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Highly efficient 1,3,4- thiadiazole impregnated cross-linked hydrogel: water retention and adsorption of phenol red for sustainable environment

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Abstract
In this study, free radical copolymer was used to prepare a hydrogel of acrylic acid poly (acrylic acid-co-2-amino-5-(2-chlorophenyl)-1,3,4-thiadiazole) (poly-OCTMAC). The poly-OCTMAC hydrogel was characterized using Fourier transform infrared spectroscopy FTIR, ^{1}H-NMR was measured using (Bruker Ultra Shield), 500 MHz, using DMSO-d6 solvent and reference. thermogravimetric analysis (TGA), and field emission scanning electron microscopy (SEM). Phenol red (PhR) dye was removed from the aqueous medium using the poly-OCTMAC hydrogel. To determine the maximum adsorption, the effects of initial dye concentration, contact time, and adsorbent dosage were investigated. The poly-OCTMAC hydrogel had a high swelling ratio of 456%. The high water penetration rate of the poly-OCTMAC hydrogel coupled with the high swelling rate exposed the internal adsorption sites for the adsorption of PhR dye. The adsorption performance of the poly-OCTMAC gel was explained by quasi-first-order adsorption models. Moreover, after three different times, the poly-OCTMAC maintained 80% absorption efficiency. The high water holding capacity of Poly-OCTMAC improved the moisture holding limit of soil for irrigation up to 24 days. As a result, Poly-OCTMAC can be used in the removal of toxic dyes as well as in irrigation systems.
Keywords: Acrylic acid, phenyl red (PhR) dye adsorption, swelling polymers, cross-linked soluble polymers.

1. INTRODUCTION

Recently, there have been several attempts to test methods for producing inexpensive and more effective biopolymer-based adsorbents for dye removal. In fact, it is now known that biopolymer-based adsorbents are effective and economical alternatives for water purification [1]. Their release into the environment leads to serious problems in ecosystems such as decreased photosynthetic pathways, decreased oxygen levels, and even suffocation of animals and plants [2]. Several techniques including chemical oxidation [3], absorption/biosorption [4,5], coagulation/precipitation/coagulation [6], oxidation processes, ion exchange, biodegradation, and membrane filtration have been proposed for dye removal from wastewater. Dyes are widely used in many industries such as textiles, rubber, paper, plastics, etc. More than 7 × 105 tons of 10,000 different commercial dyes are produced annually worldwide. It has been estimated that about 10-15% of these dyes are lost during the dyeing process and released as waste [7]. The presence of these dyes in water, even at very low concentrations, is highly visible and undesirable. Many dyes are difficult to decompose due to their complex structure, and some are toxic, mutagenic, and carcinogenic [8]. Therefore, the removal of dyes from industrial wastewater before discharge into the environment is of great importance. Many techniques such as oxidation [9–11], biological treatment [12–15], or application of activated carbon [16–21], ion exchange [22–24], and chitosan [25,26] have been developed to remove water-soluble acidic dyes. At present, the most common treatment for effective dye removal is adsorption. The adsorption behavior of the isoforms of 2-amino(substituted phenyl)-1,3,4-thiazole has also been used as a corrosion inhibitor on copper surfaces [27]. The adsorption of methyl orange and methyl blue dyes by porous polymer has also been used [28]. Polymers have received great attention due to their wide application in the fields of electrode materials, sensors, absorbents, cathode materials for rechargeable lithium-ion batteries, etc. [29–32]. As a new type of functional aromatic heterocyclic polymers, polymers containing 1,3,4-thiadiazole ring have revealed a promising future in the fields of rechargeable lithium batteries, biological and chemical sensors, clinical diagnosis and pharmaceutical studies, optoelectronic devices, and heavy metal ion absorbents due to their unique energy storage performance, electrocatalytic activity, and electron-rich properties [33–35]. Polymers containing 1,3,4-

thiadiazole ring can be synthesized either by electrochemical oxidation [36–38] or by chemical oxidation [39, 40, 41]. 1,3,4-thiadiazole has been used to prepare poly-2-mercapto-1,3,4-thiadiazole (PTT) and used to selectively adsorb mercury and silver ions from aqueous solutions. 1,3,4-thiadiazole has been used in the preparation of polymers for rechargeable metal batteries [42]. Hydrogels have received increasing attention from researchers due to their theoretical and practical importance. Hydrogels are a network of hydrophilic polymers that can swell in water and hold a large amount of water by crosslinking. Crosslinking can be provided by covalent and hydrogen bonding, van der Waals interaction and physical crosslinking [43, 44]. Hydrogels are one of the most promising types of polymers used in the development of tissue culture materials, since [45]. Polymer gels are porous fluid-saturated flexible materials.

In this research, a scientific approach will be applied that includes the preparation of a polymer gel loaded with 1,3,4-thiadiazole derivative by free radical copolymerization and its characterization will be done using F-TIR, TGA, SEM, XRD, where swelling in solvents such as water and methanol will be studied as well as solubility in acidic and basic solutions.

The main objective of the research is to study the ability of the polymer to remove Phenol Red dye in its aqueous solutions and its ability to absorb large amounts of water to maintain soil moisture for irrigation purposes.

2. EXPERIMENTAL

2.1. Materials and reagents

acrylic acid ($C_3H_4O_2$, 98%), 2-chloro benzoic acid ($C_7H_5ClO_2$, 97%), thiosemicarbazide (CH_5N_3S, 98%), Phosphorous oxy chloride ($POCl_3$, 99%) potassium hydroxide (KOH, 97%), methanol (CH_3,99.9), azobisisobutyronitril ($C_8H_{12}N_4$, 98%), benzene (C_6H_6, 99%) phenol red ($C_{19}H_{14}O_5S$, 88%) .

