






## SYNTHESIS, CHARACTERIZATION AND APPLICATION OF NEW CROSS-LINKED AND MODIFIED POTASSIUM POLYACRYLATE AS SOIL AMENDMENTS

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### ABSTRACT

This study was carried to investigated Two types of new grafted polymers were prepared through chemical blending (mixture of the crosslinked mixture polymerization) of potassium polyacrylate (KPA) and 2-amino acetic acid (glycine) with calcium nitrate to give graft polymer type-1 (KPA-g-CaGY). As well as reaction of potassium polyacrylate (KPA) with calcium nitrate and chitosan polymer to give graft polymer type-2 (KPA-g-CaCS), grafting polymers has been characterized by FT-IR, <sup>1</sup>HNMR spectroscopy and compared with the pure potassium polyacrylate. The prepared polymers were used after confirming their diagnosis and physic-chemical properties in trying to reclaiming the desertification and saline soil in (Abu Ghraib-Baghdad in July 2023) by planting Iraqi sorghum type-70. Physical properties were test (before/after planting) by XRD of (KPA-g-CaGY) showed cross linked as crystal type-1, while KPA-g-CaCS showed cross linked as amorphous type-2 compared with KPA. In addition (SEM) Scanning Electron Microscopy of the modified polymer gave clear micrographs of the type-1 (KPA-g-CaGY) and type-2 (KPA-g-CaCS), this reinforces our claim that the polymer remains in the land after planting, after more than four months, and does not change or degradation, and enhances the use of the land for more than one season without adding the polymer to it. In addition to the equilibrium swelling percentage has been investigated in media (well water pH=8.5 and tap water pH=6.9), KPA-g-CaCS shows higher uptake of water compared with KPA and PAK-g-CaGY, suggests more hydrophilicity absorption at different times.

**Key words:** 2-amino Acetic acid, Grafting Polymer, SEM, Swelling, XRD.



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### INTRODUCTION

In recent decades polymers have been used to reclaim the agricultural filed (Tadayonnejad, *et al.*, 2017; Jorge, *et al.*, 2015; Al-Obaidi, *et al.*, 2006; Pavani, *et al.*, 2023; Alwazzan, 2024). Attention should be paid to using cheap polymers that are beneficial to plants and soil and have no side effects in the future; these polymers are rapidly manufactured using natural polymers and cross-linked monomers

(Asmaa, *et al.*, 2020; Pooley, *et al.*, 2010; Elena, *et al.*, 2022; Haradhan, *et al.*, 2023). Regarding safety and environmental impacts, the use of biopolymers for preparation provides more benefits than synthetic polymers (Maghchiche, *et al.*, 2010; Sojka, *et al.*, 1998; Shigeru, *et al.*, 2017). Potassium polyacrylate (KPA) was an industrial polymer, cross-linking, and super absorbent anionic granular, it has ability to absorb large amounts

