

Results and Discussion

4. Chapter Four

Results and discussion

The removal of Cr (VI) ions usually takes place via two mechanisms: adsorption mechanism and reduction mechanism, each one affects the removal of Cr (VI) ions efficiency.

In this study, the cross linked poly (2-allyl-*N*^{1}*, N*^{3}-dihydroxymalonamide) was prepared using diethyl allyl malonate as starting monomer. This polymer was used as an adsorbent for Cr (VI) ions removal from spiked polluted water. Effect of different parameters were investigated to adjusting the optimum high efficient removal condition using batch experiments. The percentage removal was highly depended on the variation of different parameters.

The following results shows the characterization of cross linked poly (2-allyl-*N*^{1}*, N*^{3}-dihydroxymalonamide), and the application of the adsorbent in Cr (VI) removal using variation parameters: pH, adsorbents dosage, initial concentration and time.

4.1 Polymer properties and characterization

The monomer 2-allyl-*N*^{1}, *N*^{3}-dihydroxymalonamide found to be a semisolid, light yellow and water soluble which is prepared according to the hydroxamic acid preparation method that described in methodology chapter, whereas cross linked poly (2-allyl-*N*^{1}, *N*^{3}-dihydroxymalonamide) found to be deep brown, water swellable semisolid polymer and prepared by free radical polymerization.

Both the monomer and polymer were tested by reacting with $FeCl_3$ solution to confirm the attachment of hydroxamic acid by forming a magenta colored complex showing positive result, diethyl allyl malonate also tested but the result is negative as shown in fig (4.1). The magenta colored complex describes the positive result of the reaction with the 2-allyl-*N*^{1}, *N*^{3}-dihydroxymalonamide [83].

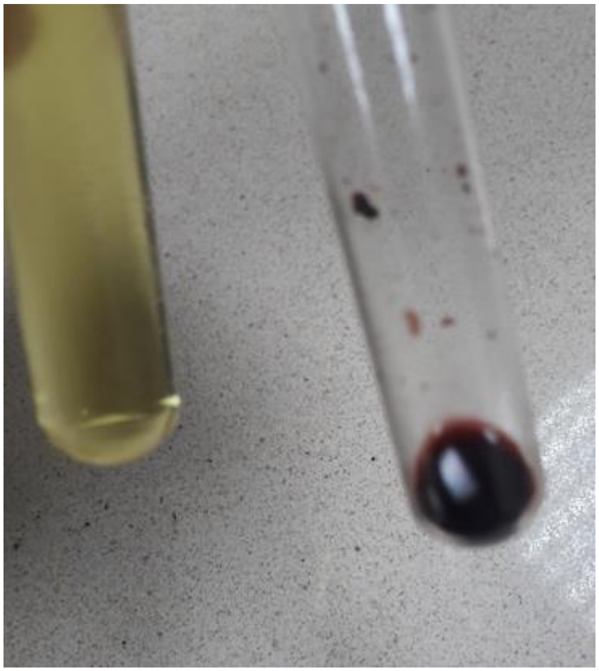


Fig (4.1): Photo of $FeCl_3$ test for 2-allyl-N^1, N^3-dihydroxymalonamide and diethyl allyl malonate.

Diana prepared in her research the same prepared cross linked polymer, poly (2-allyl-N^1, N^3-dihydroxymalonamide), then she characterized it using FTIR. Fig (4.2) below shows the molecular structure of cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) [24].

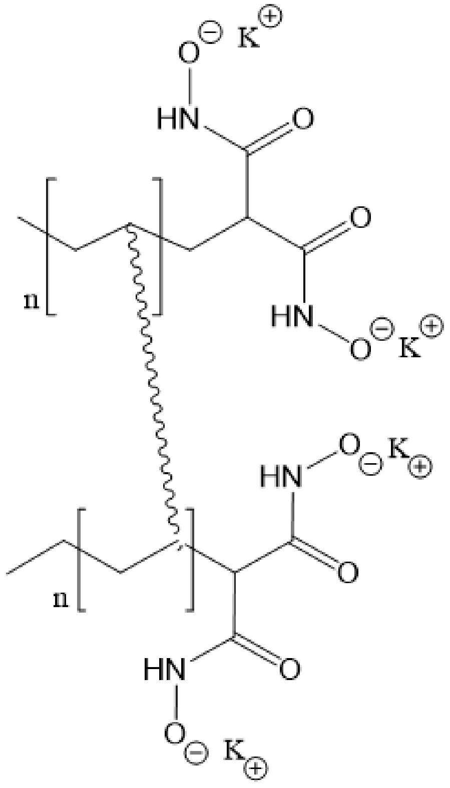


Fig (4.2): Structure of cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) [24].

4.2 Calibration curve

A calibration curve is a graphical method used to determine the concentration of a substance in an unknown sample by comparing the sample's response to that of known standards. In figure (4.3) the variation concentration of Cr (VI) vs Absorbance was summarized. The calibration curve results shown high linearity between the concentration variation and absorption with $R^2 = 0.9988$.

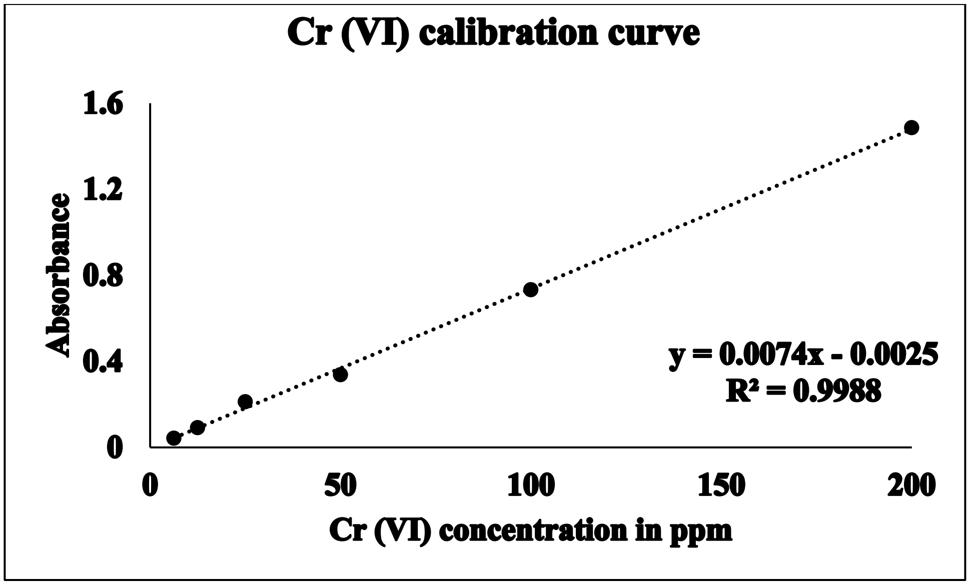


Fig (4.3): Calibration curve of Cr (VI) standards vs the absorbance that obtained using UV- Visible Spectrophotometer at room temperature.

Also in figure (4.4) the variation of total Cr concentration vs Absorbance was summarized. The results shown high linearity with $R^2 = 0.9977$.