2.2. Instrumentation

The infrared spectra of the compound and poly-OCTMAC were measured in the range of (4000-400) cm^{-1} using an IR Affinity (SHIMADZU) device using a (KBr) disk in the frequency range. (Rheometric Scientific TGA-1000) device was used to record the TGA of the prepared polymer, a (ZEISS) scanning electron microscope was used to measure the (SEM) of the prepared poly-OCTMAC, the absorbance of poly-OCTMAC recorded was measured on a (SHIMADZU-1700) dual UV-visible spectrophotometer in the presence of a quartz cell using KOH solvent, and the UV-visible spectra were measured on a (SHIMADZU 1700 UV) spectrophotometer.

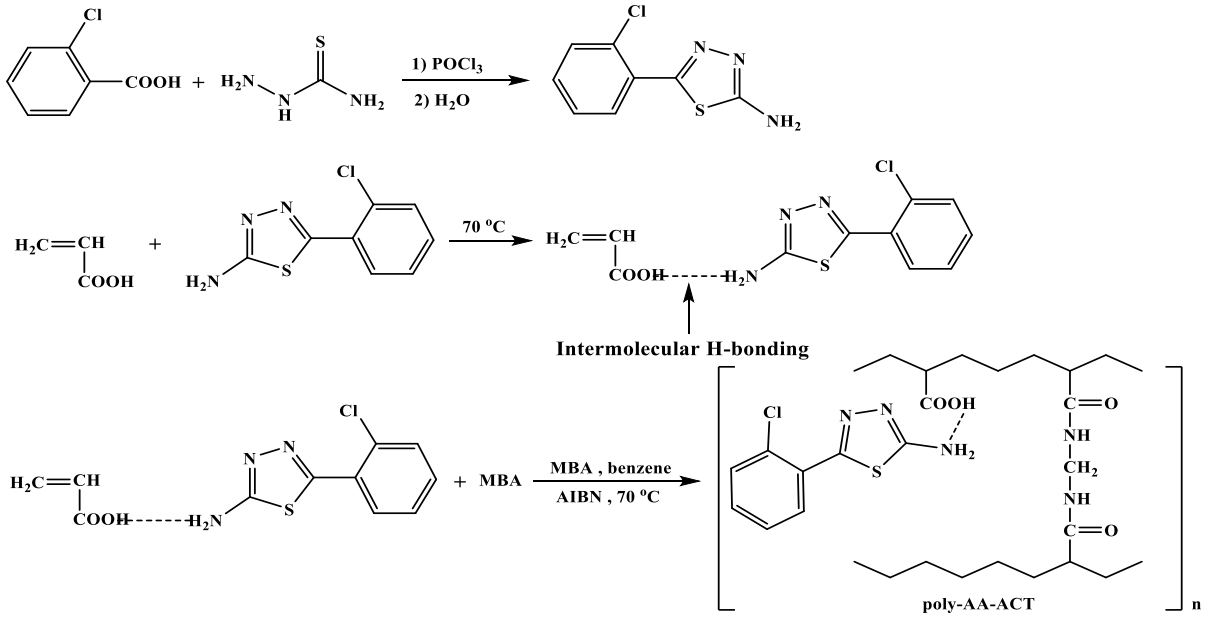
2.3. stepwise synthesis of poly-OCTMAC hydrogel

2.3.1. Synthesis of 2-amino-5-(2-chloro phenyl)-1,3,4-thiadiaozle

2-chloro benzoic acid (0.01 mol) was refluxed with thiosemicarbazide (0.01 mol) in (10 ml) $POCl_3$ in a 100 ml round bottomed flask. The reaction was then heated for three hours, after that (30 ml) of distilled water was added to it and the mixture was heated for four hours. The mixture was then neutralized using (10%) potassium hydroxide (KOH), the solution was filtered, the resulting precipitate was washed several times with distilled water, and the precipitate was left to dry.

2.3.2. Synthesis of poly-OCTMAC hydrogel

The polymer were prepared using the solution polymerization technique and the free radical polymerization method by dissolving 2-amino-5-(2-chloro phenyl)-1,3,4-thiadiaozle (0.5 g) in acrylic acid (5 ml), completely dissolving it with occasional heating, then benzene (30ml) was added to the mixture with continuous stirring, methylene bisacrylamide (1 g) was added to the mixture. At this point, N_2 gas was pumped into the mixture and the gases were removed using a Shlenk device. The mixture was reflaxed using a water bath at 75 ^{o}C. When this temperature was reached, the initiator azobisisobutyronitrile (AIBN) (0.008 mol) was added with stirring rite (300 rpm). After a while, the polymer began to form, and it was observed that (N_2) gas was released resulting from the disintegration of the initiator. After the reaction ended, methanol was added to isolate the polymer from the benzene to complete polymer formation and preciptation.



2.4. Swelling studies of hydrogels

2.4.1 Swelling

(0.5 g) of the polymer was weighed and (20 ml) of distilled water was added to it and the same amount of weight of the polymer was taken and dissolved in (20 ml) of absolute methanol. Swelling was studied through the change in weight at time (0 h, 3 h, 6 h,9 h, 12 h, 24 h) as in the equation below:

$$\text{Swelling percentage \%} = \frac{\text{ws} - \text{wd}}{\text{wd}} \times 100$$

Ws: weight after swelling

Wd: dried weight (weight before swelling).