of water (Cristiane, *et. al.*, 2012; Kalaleh, *et. al.*, 2015; Wasan, *et. al.*, 2022). Chitosan (CS) was a natural cross-linked polymer extraction from shells of shrimp and large marine fish (Renan, *et. al.*, 2015; Yélézouomin, *et. al.*, 2021; Daniel, *et. al.*, 2023). Polymer modification is required to provide special properties to the modified material, such as increased thermal stability, multiphase and physical responses, flexibility, compatibility and rigidity (Erizal *et. al.*, 2017; Bhatt. *et. al.*, 2023). Through modification, an insoluble polymer becomes soluble, and vice versa (Dong Liang *et. al.*, 2018). The most common polymer modification methods are grafting, cross linking, blending, and composite formations, it isn't essential that the polymers obtained to date from various sources have all of the attractive characteristics however, the formulation scientists seeking particular characteristics in polymers may find them lacking in the polymers available (Kasongo, 2019; Mahon, *et. al.*, 2020,). Many research laboratories have made substantial progress in chemical modification by presenting innovative reactive functional groups in the chemical structure of polymers, like -SH, -OH, NH<sub>2</sub>, X and COOH groups, which indicate possible sites for chemical modification or grafting (Yang, *et. al.*, 2022, Zhang, *et. al.*, 2020; Peng-Xiang, *et. al.*, 2024). Graft polymerization is one of the most appealing and convincing methods, in which one or more polymer side chains are chemically attached to the main polymer chain via covalent bonds to alter the rheological properties, hydrophilic ability, polymer charges, molecular chain, aggregation state, and complexation capability of the parent polymer (Won Seok, *et. al.*, 2022; Liu, *et.al.*, 2022; Kianpour, *et. al.*, 2024). *Sorghum bicolor* L. from the graminicide is one of the most important food and fodder grain crops grown in most regions of the world, five most important cereal crops in the world and is ranked fifth after wheat, rice, maize and barley, it is also considered one of the cash crops that occupies an important position in international trade and is also used in the production of bio-fuels in recent times (Alluhibi and Shamsullah, 2022).

In addition to its morphological characteristics that made it distinct from other field crops, such as its ability to tolerate drought and salinity and obtain several weeds from it when used as green fodder Sorghum is a drought resistance fodder crop, Under Iraqi condition such as environmental factors, including soil salinity, fertility, soil minerals, and water stress (Khubeab, *et.al.*, 2022). In this research two modified polymers were prepared type-1 (KPA-g-CaGY) and type-2 (KPA-g-CaCS) and used to study the reclamation of saline soil, the physico-chemical properties and processing under field experimental conditions, beginning in (Abu Ghraib-Baghdad in July 2023 and finished in October 2023).

## MATERIALS AND METHODES

**Studied traits:** All chemicals and their suppliers were used as received without further purification, Potassium poly acrylate (Hangzhou), 2-amino acetic acid, chitosan (Mwt 375,000 China), ethanol (Riedal-Dehaen 99%). FT-IR Spectroscopy-Shimadzu in college of Science, University of Baghdad. <sup>1</sup>H-NMR spectra (Bruker 400 MHz). XRD Philips PW1730- and SEM China were tested in Iran.

**Synthesis of grafting polymer (KPA-g-CaGY):** according to the methods (Zainab, *et. al.*, 2024) (Scheme1).

**Synthesis of Grafting Polymer (KPA-G-CACS):** according to the methods (Zainab, *et. al.*, 2024) (Scheme1).

**Method of Application of Polymers (Zohuriaan-Mehr, *et. al.*, 2008):** The application technique also affects how well the polymer develops in the plant. As there are elements that must be taken into consideration, including the type of polymer (gel), particle size, plant type and application rate.

**Measurement of Swelling Tea-Bag Method (El-Sayed, *et. al.*, 2011 and Ceylan, *et.al.*, 2006):** It is a traditional, fast and suitable method for testing the absorption capacity of samples weighing between 0.1-0.2 gm of (KPA-g-CaGY and KPA-g-CaCS). The sample whose absorption capacity is to be measured is placed in an acrylic/polyester fabric bag with mesh dimensions. Small and immersed in an excess amount of water or salt solutions until it

reaches equilibrium, and then the bag is lifted and hung until it stops dripping, and the water absorption is calculated according to the equation: Where  $M_0$  and  $M_t$  are the dry and wet sample masses, respectively.