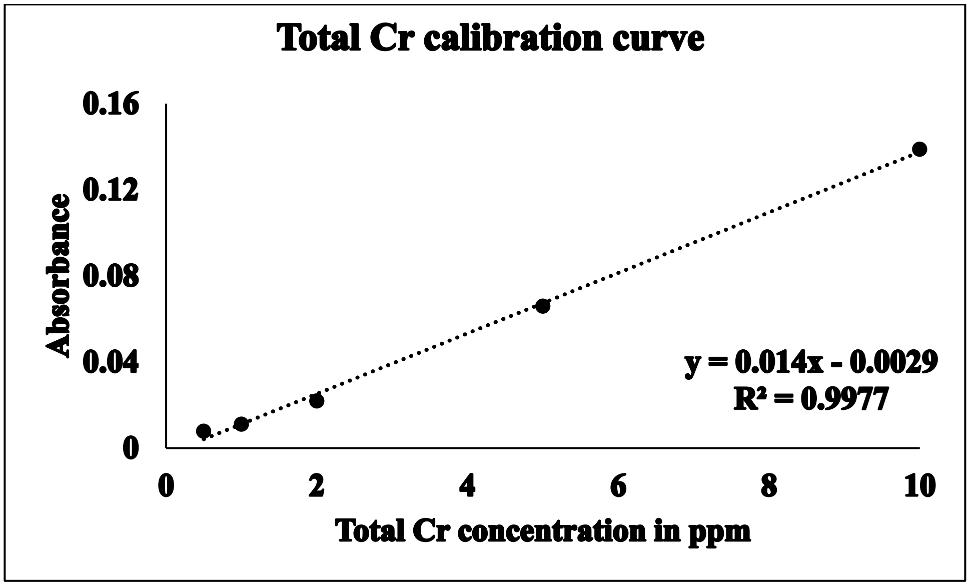


Fig (4.4): Calibration curve of total Cr standards vs absorbance, the absorbance for each standard obtained using AA instrument.

4.3 Cr (VI) removal batch experiments

Distinct batch experiments were conducted to investigate the removal efficiency of Cr (VI) from polluted water using the cross liked polymer with the variation of different parameters; the variation of pH, the variation of initial concentration of Cr (VI), the variation of polymer dosage, kinetic study and the variation of contact time, in addition to adsorption isotherms.

4.3.1 Effect of pH variation

The effect of pH variation was investigated at different pH (2, 4, 6 and 8). Figure (4.5), below and table (4.1) in appendix show the efficiency of reduction of Cr (VI) to Cr (III), the Cr (VI) adsorption efficiency and the total removal efficiency of Cr (VI) by cross linked polymer; poly (2-allyl-*N*^{1}*,* *N*^{3}-dihydroxymalonamide), at different pH values (2, 4, 6 and 8) using 0.1 g polymer dose in 50 ml 10 ppm Cr (VI) solution, the speed was 140 rpm and under room temperature. The results shows that the removal of Cr (VI) from aqueous solution was due to adsorption on the cross linked polymer more than reduction at different pH values. Adsorption capacity also calculated and plotted against pH as described in figure (4.6) below.

The results showed that the highest removal efficiency of Cr (VI) using cross linked poly (2-allyl-*N*^{1}*,* *N*^{3}-dihydroxymalonamide) was detected at pH = 2 with 87.5 % removal efficiency in which the adsorption capacity is the largest capacity and it is equal to 4.4 mg / g. After the removal was studied at different values of pH, the results indicated that approximately 64 % of removal due to adsorption on polymer whereas 23% as reduction of Cr (VI) to Cr (III). All the pH values have the same trends for % removal and adsorption capacity as in the figures.

Table (4.1) in appendix describes the pH effect on Cr (VI) removal, the absorption obtained by both UV and Atomic absorption spectrometry, concentrations of total Cr, Cr (VI) and Cr (III), % removal, and pH values.

The Cr (VI) exists in the form of negatively charged anion, as well as polymer surfaces that were protonated in acidic conditions (pH 2) more than other pH values. Theses protonated functional groups (such as carbonyl) have the ability to make interactions with the negatively charged anion Cr (VI) through electrostatic attraction. Meanwhile, the removal of Cr (VI) by cross linked polymer might still be involved in chemical reduction during adsorption process, Cr (VI) is partially reduced to Cr (III) more than the reduction at other pH values.

Cr (VI) can exist in various anionic forms in aqueous solutions depending on the pH of the solution, such as dichromate ($Cr_2O_7{}^{2-}$), hydrogen chromate ($HCrO_4{}^{-}$), or chromate ($CrO_4{}^{2-}$). It is widely recognized that at lower pH, the dominant species of Cr (VI) is $HCrO_4{}^{-}$. As the pH increases, the concentration of $HCrO_4{}^{-}$ shifts towards other forms like $CrO_4{}^{2-}$ and $Cr_2O_7{}^{2-}$. Therefore, it was concluded that the primary form of Cr (VI) adsorbed by the cross linked polymer in this study is $HCrO_4^-$.

Also it is well known that hydroxamic acids needs acidic conditions to form better complexes so that the removal on pH 2 more than the removal on other pH values [18, 34, 79, 80, 83 - 90].

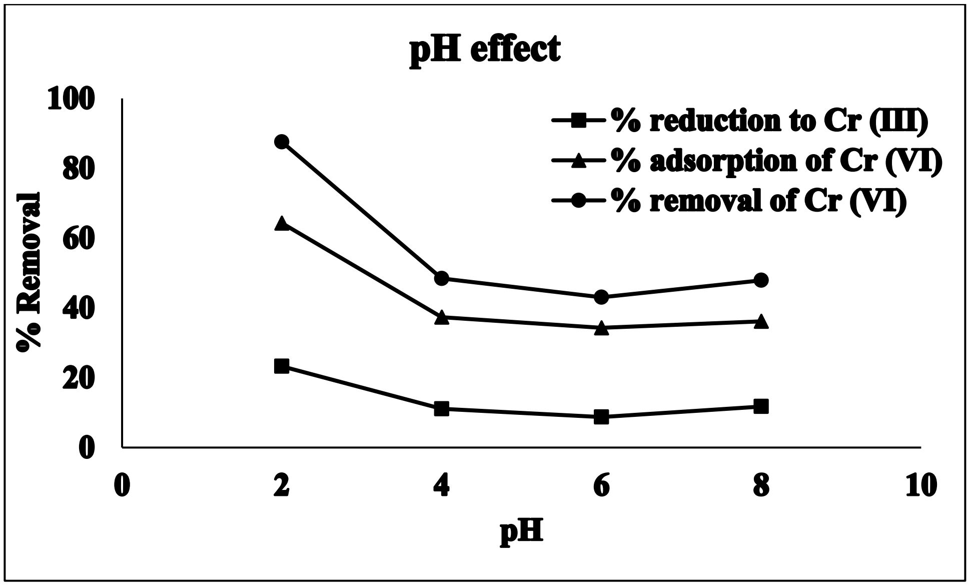


Fig (4.5): pH effect on Cr (VI) removal by cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) at room temperature, initial concentration of Cr (VI) = 10 ppm, contact time 2hrs, 140 rpm, and the polymer dosage= 0.1 g.