2.5. Adsorption studies of PhR dye

PhR adsorption analysis on poly(OCTMAC) hydrogel was performed via UV–vis spectroscopey parameters, contact with time, to achieve ideal adsorption conditions. Spectrophotometric analysis was carried out through UV–Vis spectra by using double-beam spectrophotometer at 432 nm, deionized water was utilized as reference solvent. For a specific PhR adsorption, 0.1 g of poly(OCTMAC) hydrogel dose was used.

$$\text{Adsorption efficiency \%} = \frac{\text{Co} - \text{Ct}}{\text{Co}} \times 100$$

2.6. Water-retention study of poly-OCTMAC hydrogel

The poly-OCTMAC hydrogel water retention analysis was performed in soil collected from Diyala Iraq University. 20 g soil and 2 g poly-OCTMAC hydrogel were homogenized in a plastic container, 30 mL water was added gradually, and the weight (W1) was determined using a weighing machine. The container was weighed every day (W2) and kept at room temperature until there was no consistency in weight loss. The water loss ratio (W %) of soil samples was calculated through standard formula as:

$$\text{Water retention \%} = \frac{W1 - W2}{30} \times 100$$

3. RESULTS AND DISCUSSION

3.1. Mechanism of hydrogel formation

Poly-OCTMAC hydrogel was formed using the copolymerization method. The temperature used in the copolymerization created the reactive sites on the monomers and led to the polymerization propagation. AIBN was a thermo-initiator, and when decomposed at high temperatures, it formed isobutyronitrile radicals and initiated the polymerization of acrylic acid to produce the network chain of poly-OCTMAC.

The monomer molecules that were in direct contact with the active sites became radical acceptors, leading to the chain initiation, and then the surrounding molecules became free radical donors. The vinyl units of the cross-linking agents could react with the polymer network during the chain propagation to develop the poly-OCTMAC hydrogel network. Table (1) shows the swelling percentage in water for 24 h, Table (2) shows the swelling percentage in methanol after 24 h, and Figure (1) and (2) show the swelling percentage in water and methanol.

Table (1): Percentage of swelling in water after 24 hours poly-OCTMAC

No	Swelling						Percentage of swelling
	0 h	3h	6 h	9 h	12 h	24 h	
poly-OCTMAC	0.5	0.68	0.73	1.02	1.15	2.78	456%

Table (2): Swelling ratio of polymers prepared in methanol poly-OCTMAC

No	Swelling						Percentage of swelling
	0 h	3h	6 h	9 h	12 h	24 h	
poly-OCTMAC	0.5	0.65	0.80	1.09	1.64	2.65	430%

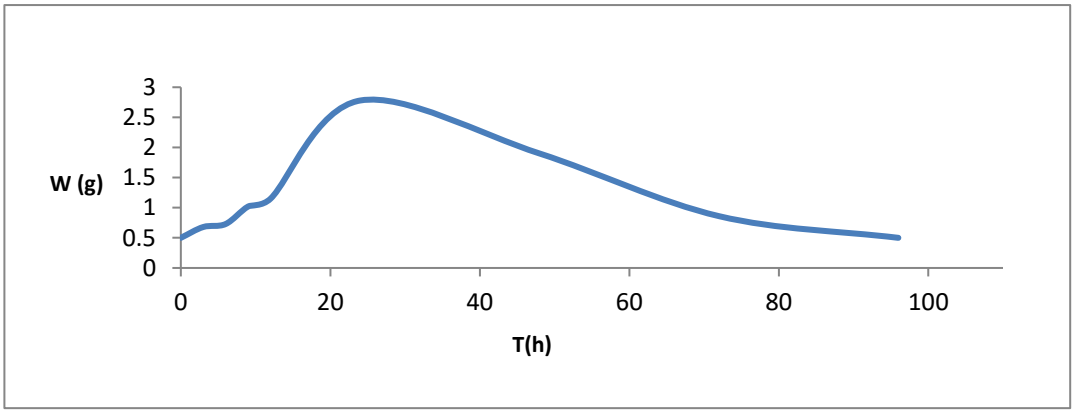


Fig.1. swelling of poly-OCTMAC in water after 24h.

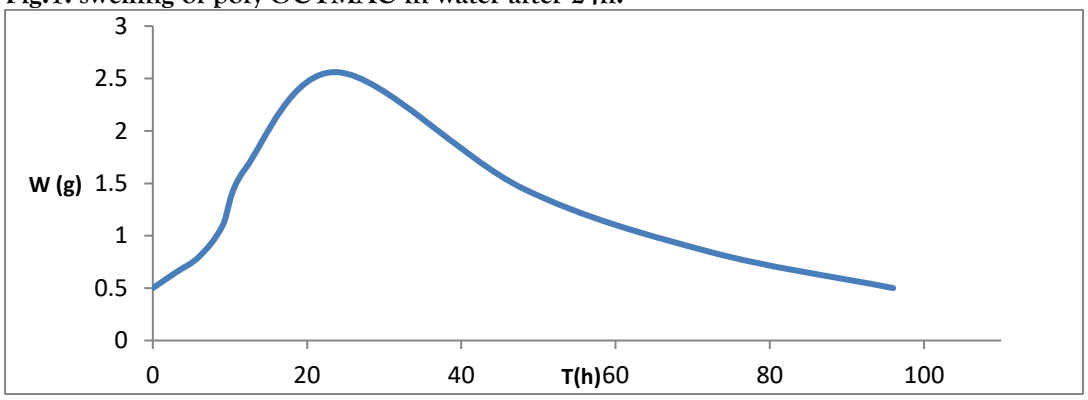


Fig . 2. swelling of poly-OCTMAC in methanol after 24 h.