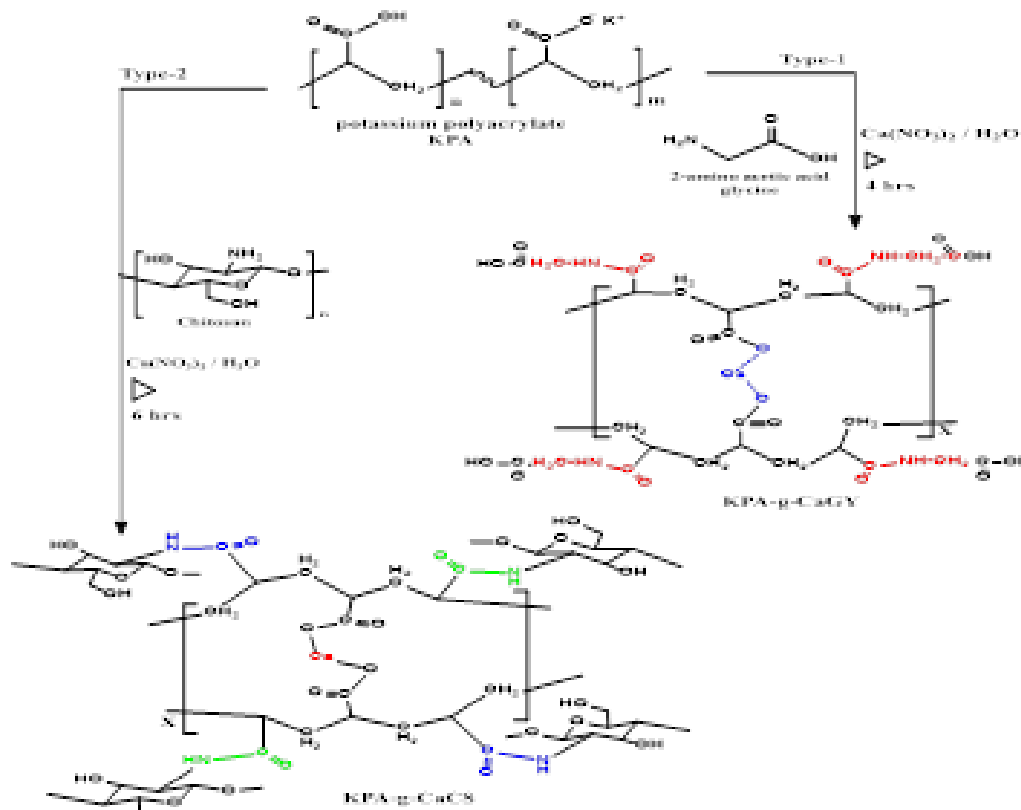
$$\% \Delta M = \frac{M_t - M_0}{M_0} \times 100$$

**Water Retention (Zohuriaan-Mehr and Kabiri, 2008):** according to the (Zohuriaan-Mehr and Kabiri, 2008) methods. Where  $W_{t1}$  and  $W_{t2}$  are the dry and wet sample masses, respectively.

$$\% (\Delta W) = \frac{W_{t2}}{W_{t1}} \times 100$$

**Agricultural Application on Samples of Sorghum Plants (El-Sayed, *et. al.*, 2011 and Ceylan, *et. al.*, 2006):** We also tested the

effectiveness of the effect of adding superabsorbent polymeric materials prepared as a land and soil amendment on sorghum samples. The composite material was added to the soil at a rate of 0.1% by weight. The beneficial and positive effect of the compound material on the growth of sorghum plants sixteen weeks after the start of planting. Sample groups 1 to 4 were planted by adding the composite material to the soil, while samples 0 were left as control. It can be clearly seen that adding the composite material had a qualitative effect on plant growth.



**Scheme 1. Suggest Synthesis Route of Grafting Polymer (KPA-g-CaGY) and (KPA-g-CaCS)**

**Table 1. Some Chemical Properties of Water Used in Irrigation**

Classify water	SAR	SO <sub>4</sub> <sup>2-</sup>	HCO <sub>3</sub> <sup>-</sup>	Cl <sup>-</sup>	Na <sup>+</sup>	K <sup>+</sup>	Mg <sup>2+</sup>	Ca <sup>2+</sup>	EC	pH
C4S1	(mmol L <sup>-1</sup> ) <sup>0.5</sup>				mmol L <sup>-1</sup>				dS m <sup>-1</sup>	-
	7.093	6.194	0.481	11.18	11.86	0.281	2.204	3.384	2.500	7.46