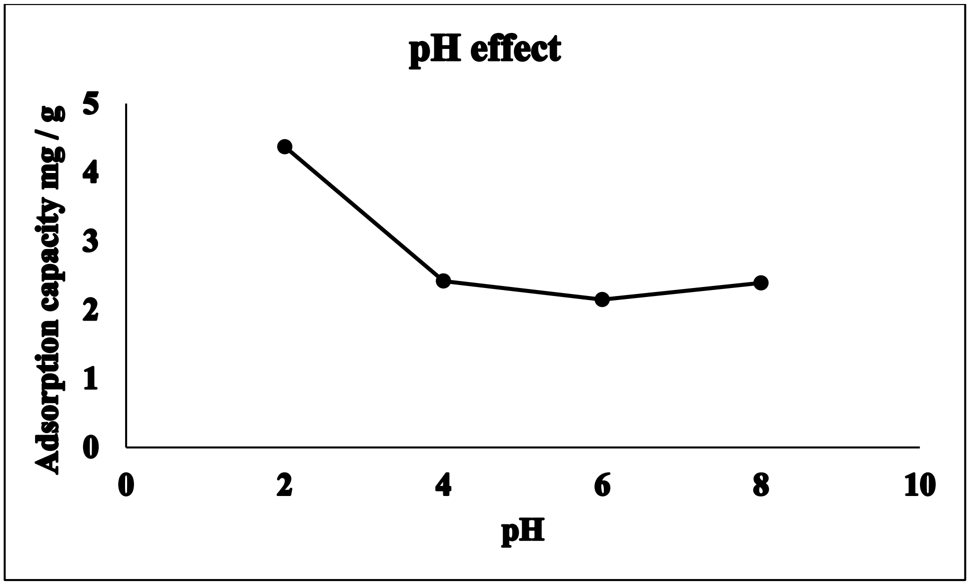


Fig (4.6): pH effect on Cr (VI) removal by cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) at room temperature, initial concentration of Cr (VI) = 10 ppm, contact time 2hrs, 140 rpm, and the polymer dosage= 0.1 g.

4.3.2 Effect of initial concentration of Cr (VI) variation

The effect of initial concentration of Cr (VI) variation was investigated at different initial Cr (VI) concentrations; 10, 20, 50 and 100 ppm. Figure (4.7) below and table (4.2) in appendix show the efficiency of reduction of Cr (VI) to Cr (III), the Cr (VI) adsorption efficiency and the total removal efficiency of Cr (VI) by cross linked polymer poly (2-allyl-N^1, N^3-dihydroxymalonamide) at different initial Cr (VI) concentrations (10, 20, 50 and 100 ppm) using 0.1 g polymer dose in 50 ml solution, the speed was 140 rpm, pH was adjusted to 2 and under room temperature. Adsorption capacity also calculated and plotted against initial concentration of Cr (VI) as described in figure (4.8) below.

The results indicated that the adsorption mechanisms is higher than the reduction for all initial concentration with highest removal efficiency in low initial concentration; 10 ppm of Cr (VI) ions for both removal mechanisms, whereas the higher initial concentration 100 ppm show lowest removal efficiency in both mechanisms adsorption and reduction. But the adsorption capacity as in figure (4.8) have the opposite trend, the adsorption capacity increased with the increases in the initial concentration of Cr (VI).

The increasing of initial concentration of heavy metal will increase the adsorption capacity due to the increasing of concentration gradient between the adsorbent and solution according to the collision theory, but this is only applicable before the polymer saturation level is reached, because the adsorption sites on the polymer surface are fixed so that the polymer can adsorb just limited amounts of Cr (VI) ions, so that when the initial concentration of Cr (VI) increase the adsorption sites on the polymer surface will be occupied, then if the concentration of Cr (VI) increased again there is no way to increase the adsorption because the adsorption sites on the polymer surface was fully occupied and reached the saturation [18, 79, 80, 85 - 88, 91 and 92].

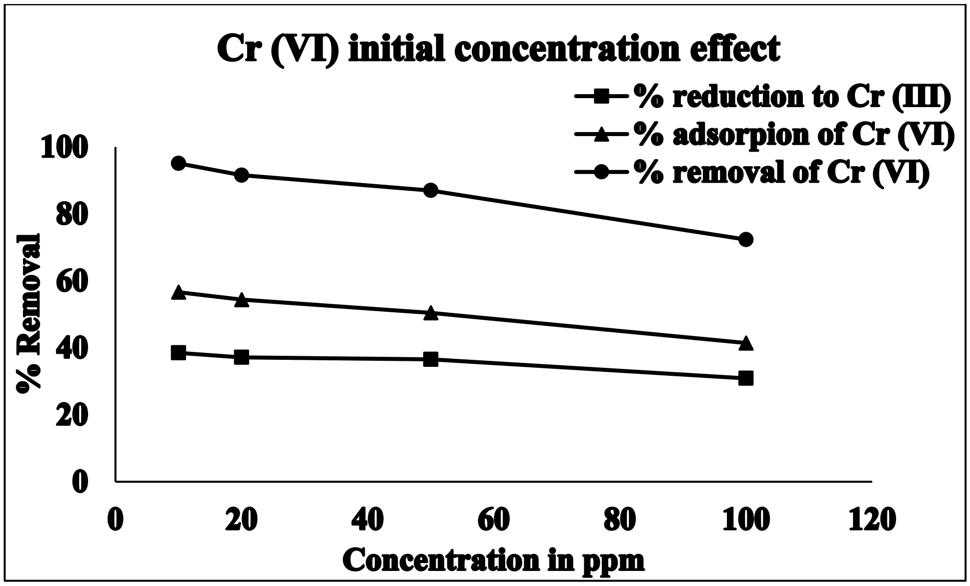


Fig (4.7): Cr (VI) initial concentration effect on % removal by cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) at room temperature, contact time 2hrs, 140 rpm, pH =2, and the polymer dosage= 0.1 g. The concentration in ppm.

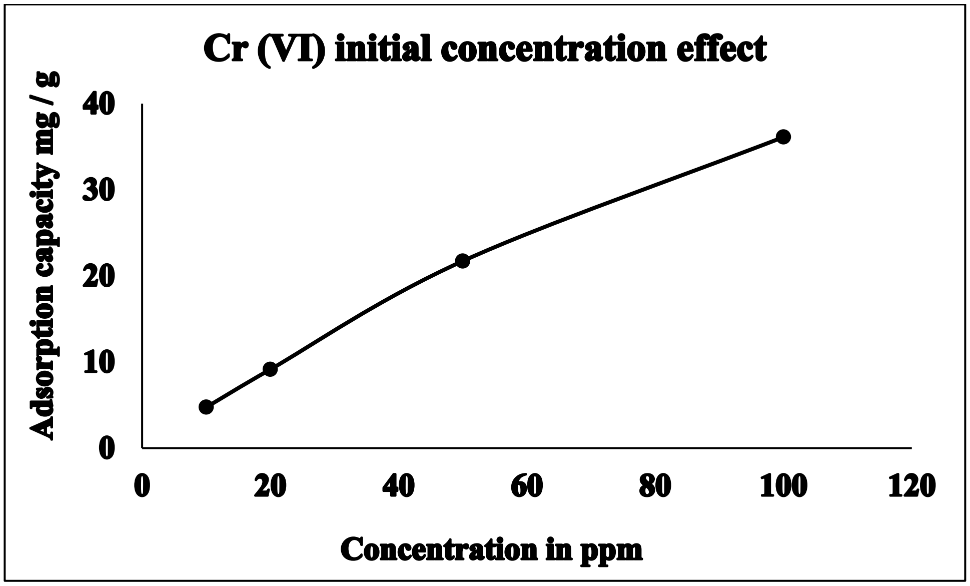


Fig (4.8): Cr (VI) initial concentration effect on adsorption capacity by cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) at room temperature, contact time 2hrs, 140 rpm, pH =2, and the polymer dosage = 0.1 g. The concentration in ppm.

4.3.3 Effect of polymer dosage variation

The effect of polymer dosage variation was investigated at different polymer doses; 0.1, 0.2, 0.3 and 0.4 g. Figure (4.9) below and table (4.3) in appendix shows the efficiency of reduction of Cr (VI) to Cr (III), the Cr (VI) adsorption efficiency and the total removal efficiency of Cr (VI) by cross linked polymer poly (2-allyl-*N*^{1}, *N*^{3}-dihydroxymalonamide) at different polymer doses (0.1, 0.2, 0.3 and 0.4 g) using 50 ml of 10 ppm Cr (VI) solution, pH was adjusted to 2, the speed was 140 rpm and under room temperature. Adsorption capacity also calculated and plotted against polymer dose as described in figure (4.10) below.