3.3. Characterization

3.3.1. FTIR of 2-amino-5-(2-chloro phenyl)-1,3,4-thiadiaozle

The compound was characterized by infrared spectrum, where absorption bands (3248, 3300) cm^{-1} were observed, which belong to the (NH_2) band, the band at (3015) cm^{-1}, which belongs to the aromatic (C-H) group, and the band at (1635) cm^{-1}, which belongs to the (C=N) group. As for the aromatic (C=C) band, it appeared at (1519) cm^{-1}. The figure (3) shows the infrared spectrum of the compound 2-amino-5-(2-chloro phenyl)-1,3,4-thiadiaozle.

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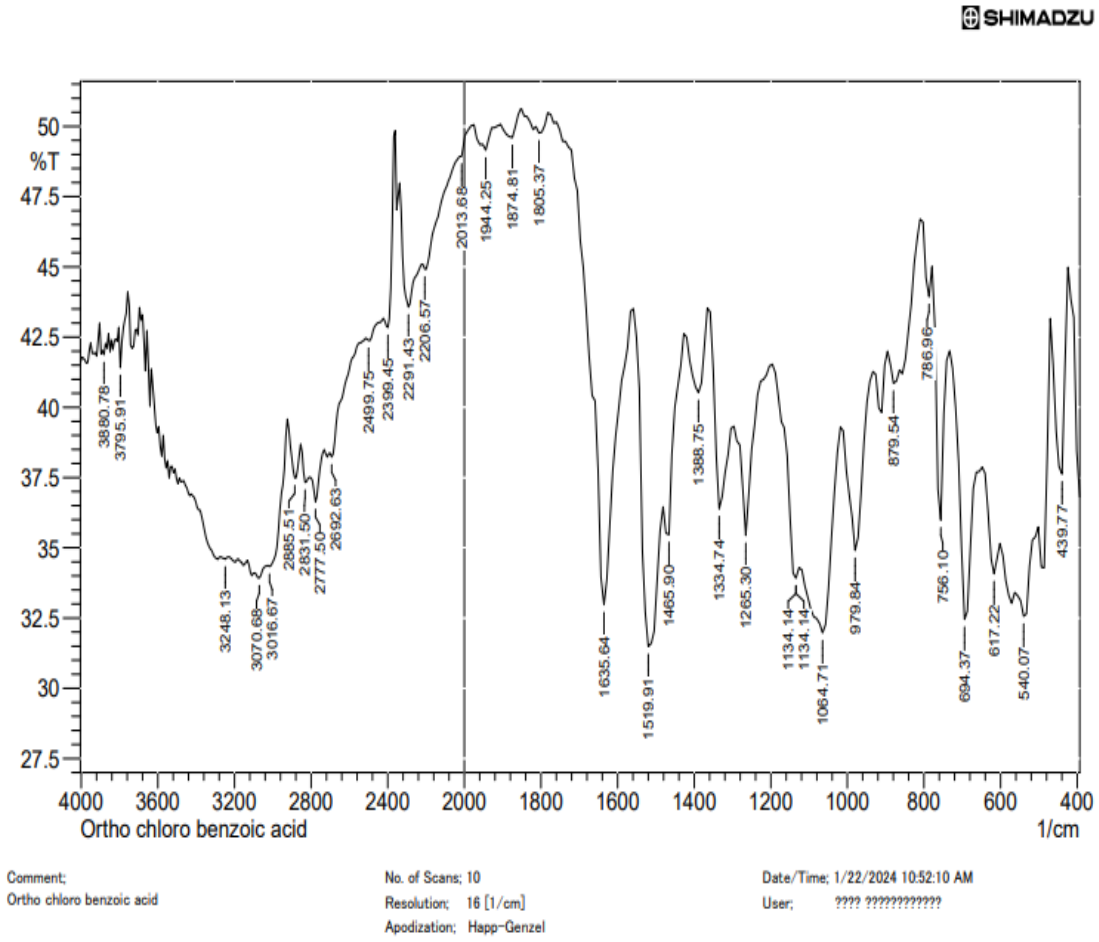
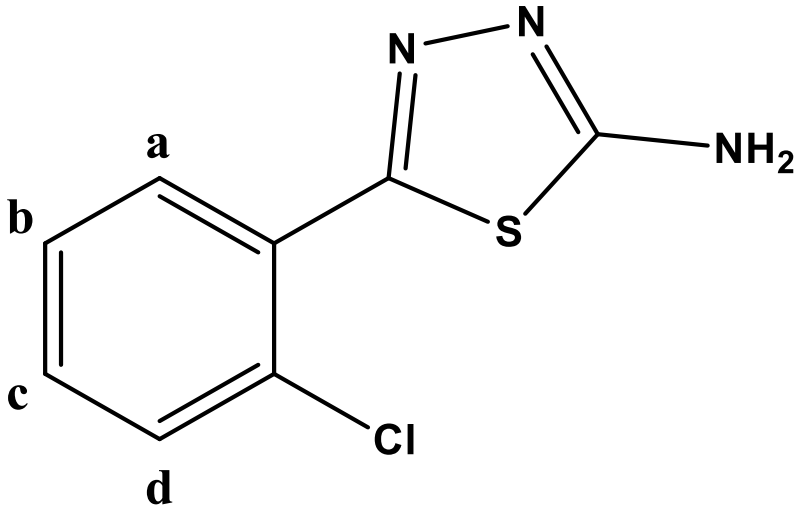


Fig .3. FT-IR Spectrum (stretching vibrations) of 2-amino-5-(2-chloro phenyl)-1,3,4-thiadiaozle.