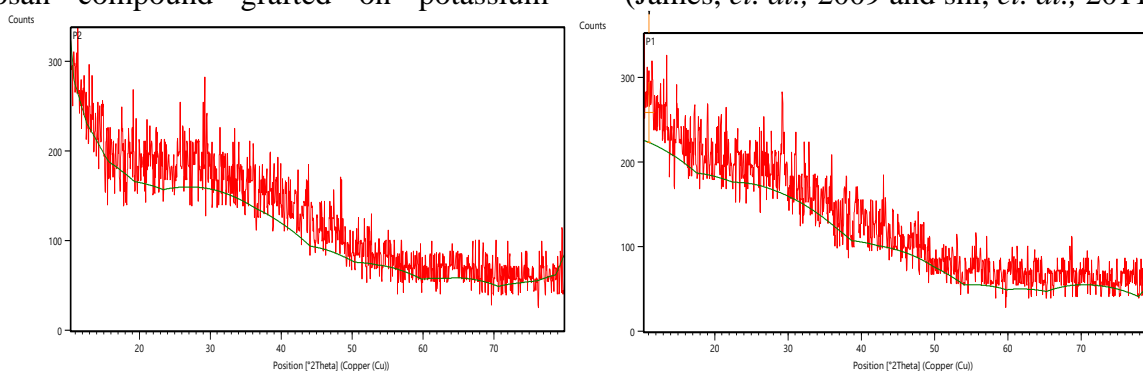
## RESULTS AND DISCUSSION

Polymers are the basic foundation for most of the basics of life that are necessary to keep pace with development and progress, there is hardly anything in our lives that is devoid of a

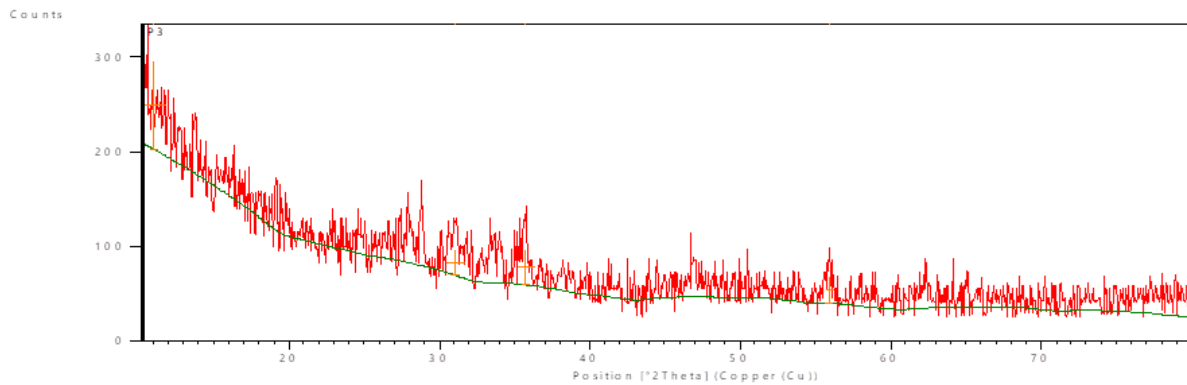
polymeric part in it, starting from the structure of the human body, which contains blood, lipid, starch, giant molecules loaded on it, foods, medicines, and tools used, to the simplest things in our lives (Mahon, *et.al.*,