The variation dosage of cross linked poly (2-allyl-*N*^{1}, *N*^{3}-dihydroxymalonamide) was studied using constant pH, initial Cr (VI) concentration, temperature, contact time and shack speed. The results indicated that as the polymer dosage increased, the removal efficiency of Cr (VI) also increased, the adsorption mechanisms in variation polymer weights were higher than the reduction. This was attributed to the increase in adsorption sites of the polymer compared to the initial concentration before saturation i.e. more active adsorption sites are available, thereby enhancing the removal of the Cr (VI) ions [18, 79, 80, 85 - 88, and 91].

However, it was found that the adsorption capacity decreased by increasing the cross linked polymer weight. If the polymer weight increased with keeping the Cr (VI) initial concentration constant, the quantity of Cr (VI) adsorbed per unit mass decreased because there were fewer Cr(VI) ions available for adsorption on each unit mass of the cross linked poly (2-allyl-*N*^{1}, *N*^{3}-dihydroxymalonamide). This reduction in adsorption capacity is primarily attributed to the presence of unsaturated sites during the adsorption process [18, 79, and 80].

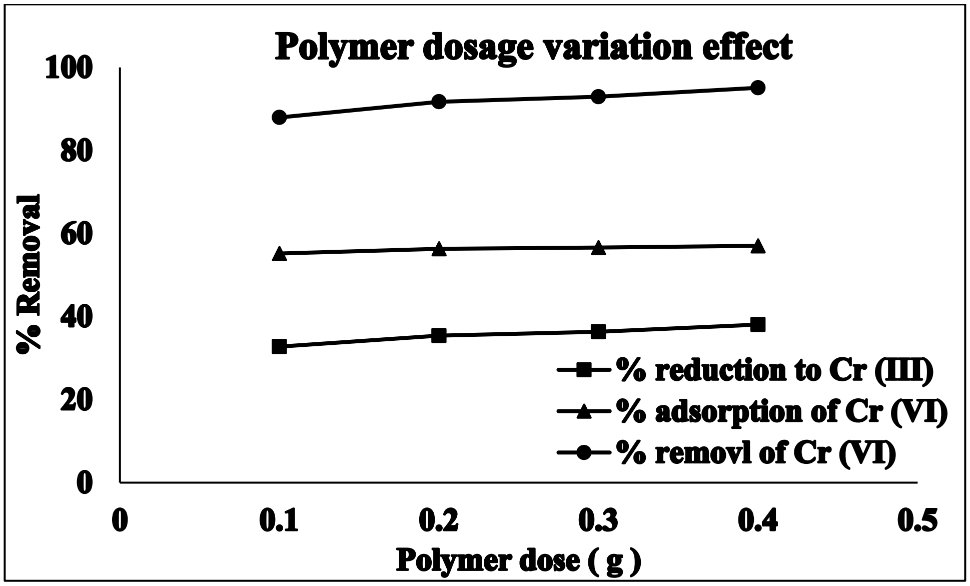


Fig (4.9): Polymer dosage variation effect on Cr (VI) removal by cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) with initial concentration of Cr (VI) = 10 ppm, at room temperature, contact time 2hrs, 140 rpm, and pH 2.

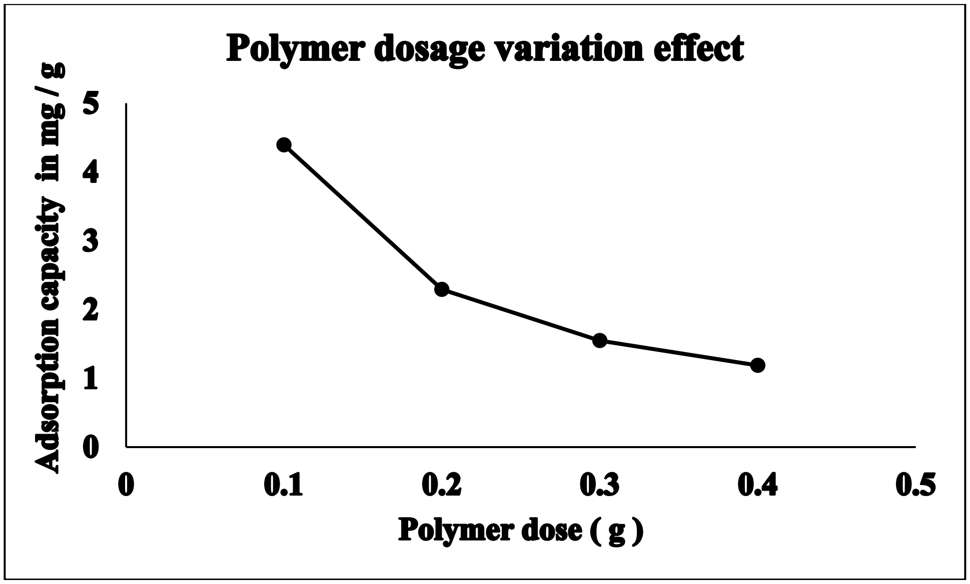


Fig (4.10): Polymer dosage variation effect on Cr (VI) removal by cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) with initial concentration of Cr (VI) = 10 ppm, at room temperature, contact time 2hrs, 140 rpm, and pH 2.

4.3.4 Kinetic study and the effect of contact time

The effect of contact time was investigated at different contact time; from 0 to 300 seconds. Figure (4.11) below and table (4.4) in appendix shows the efficiency of reduction of Cr (VI) to Cr (III), the Cr (VI) adsorption efficiency and the total removal efficiency of Cr (VI) by cross linked polymer poly (2-allyl-*N*^{1}, *N*^{3}-dihydroxymalonamide) at different contact time (from 0 to 300 seconds) using 0.1 g polymer dose in 50 ml 10 ppm Cr (VI) solution, pH was adjusted to 2, the speed was 140 rpm and under room temperature.

Adsorption capacity also calculated and plotted against contact time as described in figure (4.12) below.

One of the important parameters that affects the adsorption of Cr (VI) ions is the contact time. Generally, removal of metal ions increases with an increase in contact time until the equilibrium is achieved.

The results showed that as the contact time between the cross linked polymer and Cr (VI) ions increased the percentage removal of Cr (VI) and adsorption capacity were increased.

The behaviors for the adsorption mechanisms was higher percentage removal than the percentage of the reduction of Cr (VI) to Cr (III). Also the removal process is very fast and the steady state reached after about 3 minutes from the beginning of the process.

Furthermore, adsorption capacity curve have the same properties as removal curve as described in figure (4.12) below. The changes in the adsorption of Cr (VI) capacity were fast for the first 3 min.

From initial rate law method that described in methodology chapter, the order of the reaction was obtained from the slope of the curve that set up from plotting natural logarithm of the rate against the natural logarithm of the concentration.

The results showed that the process is first order as obtained from kinetic calculations that described in table (4.5) in appendix using initial rate law method and the slope was found to be 0.9026 which is approximately equal to 1, see fig (4.13 and 4.14) below. Also it is well known in physical chemistry that adsorption is a first order reaction. [18, 79, 81, 85 - 88, and 91].