3.3.2. ^{1}H-NMR Spectroscopy 2-amino-5-(2-chloro phenyl)-1,3,4-thiadiaozle



8.32 ppm associated with the (-NH_2) group (S, 2H), the aryl ring showed a vibration at 8.12 ppm associated with the proton (a) as in (d, 1H), (b) exbits a signil at 8.11 ppm as in (t, 1H), (c) a vibration at 8.06 associated with (t,1H) and (d) at 8.05 (d, 1H). Figure (4) shows the nuclear resonance spectrum of the compound 2-amino-5-(2-chloro phenyl)-1,3,4-thiadiaozle.

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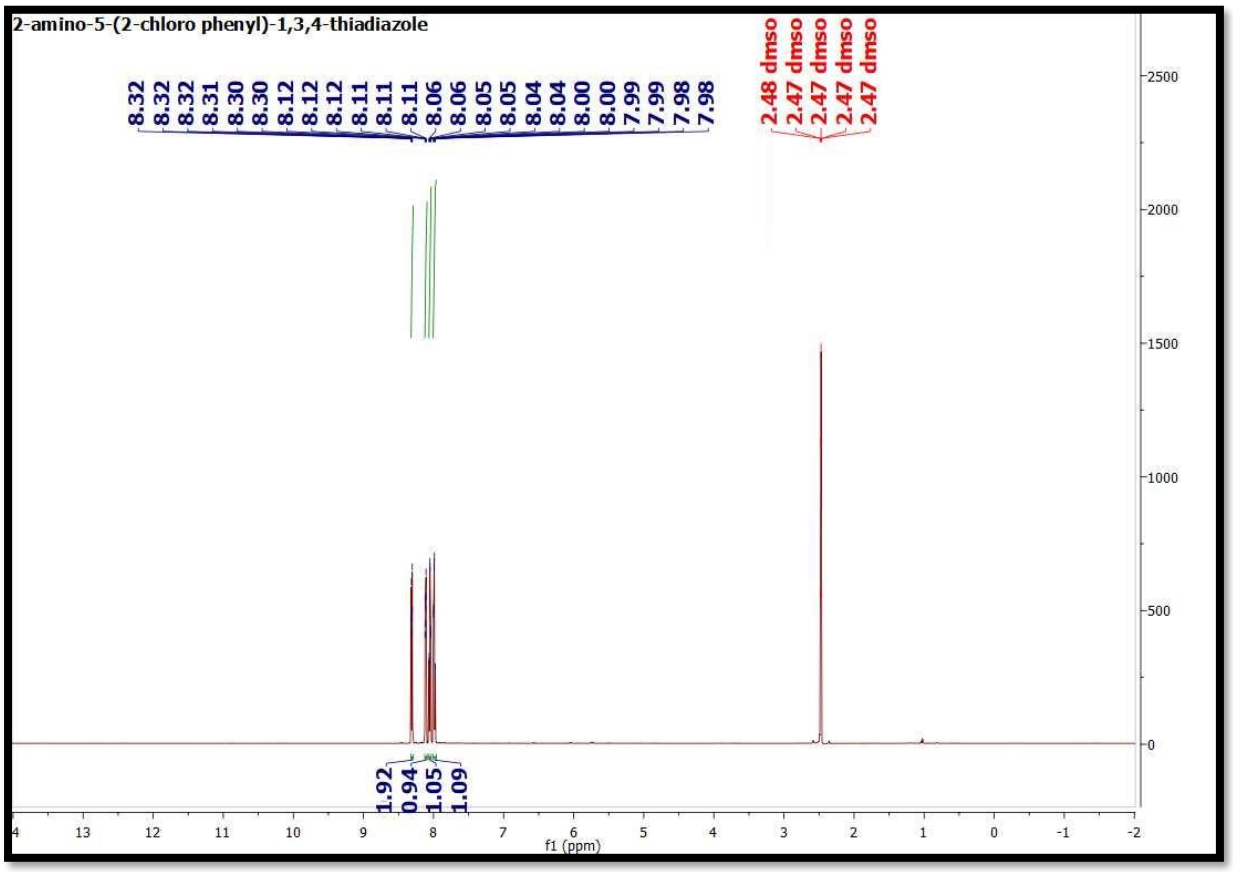


Fig .4. ^{1}H-NMR Spectrum of 2-amino-5-(2-chloro phenyl)-1,3,4-thiadiaozle.

3.3.3 FTIR of Spectrum of poly-OCTMAC

prepared polymer was identified by infrared spectroscopy, where the absorption bands (2700-3400) cm^{-1} were observed, which belong to the (O-H) range, while the (N-H) group showed absorption at (3240-3124) cm^{-1}, while the (aromatic C-H) group showed absorption at 3086 cm^{-1}, while the (aliphatic C-H) group showed absorption at (2931-2947) cm^{-1}, while the (C=O acid) group showed a broad absorption at 1728 cm^{-1}, while the (C=O amide) group was overllaped with the carbonyl of in carboxylic group, also the (C=N) group was overllaped with the carbonyl in acid group. Figure (5) shows the Infrared spectrum (stretching vibrations) of the prepared polymer.