2020). Pure potassium polyacrylate (KPA) was used in an attempt to reclaim saline soil in the (in July 2023-Abu Ghraib-Baghdad), compared for two types of modified grafted polymers using potassium polyacrylate and glycine with calcium nitrate (KPA-g-CaGY) and once using potassium polyacrylate and chitosan with calcium (KPA-g-CaCS). Initial diagnostic FT-IR measurements confirmed the formation of grafted polymer (KPA-g-CaGY) in (KBr):  $\nu/\text{cm}^{-1}$ , 3479-2513 of (OH), 3296 (NH), 2980, 2820 of (CH aliph.), 1717 (C=O carboxylic group) and 1668 of (C=O amide group). In addition, FT-IR (KBr):  $\nu/\text{cm}^{-1}$  of compound (PAK-g-CaCS) 3438-2563 of (OH), 3238 (NH), 2927, 2802 (CH aliph.), 1639 (C=O) shifts of carboxylic group, and 1625 of amid group (C=O) (William, *et. al.*, 2006 and Silverstein, *et.al.*, 1991). Likewise,  $^1\text{H}$ NMR analysis of the compound showed the presence of the glycine compound grafted on potassium polyacrylate showed  $^1\text{H}$ NMR (DMSO- $d_6$ ) of 25H of (KPA-g-CaGY):  $\delta$  2.98 (d, 6H,  $\text{CH}_2$  of methyl group), 4.59 (d, 12H,  $\text{CH}_2$  of methylene group), 1.21 (d, 3H, CH), 9.03 (s, 1H, 2NH), 12.5 (s, 2H, OH). As well as  $^1\text{H}$ NMR (DMSO- $d_6$ ) of 25H of (KPA-g-CaCS) the heterogeneous ring belonging to the chitosan compound grafted on potassium

polyacrylate appeared in  $\delta$  2.42 (d, 6H,  $\text{CH}_3$  of methyl group), 2,55 (d, 12H,  $\text{CH}_2$  of methylene group), 3.81 (d, 3H, CH methane group), 7.14 (d, 3H of tetrahydropyran), 8.07 (s, 1H, 2NH), 7.74 (s, 2H, OH) (Gorbunova *et.al.*, 2012). XRD technology was used to determine whether the polymer was a cross-linked or amorphous polymer. As a result, we used this technique to elucidate the types of grafted polymers -1 and -2. The width of the separate peaks of the grafted polymer (KPA-g-CaGY) in figure (1) is shown at measurement at  $25^\circ\text{C}$  [ $2\theta$ -11.0215]. This indicates that the compounds produced are not regular, which indicates that they are amorphous or not crystalline. While we observe that the polymer (PAK-g-CaCS) measured before adding it to the soil (before planting) has the same composition after planting with a slight change in the creep of the measured amorphous peaks, indicating enhanced stability, it shows noticeable crystalline figure (2) Measurement at  $25^\circ\text{C}$  [ $2\theta$  11.0399 and 55.9253]. This reinforces our claim that the polymer remains in the land after planting, after more than four months, and does not change or degradation, and enhances the use of the land for more than one season without adding the polymer to it (James, *et. al.*, 2009 and shi, *et. al.*, 2011).



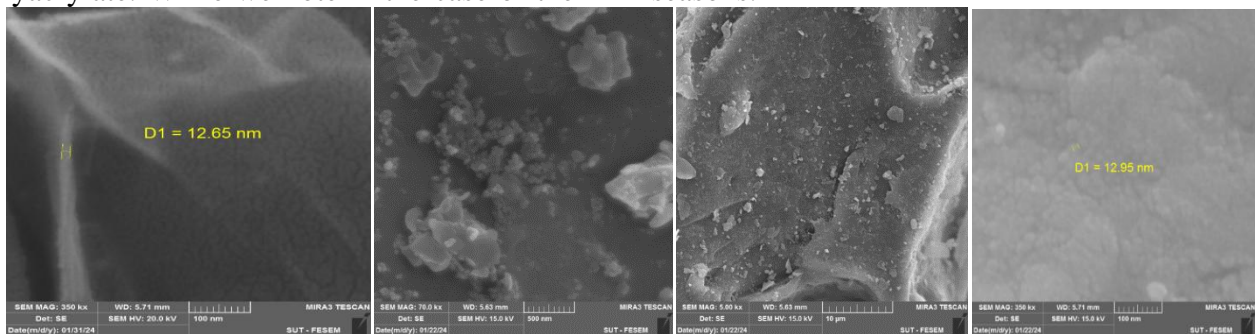
**Figure 1. XRD- of Modified polymers (a) type-1 KPA-g-CaGY, (b) type-2 KPA-g-CaCS before Planting**