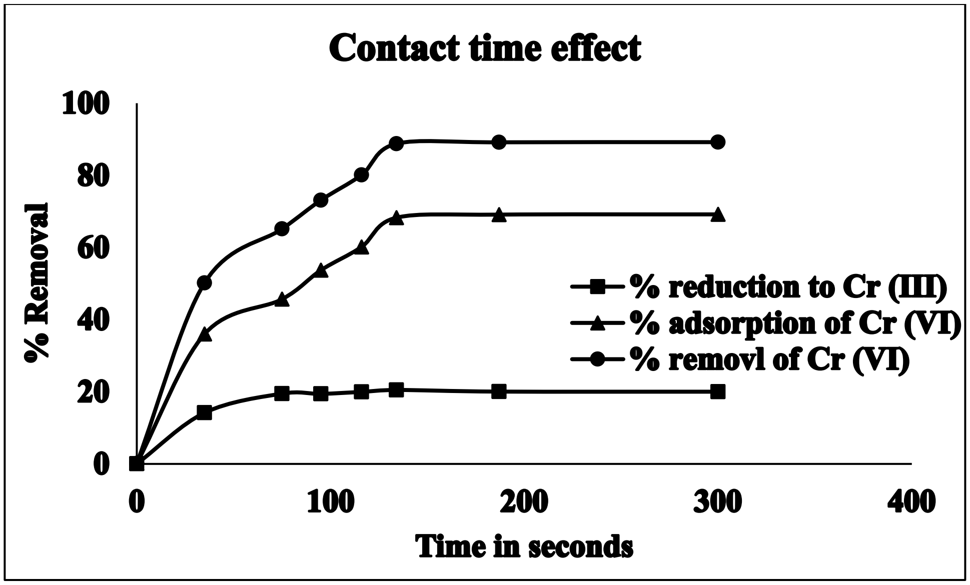


Fig (4.11): Contact time effect on Cr (VI) removal by cross linked poly (2-allyl-*N*^{1}, *N*^{3}-dihydroxymalonamide) at initial concentration of Cr (VI) = 10 ppm, room temperature, contact time 5 min, 140 rpm, and the polymer dosage= 0.1 g.

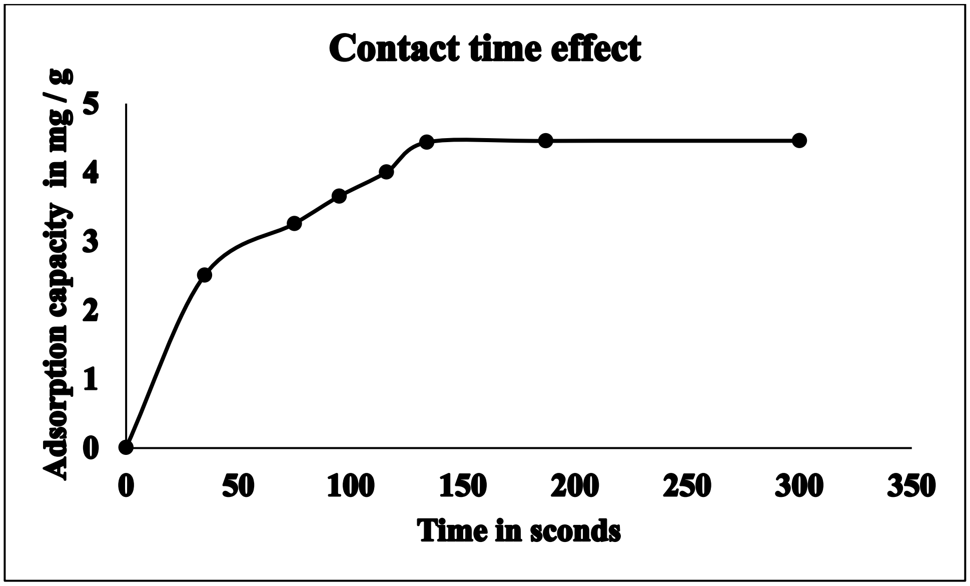


Fig (4.12): Contact time effect on Cr (VI) adsorption capacity by cross linked poly (2-allyl-*N*^{1}, *N*^{3}-dihydroxymalonamide) at initial concentration of Cr (VI) = 10 ppm, room temperature, contact time 5 min, 140 rpm, and the polymer dosage= 0.1 g.

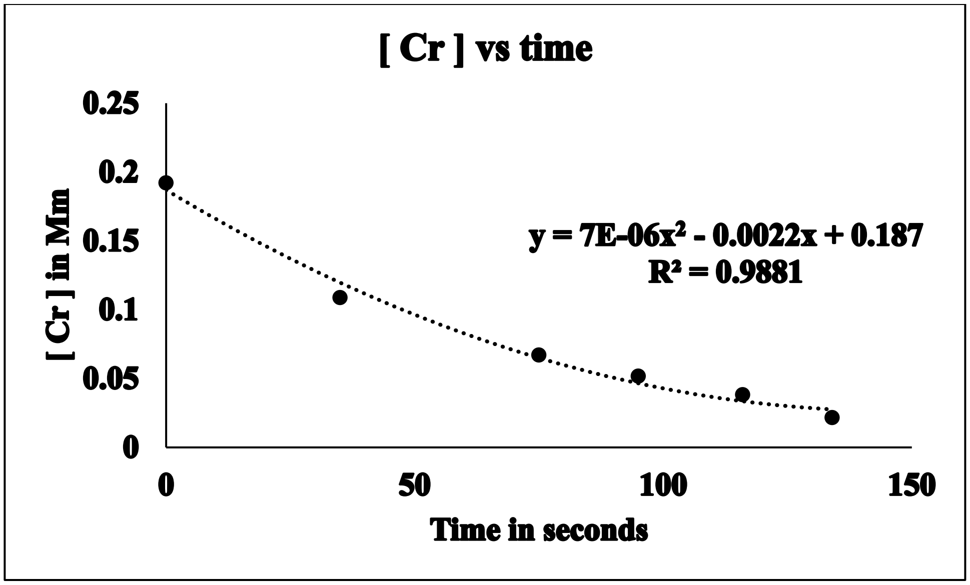


Fig (4.13): [Cr] vs time; Cr (VI) concentration in mM plotted against time in seconds, for the removal of Cr (VI) using cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) at initial concentration of Cr (VI) = 10 ppm, room temperature, contact time 5 min, 140 rpm, and the polymer dosage= 0.1 g. From the curve the following equation was obtained: y = 7E-06 x^2 - 0.0022 x + 0.187, R^2 = 0.9881.

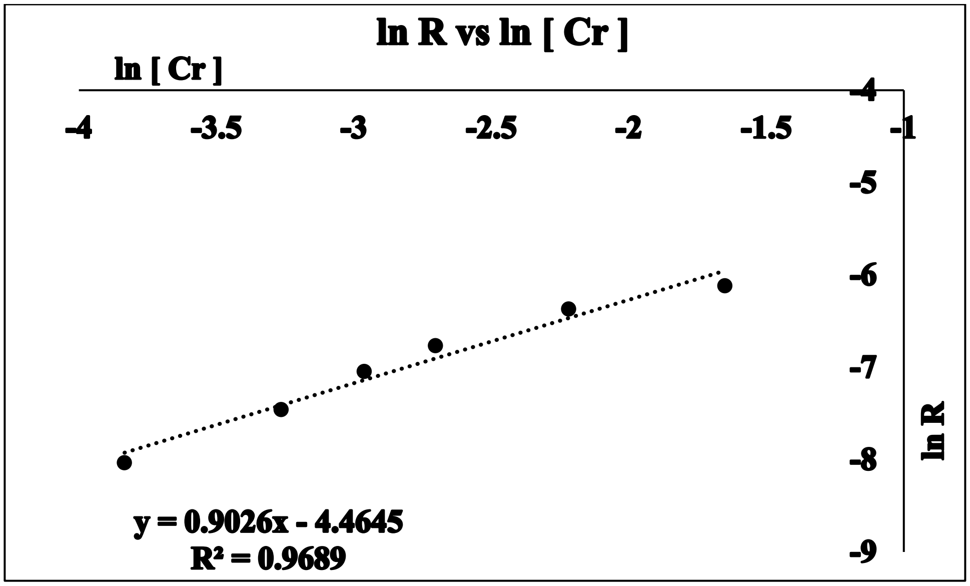


Fig (4.14): ln R vs ln [Cr]; ln concentration of Cr (VI) in mM plotted against ln R, for the removal of Cr (VI) using cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) at initial concentration of Cr (VI) = 10 ppm, room temperature, contact time 5 min, 140 rpm, and the polymer dosage= 0.1 g. From the curve the following equation was obtained: y = 0.9026 x – 4.4645, R^2 = 0.9689.