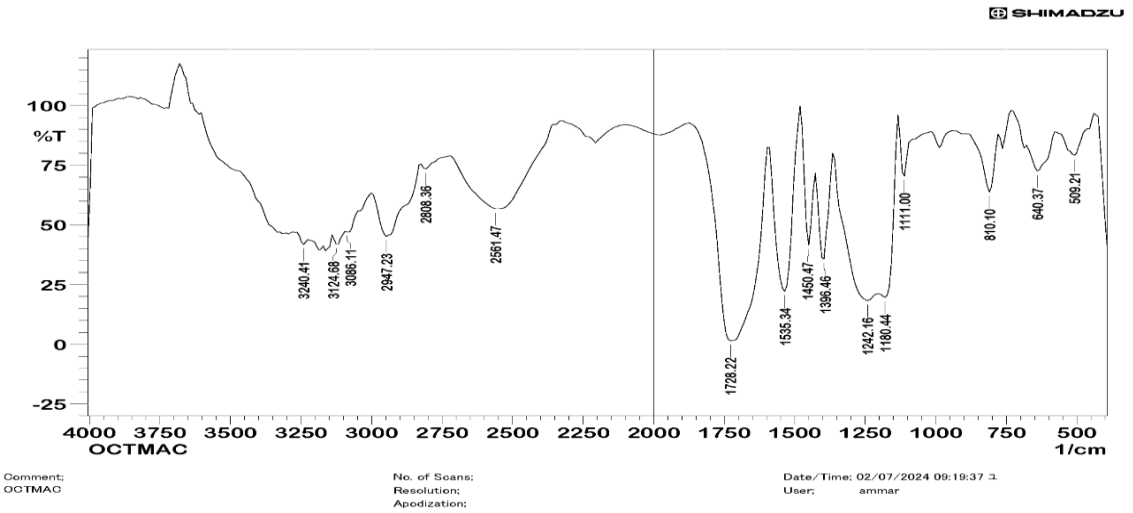


Fig .5. FT-IR Spectrum (stretching vibrations) of poly-OCTMAC.

3.3.3. TGA analysis

thermal decomposition spectrum of the polymer (poly-OCTMAC) shows the first weight reduction point due to the remaining volatile materials on the polymer chain at a temperature of 100-200 °C with a reduction rate of 35% of the total weight, the second from 200-300 °C with a reduction rate of 25% of the remaining weight, and the third from 300-400 °C with a reduction rate of 20% of the remaining weight, so that the remaining weight stabilizes at this rate until a temperature of 800 °C as shown in Fig (6).

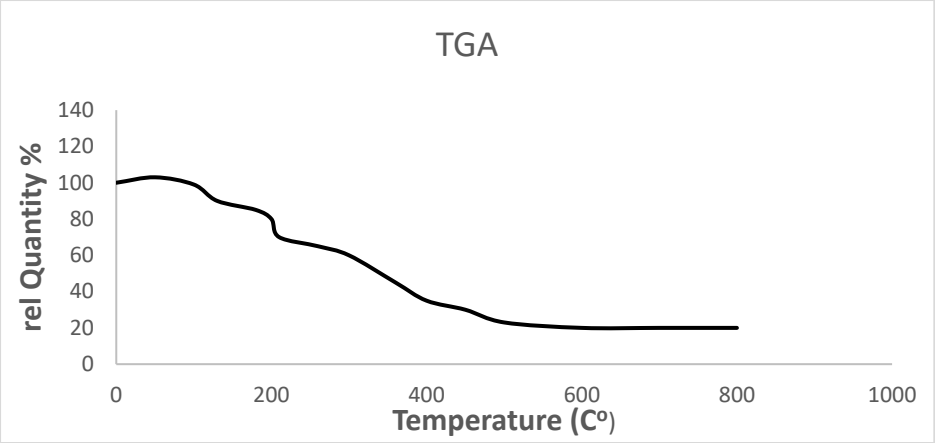


Fig .6. TGA of poly-OCTMAC.

3.3.4 XRD analysis

XRD patterns of thiadiazole and poly-OCTMAC hydrogel are described in the 2θ range of 5°–90° Fig (7). thiadiazole spectra showed fundamental diffraction peaks at 19.5°, 26.2°, 30.4°, 34.5°, 48.1°, and 52.3° corresponded to (111), (220), (311), (400), (511), and (440), respectively (Parthiban et al., 2020), ,which demonstrated the crystalline characteristic of thiadiazole. The grafting of thiadiazole on polymer showed major transformation in the XRD pattern of poly-OCTMAC hydrogel. The hydroxyl groups of acrylic acid were affected by thiadiazole, which destroyed the crystalline structure and transformed it to amorphous.