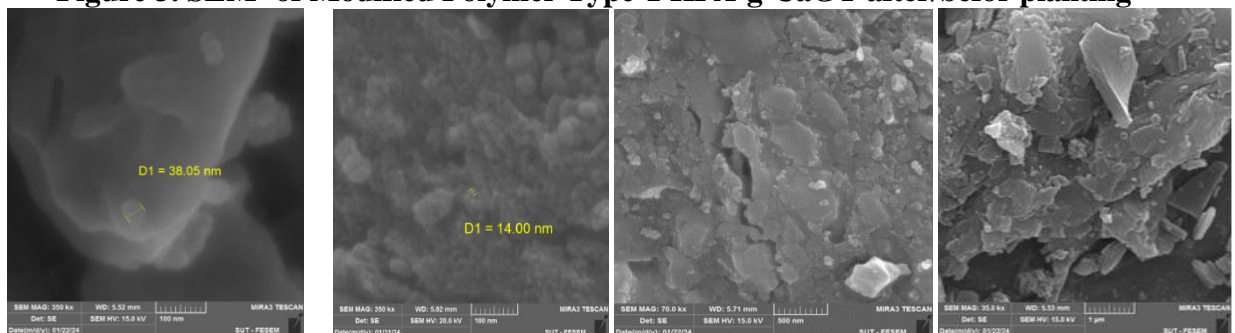
**Figure 2. XRD- of Modified polymer of type-2 KPA-g-CaGY After Planting**

EM measurement (Khubeab, *et. al.*, 2022) gave clear micrographs of the type-1 and type-2, where the powder morphology was examined using SEM technique. Figure (3) clearly shows that the original backbone of KPA formed needle-like crystalline structures, as SEM images of the surface of the type-1 graft polymer (KPA-g-CaGY) molecules appear in Figure (4). The microscopic image of the cross section (KPA) after achieving equilibrium at magnification form 12X is a highly porous structure with sufficiently large pores and the pore size distribution may be due to the amino acid loaded on the potassium polyacrylate. While we note in the case of the

type-2 grafted polymer (KPA-g-CaCS) before planting, it shows the presence of granular structures belonging to the cross-linked polyacrylates embedded in the chitosan particles (Noor, *et. al.*, 2015; Benito, *et. al.*, 2023). We find that it appears in the polymer after cultivation. The wide pore structure has a sponge-like shape and is interconnected in a cross-linked fabric. This indicates the presence of cross-linking in the grafted polymer structure. This indicates that the polymer remained in the ground for a period exceeding four months, which opens the door to using the land without adding polymer for other new seasons.



**Figure 3. SEM- of Modified Polymer Type-1 KPA-g-CaGY after/befor planting**



**Figure 4. SEM – of Modified Polymer Type-2 KPA-g-CaCS after/befor Planting**

The large macropore size of KPA-g-CaCS causes water to easily enter the polymer structure so that the resulting swelling ratio is

very large (Zohuriaan-Mehr and Kabiri, 2008; Hassan, *et. al.*, 2023). Swelling behavior of modified graft polymers, two experiments