4.4 Adsorption isotherms

Cross linked poly (2-allyl-*N*^{1}, *N*^{3}-dihydroxymalonamide) adsorption of Cr (VI) behavior was studied using Langmuir, Freundlich and Temkin isotherm models. The behavior studied using the variation concentration of Cr (VI) experiment's results.

Adsorption isotherm models gives a mathematical idea (by curve) about the relationship between the amounts of the matter that will be adsorbed on the surface of polymer and the amounts that will still in the solution at the equilibrium at known temperature [81].

Langmuir isotherm assumes that adsorption will be monolayer, the solid surface is uniform and the adsorption process is independent on the interaction between near molecules.

Because of some Langmuir assumptions failures, such as that often the adsorption enthalpy's negativity begin to be less when the ratio between numbers of adsorption sites occupied to the available sites increases. So that other isotherm models developed, such as Freundlich and Temkin isotherms.

Freundlich isotherm assumes that adsorption will be multilayer, heat of adsorption is equal to heat of desorption and adsorption capacity depends on the adsorbate concentration. Temkin isotherms assumes that for all molecules the heat of adsorption will be decreased in the linear form as the coverage of the solid surface matter increase [93 - 95].

Figures (4.15, 4.16 and 4.17) describes the application of the three models, and the table (4.6) shows the calculations by the equations of the three models that described in methodology chapter.

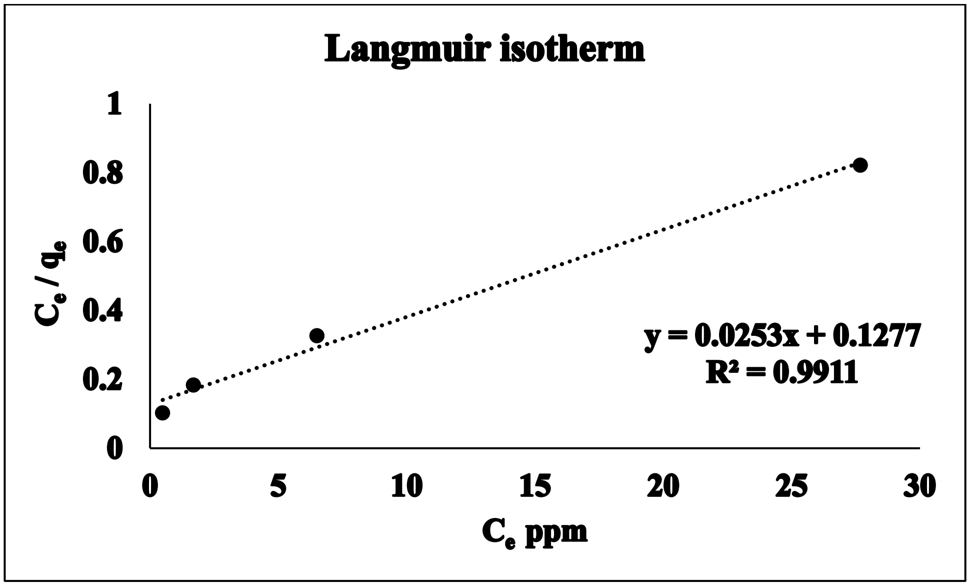


Fig (4.15): Langmuir isotherm model was plotted, using cross linked poly (2-allyl-*N*^{1}, *N*^{3}-

dihydroxymalonamide), polymer dosage = 0.1 g, at room temperature, contact time 2hrs, 140 rpm, and the pH = 2, the curve obtained is: y = 0.0253 x + 0.1277, $R^2 = 0.9911$, $Q_{max} = 39.53$ mg / g, b = 0.198 l/mg.

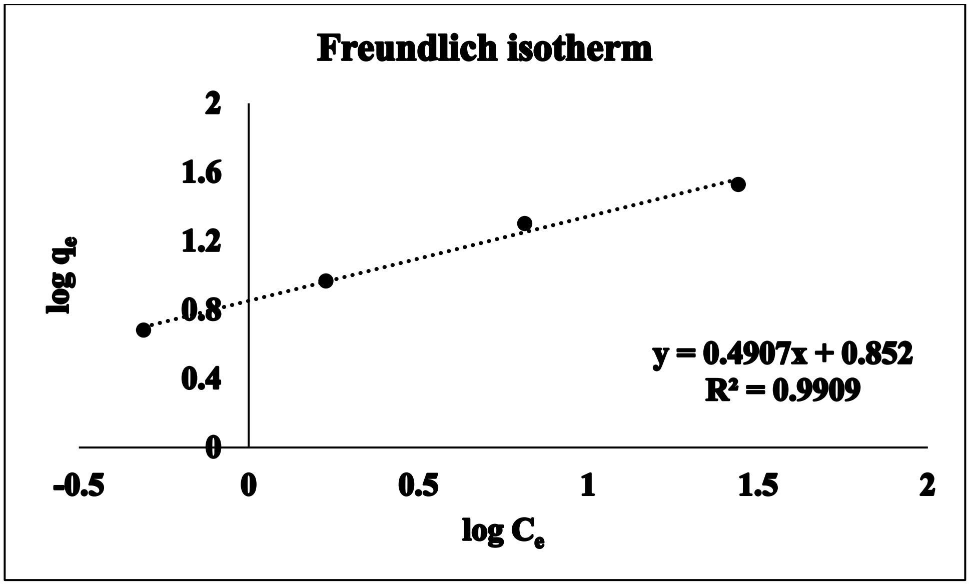


Fig (4.16): Freundlich isotherm model was plotted, using cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide), polymer dosage= 0.1 g, at room temperature, contact time 2hrs, 140 rpm, and the pH = 2, the curve obtained is: y = 0.4907 x + 0.852, $R^2 = 0.9909$, $n^{-1} = 0.4907$, $k_f = 7.1121$.