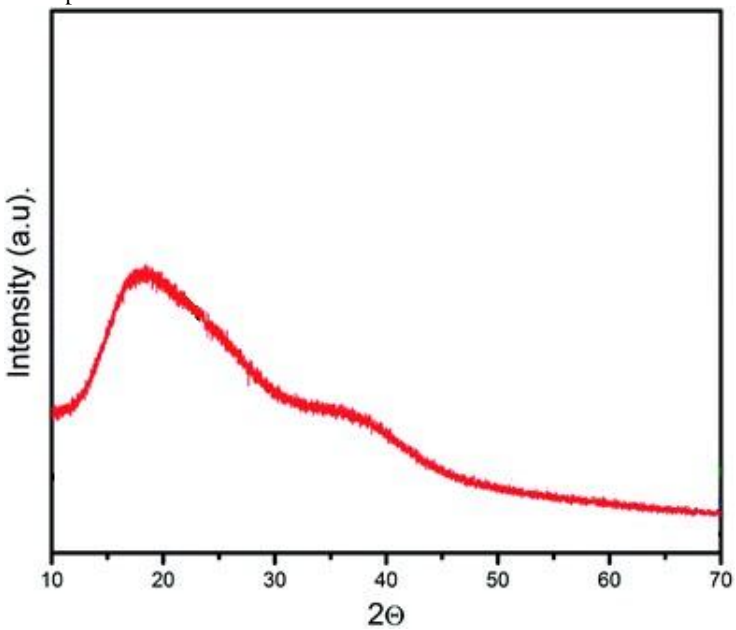


Fig. 7. XRD of the poly-OCTMAC

3.3.5 SEM analysis

The morphology study of the synthesis polymer was conducted by SEM technique to know more about the surface of the polymer Figure (8). The pores on the surface are visible and the presence these pores indicates the possibility of trans potation and retention of water

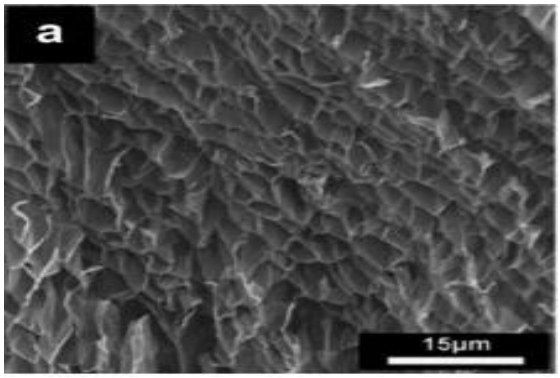


Fig.8. SEM morphology of poly-OCTMAC.

3.3.6 5-(2-chloro phenyl)-2-amino-1,3,4-thiadiazole retention

Identification of 1,3,4-thiadiazole complex loaded on polymer using UV-visible technique. A buffer solution of pH = 8 was prepared, then 0.1 g of the polymer was weighed. This amount was added to 20 ml of the buffer solution and left for an hour. After that, the solution was filtered and the filtrate was placed in a UV-visible spectrometer, where the maximum absorption of the decomposed thiadiazole compounds was measured. This indicates that these compounds are loaded on the polymer. Fig (8) shows IR short for the retained 5-(2-chloro phenyl)-2 amino-1,3.4-thiadiazole

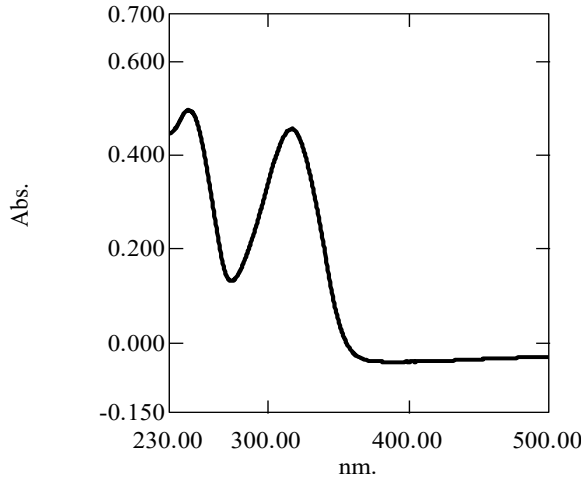


Fig. 9. UV-Vis of of poly-OCTMAC.

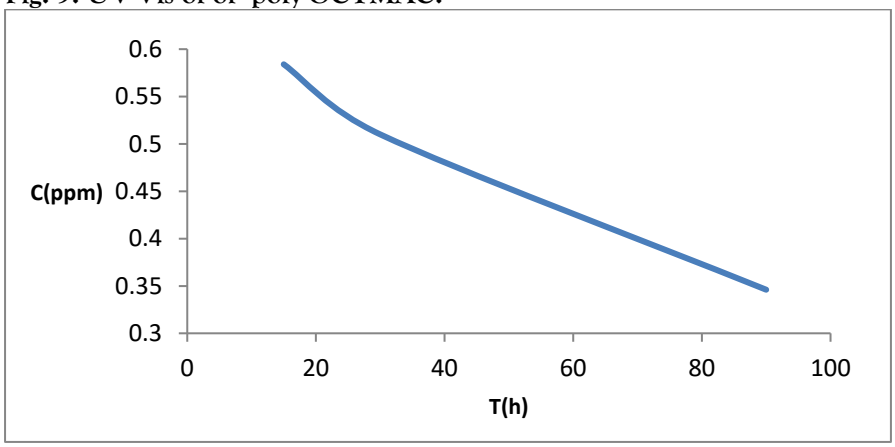
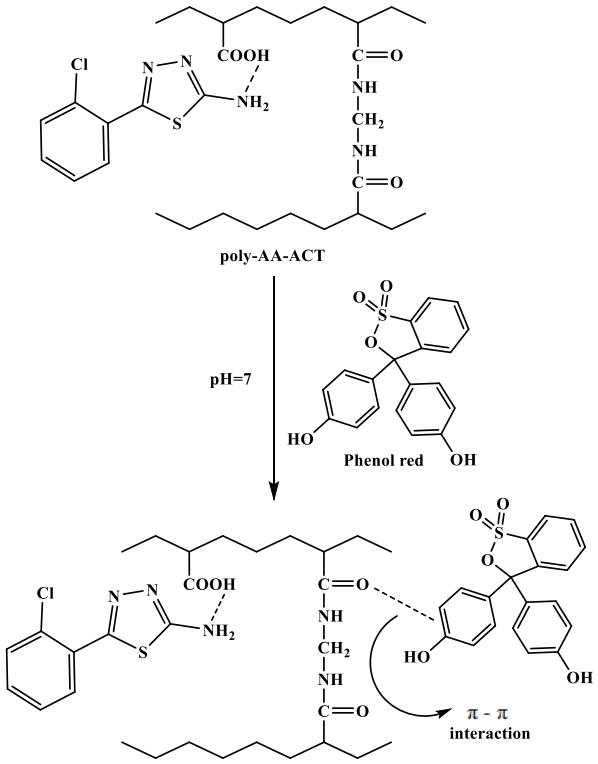