were conducted to measure the swelling percentage of the two types of modified polymers in the laboratory measurement temperature 25°C, using tap water pH=6.9 once and using well water again pH=8.5, as it is used in watering the white sorghum plant that is the subject of the research. It was noted that the degree of swelling of the first grafted polymer (KPA-g-CaGY) was lower than that of the second grafted polymer (KPA-g-CaCS) under the same experimental conditions (in tap water pH=6.9). When compared with the same polymer using (well water pH=8.5), it was noted that the (KPA-g-CaCS) grafted polymer had a higher percentage of retaining water for the longest possible period table 2. Which suggests that the second type of polymer grafted with the chitosan polymer is better than the first one due to the presence of the chitosan ring, which is characterized by retaining water for long periods (loading chitosan on modified potassium polyacrylate with calcium is better compared to loading glycine on modified potassium polyacrylate with calcium). The ionic strength of a salt solution is related to both the concentration and charge of each individual ion, in addition,

poly charged positive ions (+2 or carboxylates), which leads to an increase in the degree of cross-linking of carboxamide inside the polymer through the formation of a complex with groups, we can deduce the effects of the charge and concentration of the ion the ion network, and as a result, the swelling decreases. DSC measuring the thermal decomposition of the prepared polymers, it was observed that the graft polymer type-2 was superior to the degree of thermal decomposition starting from 200 to 255°C in the first stage, in the second stage from 400-507°C, and in the last stage at 813°C. This may be attributed to the presence of the chitosan ring in the polymer structure figure 5. As for the type-1 graft polymer, it was observed that it loses 35°C in the first stage, then reaches 209°C in the second stage, and disintegrates in the last stage at a degree of 411°C due to the presence of the amino acid glycine figure 5. The difference in disintegration above is attributed to the type of chemical substance grafted onto two types of the graft polymers (Zohuriaan-Mehr, *et. al.*, 2008 and Hassan *et. al.*, 2023).

**Table 2. Swelling Ratio of Graft Polymer (KPA-g-CaGY and KPA-g-CaCS) in well water pH=8.5 and Tap Water pH= 6.9**

Time/ Min.	Well Water pH = 8.5 Swelling %		Tap Water pH = 6.9 Swelling %	
	KPA-g-CaGY	KPA-g-CaCS)	KPA-g-CaGY	KPA-g-CaCS)
60	15	20	30	35
90	25	28	40	55
120	39	33	55	67
150	58	46	69	75
180	65	59	70	80
210	70	66	85	84
240	85	75	93	90
270	88	80	93	95
300	88	84	93	96
600	90	89	93	97
900	90	92	93	97
1200	90	92	93	97
1500	90	92	90	97
1800	90	92	90	97

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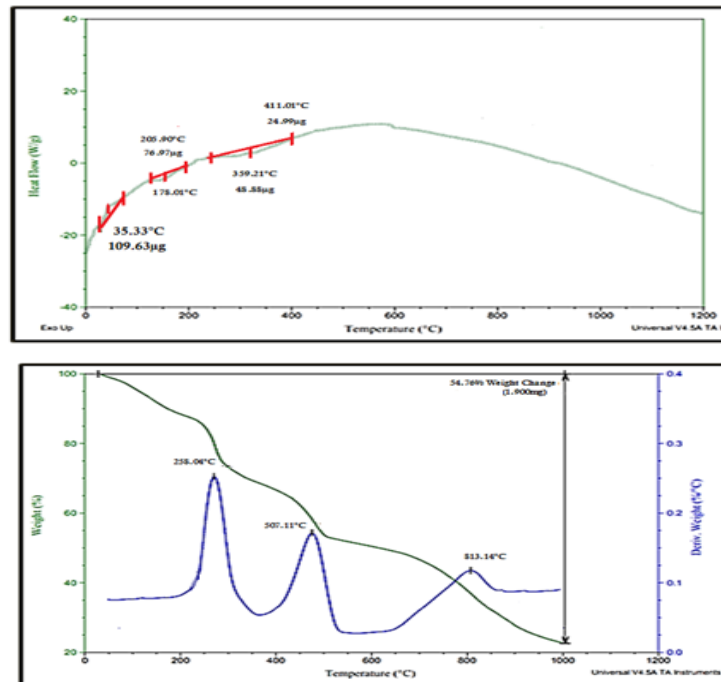