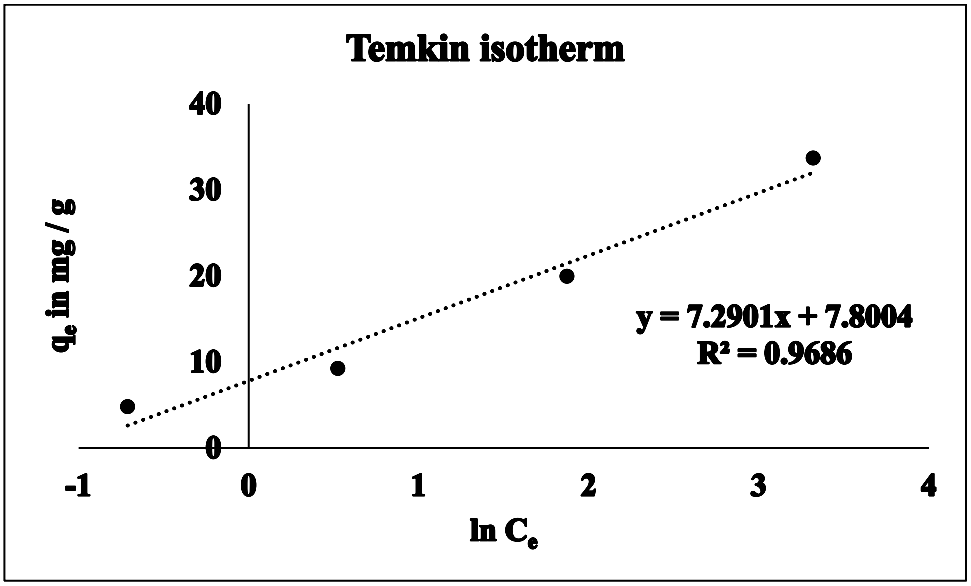


Fig (4.17): Temkin isotherm model was plotted, using cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide), polymer dosage= 0.1 g, at room temperature, contact time 2hrs, 140 rpm, and the pH = 2, the curve obtained is: y = 7.2901 x + 7.8004, $R^2 = 0.9686$, $B_T = 7.2901$, $k_T = 1.07$.

Table (4.6): Shows initial concentrations (I), the absorbance (abs) using UV- Visible Spectrophotometer, Cr (VI) concentration, and isotherm models calculations.

I	UV absorbance	C_e ppm	q_e (mg/g)	C_e/q_e	log C_e	log q_e	ln C_e
10	0.0005	0.491	4.800	0.102	-0.309	0.681	-0712
20	0.0094	1.691	9.251	0.183	0.228	0.966	0.525
50	0.0451	6.505	19.950	0.326	0.813	1.300	1.873
100	0.2021	27.679	33.701	0.821	1.442	1.528	3.321

Q_{max} = 39.53 mg / g obtained from Longmuir model. R^2 of the curves of Cr (VI) removal by cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) shows that the removal process fits Langmuir, Freundlich and Temkin isotherms very well and fits the Langmuir and Freundlich isotherms better and almost similar with each other in R^2 value, so that the polymer may be covered by more than mono layer because of adsorption of Cr (VI), and not just limited to mono coverage. Table (4.7) below describes the three isotherms parameters [79, 81, 86 - 88, and 96].

Table (4.7): The table describes the parameters of Langmuir, Freundlich and Temkin isotherms using linear form.

Isotherm	R^2	Parameters	
Langmuir	0.9911	Q_{max} = 39.53 mg / g	b = 0.198 l/mg
Freundlich	0.9909	n^{-1} = 0.4907	k_f = 7.1121
Temkin	0.9686	B_T = 7.2901	k_T = 1.07

All isotherms have high R^2 values, to avoid any mistakes because of linearity, the non- linear forms of theses isotherms was used, because several researches described that non- linear fitting is more precise than the linear one. In non- linear fitting, the data sets were not transformed, which leads to distortions in the error distributions [79, 82 and 92].

Langmuir, Freundlich and Temkin non- linear isotherm models were obtained using Microsoft excel Solver or Origin software, table (4.8) below describes the three isotherms parameters using the non- linear forms.

Table (4.8): The table describes the parameters of Langmuir, Freundlich and Temkin isotherms using non- linear form.

Isotherm	R^2	Parameters	
Langmuir	0.9654	Q_{max} = 40.75 mg / g	b = 0.1627 l/mg
Freundlich	0.9958	n^{-1} = 0.4383	k_f = 7.9873
Temkin	0.9676	B_T = 7.2901	k_T = 2.9154

The results shows that Freundlich non- linear isotherm model have the higher R^2 value (0.9958), so that the process is heterogeneous adsorption, and the polymer may be covered by more than one layer as discussed in Freundlich isotherm assumptions above. Also Q_{max} = 40.75 mg / g as obtained from non- linear Longmuir isotherm model.

The n^{-1} in Freundlich isotherm models is a parameter that indicates the degree of non- linearity in the adsorption process. Here, n^{-1} value is less than 1, so that a favorable adsorption will takes place, and the process of adsorption is more likely to occur as the concentration increases [18, 79, 80, 82 and 92].

4.5 Comparison

The Q_{max} of cross linked poly (2-allyl-*N*^{1}, *N*^{3}-dihydroxymalonamide) at pH = 2, temp. = 25°C, was found to be 40.75 mg / g. Comparing the result with other works in the table (4.9) gives us a conclusion that the work have good results, high efficiency, and very fast method compared to other works as shown below.

Table (4.9): Materials used in Cr (VI) removal in other works and there Q_{max} compared to this work.

Materials used in Cr (VI) removal	**Q_{max} (mg / g)**	**References**
Polystyrene microplastics	0.473	[22]
Polypropylene microplastics	0.624	[22]
low density polyethylene microbeads	1.9171	[75]
Polyvinyl chloride microplastics	2.44	[22]
Cross linked magnetite - carboxymethyl cellulose - chitosan hydrogel beads	3.93	[41]
Polyethylene microplastics	4.70	[22]
Poly (m - aminobenzene sulfonate) with cellulose nanofibrils	5.263	[73]
Polypyrrole with corncob biochar	19.23	[76]
Micrometer sized polystyrene particles which modified with electromagnetic polyaniline	20.289	[74]
Poly (sodium acrylate) modified magnetite nanoparticles	39.99	[72]
Cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide)	40.75	This work

Chapter Five

Conclusion and Recommendations

5. Chapter Five

Conclusion and Recommendations

5.1 Conclusion

Heavy metals affect humans' life in various fields, one of them is wastewater treatment. Cr (VI) may cause so many harmful effects on human beings and the environment such as stomach cancer, liver damages, asthma and mutagenic effects. It is necessary to remove such toxic metal from our environment. The aim of the study is the removal of Cr (VI) using cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide). It was found that the best removal efficiency of Cr (VI) is in acidic medium when pH = 2 and the process is first order as obtained from kinetic calculations. Adsorption isotherm models were studied, and it was found that the Cr (VI) removal by cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) process fits Langmuir, Freundlich and Temkin isotherms very well, and fits the Freundlich isotherm better in R^2 value. The experiments showed that Q_{max} = 40.75 mg / g. Cr (VI) removal using cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide) is simple, fast and not harming the environment.

5.2 Recommendations

Some of suggestions for future researches related to this work:

1. Investigate the removal of other heavy metals using the cross linked polymer, poly (2-allyl-N^1, N^3-dihydroxymalonamide).
2. Food and agricultural applications of the cross linked polymer, poly (2-allyl-N^1, N^3-dihydroxymalonamide).
3. The medical applications of the cross linked poly (2-allyl-N^1, N^3-dihydroxymalonamide), in vitro and in vivo.

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Appendix

Table (4.1): Shows various pH, the absorbance (abs) using UV- Visible Spectrophotometer and AAS, and the % removal calculations results. All concentrations in ppm.

pH	2	4	6	8
AAS absorbance	0.0473	0.0852	0.0895	0.0869
Total Cr concentration in ppm	3.572	6.264	6.570	6.390
UV absorbance	0.0061	0.0351	0.0391	0.0355
Cr (VI) in solution concentration in ppm	1.246	5.157	5.696	5.211
Cr (III) in solution concentration in ppm	2.326	1.108	0.874	1.174
Adsorbed Cr (VI) concentration in ppm	6.428	3.736	3.430	3.615
Adsorption percentage (%)	64.279	37.355	34.301	36.148
Reduction percentage (%)	23.262	11.076	8.736	11.744
Percentage of Cr (VI) in solution (%)	12.460	51.569	56.963	52.108
Percentage of Cr (VI) removal (%)	87.541	48.431	43.037	47.892
Adsorption percentage + reduction percentage + percentage of Cr (VI) in solution (%)	100	100	100	100

Table (4.2): Shows initial concentrations (I), the absorbance (abs) using UV- Visible Spectrophotometer and AAS, and the % removal calculations results. All concentrations in ppm.