Fig.10. Adsorption of Phenol Red dye with poly-OCTMAC.

3.4.1. Comparison of poly-OCTMAC hydrogel performance with other adsorbents

Comparison of poly-OCTMAC hydrogel performance with other adsorbents demonstrated that the synthesized poly-OCTMAC hydrogel had a comparatively very high adsorption capacity than that of previously reported adsorbents. As natural toxic free inexpensive polymer, the occurrence of acryilc acid , MBA forms biodegradable and ecofriendly hydrogel, offers significant functional group (-NH, -OH)

units onto the surface of hydrogel, and make efficient PhR adsorption. Moreover, simple synthesis, ecological friendliness, high adsorption.



3.5. Water-retention study of polymer

Water retention analysis was performed using polymer in soil collected from Baladrooz district in Diyala province. 20 g of soil and 2 g of polymer were taken in a plastic container and 30 ml of water were added gradually. The weight (W1) was determined using a weighing machine. The container was weighed every day (W2) and kept at room temperature so that there was no stability in weight loss. The percentage of water loss (W%) for soil samples was calculated using the standard formula. Figure (11) shows water retention using soil and Figure (12) shows water retention using the polymer poly-OCTMAC.

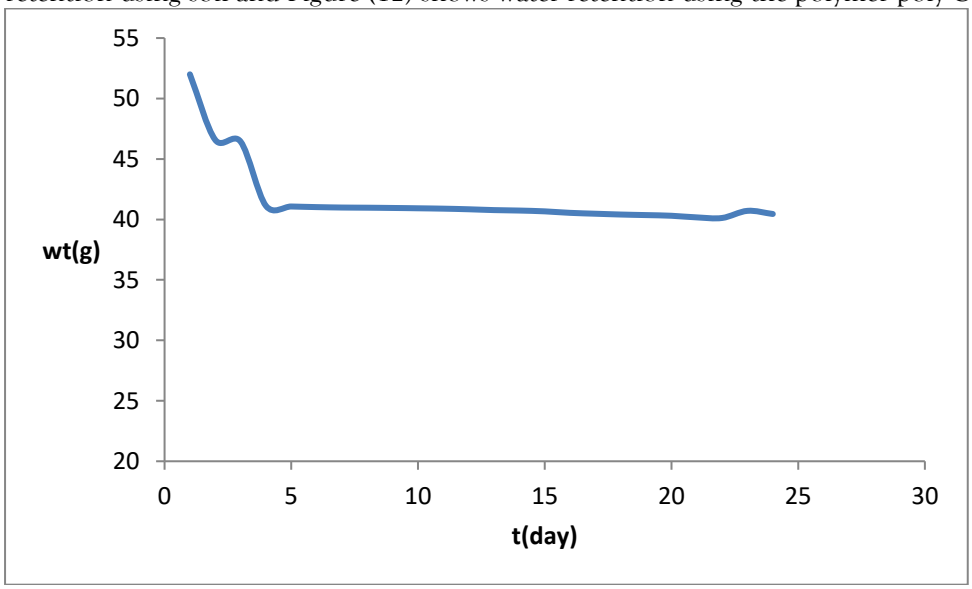


Fig.10. water retention of soil.

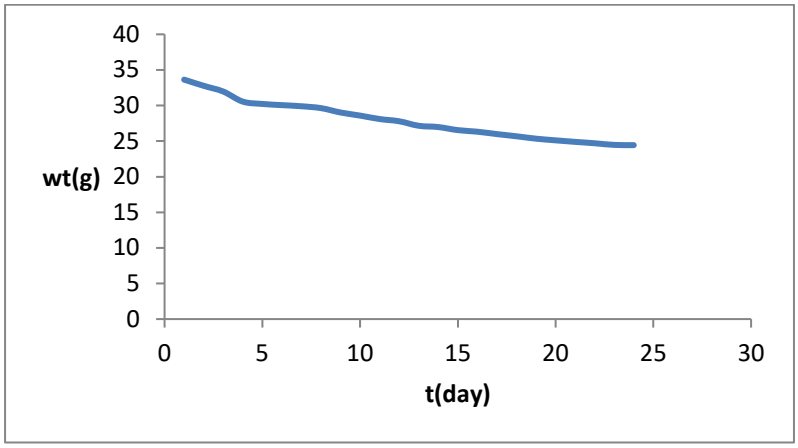


Fig.12. water retention of poly-OCTMAC-soil.

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