Figure 5. DSC of graft polymer type-1 and type-2

## CONCLUSION

The synthesized grafted polymers demonstrated significant potential as soil-conditioning materials for improving the physical properties of saline and decertified soils. The incorporation of glycine and chitosan into the potassium polyacrylate structure enhanced the cross-linking efficiency and water retention ability of the modified polymers. Among the prepared materials, KPA-g-CaCS showed superior swelling behavior, indicating its greater suitability for increasing soil moisture availability under different pH conditions. The long-term stability of the polymers in soil without noticeable degradation suggests their effectiveness for sustained agricultural use across multiple growing seasons. Therefore, the application of these grafted polymers may represent a promising and cost-effective strategy for soil reclamation and improving crop productivity in arid and semi-arid regions.

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## CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

## AUTHOR/S DECLARATION

We confirm that all Figures and Tables in the manuscript are original to us. Additionally, any Figures and images that do not belong to us have been incorporated with the required permissions for re-publication, which are included with the manuscript.

-Author/s signature on Ethical Approval Statement.

-Ethical Clearance and Animal welfare

-Funds:

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## تحضير وتشخيص وتطبيق بوتاسيوم بولي أكريليت المحور الجديد المترابط كمصلحات للتربة

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### المستخلص

اجريت هذه الدراسة للتحقق من تحضير نوعين من البوليمرات المحورة باستخدام (طريقة مزج الخليط المتشابك) لبوتاسيوم بولي اكريليت (KPA) والحامض الاميني الكلايسين مع نترات الكالسيوم لتحضير النوع الاول (KPA-g-CaGY), وكذلك تم مفاعلة بوتاسيوم بولي اكريليت (KPA) مع بوليمر الكايتوسان ونترات البوتاسيوم لتحضير النوع الثاني (KPA-g-CaCS), مقارنة بالبوليمر النقي بوتاسيوم بولي اكريليت شخصت البوليمرات المحضرة بتقنية طيف الاشعة تحت الحمراء (FT-IR) والرنين النووي المغناطيسي (<sup>1</sup>HNMR). تم استخدام البوليمرات المحضرة بعد التأكد من تشخيصها وخواصها الفيزيائية والكيميائية في استصلاح تربة ملحية في (أبو غريب - بغداد - تموز 2023) بزراعة الذرة البيضاء صنف 70. تم اختبار الخواص الفيزيائية (قبل / بعد الزراعة) بواسطة حيود الاشعة السينية (XRD) حيث اعطت زوايا حادة واضحة اظهرت ارتباطا متقاطعا كنوع بلوري للبوليمر الاول (KPA-g-CaGY) ونوع متشابك للبوليمر الثاني (KPA-g-CaCS), بالإضافة الى ذلك اعطت صورة الماسح المجهرى (SEM) صورة واضحة لعمود التركيب بوتاسيوم بولي اكريليت (KPA-g-CaGY) للنوع الاول وصورة مجهرية واضحة بشكل حبيبات للبوليمر الثاني (KPA-g-CaCS). وهذا يدل بأن البوليمر يبقى في التربة بعد الزراعة لمدة أكثر من أربعة أشهر ولا يتغير أو يتحلل، ويعزز استخدام التربة لأكثر من موسم دون إضافة البوليمر إليها. بالإضافة إلى نسبة الانتفاخ التي تم قياسها في وسطين مختلفين الاول الاس الهيدروجيني لماء البئر = 8.5 ودرجة الاس الهيدروجيني لماء الحنفية = 6.9 اشارت الى ان نسبة احتفاظ البوليمر بالماء والوصول الى حالة التوازن كانت للنوع الثاني (KPA-g-CaC) افضل من البوليمر النوع الاول (KPA-g-CaGY) .

الكلمات المفتاحية: 2-امينو استك اسيد، البوليمرات المحورة، الماسح المجهرى، حيود الاشعة السينية، الانتفاخ.

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