Initial concentrations (I)	10	20	50	100
AAS absorbance	0.0581	0.1254	0.3458	0.8213
Total Cr concentration in ppm	4.339	9.120	24.777	58.556
UV absorbance	0.0005	0.0094	0.0451	0.2021
Cr (VI) in solution concentration in ppm	0.491	1.691	6.505	27.679
Cr (III) in solution concentration in ppm	3.849	7.429	18.272	30.878
Adsorbed Cr (VI) concentration in ppm	5.661	10.880	25.223	41.444
Adsorption percentage (%)	56.607	54.399	50.446	41.444
Reduction percentage (%)	38.486	37.146	36.544	30.878
Percentage of Cr (VI) in solution (%)	4.907	8.455	13.011	27.679
Percentage of Cr (VI) removal (%)	95.093	91.545	86.989	72.321
Adsorption percentage + reduction percentage + percentage of Cr (VI) in solution (%)	100	100	100	100

Table (4.3): Shows polymer dosage, the absorbance (abs) using UV- Visible Spectrophotometer and AAS, and the % removal calculations results. All concentrations in ppm.

Polymer dosage in g	0.1	0.2	0.3	0.4
AAS absorbance	0.0601	0.0585	0.0581	0.0575
Total Cr concentration in ppm	4.481	4.368	4.340	4.297
UV absorbance	0.0058	0.0030	0.0021	0.0005
Cr (VI) in solution concentration in ppm	1.206	0.828	0.707	0.491
Cr (III) in solution concentration in ppm	3.276	3.540	3.633	3.806
Adsorbed Cr (VI) concentration in ppm	5.519	5.632	5.661	5.703
Adsorption percentage (%)	55.186	56.323	56.607	57.033
Reduction percentage (%)	32.759	35.399	36.328	38.060
Percentage of Cr (VI) in solution (%)	12.055	8.279	7.065	4.907
Percentage of Cr (VI) removal (%)	87.945	91.721	92.935	95.093
Adsorption percentage + reduction percentage + percentage of Cr (VI) in solution (%)	100	100	100	100

Table (4.4): Shows contact time in seconds (t), the absorbance (abs) using UV- Visible Spectrophotometer and AAS, and the % removal calculations results. All concentrations in ppm.

contact time in seconds (t)	0	35	75	95	116	134	187	300
AAS absorbance	0.1376	0.0871	0.0735	0.0622	0.0531	0.0417	0.0405	0.0404
Total Cr concentration in ppm	9.987	6.399	5.433	4.631	3.984	3.174	3.089	3.082
UV absorbance	0.0709	0.0338	0.0227	0.0168	0.0116	0.0116	0.0052	0.0049
Cr (VI) in solution concentration in ppm	9.985	4.982	3.485	2.689	1.988	1.125	1.084	1.080
Cr (III) in solution concentration in ppm	0.002	1.418	1.949	1.942	1.997	2.050	2.005	2.002
Adsorbed Cr (VI) concentration in ppm	0.013	3.601	4.567	5.369	6.016	6.826	6.911	6.918
Adsorption percentage (%)	0.131	36.005	45.667	53.694	60.159	68.257	69.110	69.181
Reduction percentage (%)	0.002	14.179	19.487	19.416	19.965	20.497	20.049	20.019
Percentage of Cr (VI) in solution (%)	99.849	49.816	34.846	26.889	19.877	11.246	10.841	10.800
Percentage of Cr (VI) removal (%)	0.151	50.184	65.154	73.111	80.123	88.754	89.159	89.200
Adsorption percentage + reduction percentage + percentage of Cr (VI) in solution (%)	100	100	100	100	100	100	100	100

Table (4.5): Shows contact time in seconds (t), the absorbance (abs) using UV- Visible Spectrophotometer period of time, and kinetic calculations.

t	UV abs	[Cr (VI)] ppm	[Cr] mM	R mM/s	ln [Cr]	ln R
0	0.0709	9.985	0.192	0.0022	- 1.650	- 6.119
35	0.0388	5.656	0.109	0.0017	- 2.219	- 6.371
75	0.0227	3.485	0.067	0.0012	- 2.703	- 6.768
95	0.0168	2.689	0.052	0.0009	- 2.963	- 7.047
116	0.0116	1.988	0.038	0.0006	- 3.264	- 7.459
134	0.0052	1.125	0.022	0.0003	- 3.834	- 8.035

Fig (4.18): Photo of batch experiment of polymer dosage variation effect on Cr (VI) removal by poly (2-allyl-*N*^{1}, *N*^{3}-dihydroxymalonamide) at initial concentration of Cr (VI) = 10 mg L^{-1}, room temperature, contact time 2hrs, the speed is 140 rpm, and pH 2. The four Erlenmeyer flasks contains 0.1, 0.2, 0.3, and 0.4 g of the polymer respectively starting from left.

Fig (4.18): Photo of batch experiment of polymer dosage variation effect.

الملخص

للبوليمرات تطبيقات كثيرة في حياة الإنسان، ونتيجة لذلك فإن أكثر من نصف المواد الكيميائية المحضرة في المجال الصناعي هي بوليمرات. ومن هذه التطبيقات المهمة معالجة المياه الملوثة. ولا شك أن الكروم السداسي التكافؤ هو أحد الملوثات الرئيسية لأنه قد يسبب العديد من الآثار الضارة على البشر والبيئة. لذلك تمت دراسة إزالة الكروم السداسي التكافؤ باستخدام بوليمر المتقاطع بولي (2- اليل - N^3, N^1 - ثنائي هيدروكسيمالوناميد)، وأجريت تجارب مختلفة لمعرفة كفاءة إزالة الكروم السداسي التكافؤ من المياه الملوثة باستخدام البوليمر المتقاطع بمتغيرات مختلفة: الرقم الهيدروجيني، والتركيز الأولي، ووزن الجرعة، وآلية الازالة مع الزمن بالإضافة إلى معادلة الادمصاص، ثم تمت مقارنتها بأعمال أخرى. وجد أن أفضل كفاءة إزالة للكروم السداسي التكافؤ تكون في وسط حمضي عندما يكون الرقم الهيدروجيني = 2، و كلما زاد التركيز الأولي للكروم السداسي التكافؤ قلت نسبة الإزالة، وبزيادة جرعة البوليمر تزداد أيضًا كفاءة إزالة الكروم السداسي التكافؤ ، وكذلك تزداد إزالة الكروم السداسي التكافؤ بزيادة زمن التلامس ، و كانت العملية من الرتبة الأولى كما تم الحصول عليها من نتائج الحسابات آلية الازالة مع الزمن. تمت دراسة سلوك امتزاز بولي (2- اليل - N^3, N^1 - ثنائي هيدروكسيمالوناميد) عند تغير التركيز للكروم السداسي التكافؤ باستخدام نماذج امتزاز لانجموير وفرويندليش وتمكين. يوضح R^2 لمنحنيات إزالة الكروم السداسي التكافؤ بواسطة بولي (2- اليل - N^3, N^1 - ثنائي هيدروكسيمالوناميد) أن عملية الإزالة تناسب معادلة لانجموير وفرويندليش وتمكين بشكل جيد للغاية، وتناسب معادلة فرويندليش بشكل أفضل، وان السعة القصوى للامتزاز حسب لانجموير يساوي 40.75 ملغم / غم.