Received: 7 July 2023 | Revised: 8 August 2023 | Accepted: 10 August 2023
DOI: 10.1002/pen.26474

RESEARCH ARTICLE

spe INSPIRING PLASTICS PROFESSIONALS POLYMER ENGINEERING AND SCIENCE WILEY

Synthesis, characterization, and swelling behavior of a *poly*(acrylamide-*co*-acrylic acid) copolymer: Application to water retention in two sandy agricultural soils in sub-Saharan region

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Funding information
Researchers Supporting Project, Grant/Award Number: RSP-2023R78

Abstract

The application of copolymer hydrogels based on acrylamide (AAm) and acrylic acid (AA) in sub-Saharan soils can increase the quantity of water retained by the soil and improve plant establishment and growth. The aim of this work was to synthesize and characterize a copolymer based on acrylamide and acrylic acid and to evaluate its swelling capacity with different physicochemical parameters. This study also aimed to investigate the effect of metallic cations on the swelling capacity of the copolymer and to assess the improvement in water retention in sandy soils. The copolymer material was prepared by free-radical polymerization in aqueous solution at a temperature of 60°C and characterized by Fourier transform infrared (FTIR) and x-ray diffraction (XRD), and its average molecular weight was estimated from intrinsic viscosity measurements. The swelling capacity was evaluated with different physicochemical parameters, and the effect of metallic cations on the swelling capacity was investigated. Water retention in two sandy soils was also evaluated. The copolymer material containing 50% AAm and 50% AA can retain more than 8100% of water. The swelling ratio increased with increasing temperature, with a maximum swelling ratio at 65°C. The presence of metallic cations reduced the maximum swelling ratio of the copolymer material as the cation concentration increased. The copolymer material at 1% in a composition with the soil showed an improvement in the water retention of 12.2% and 7.9% for soil A and 7.9% for soil B. Soil air volume is improved by 19% for both soils. This study shows that the copolymer material has a high swelling ratio and can improve water retention in sandy soils. The copolymer can be used in agriculture to protect and improve plant growth and yield.

Highlights

- Synthesis and characterization of a copolymer based on acrylamide and acrylic acid.
- Evaluating the swelling capacity with different physicochemical parameters.

- The application of this copolymer in sub-Saharan soils increases the quantity of water retained.
- The copolymer is prepared by polymerization in aqueous solution and characterized by FTIR and XRD.
- This study shows that the copolymer has a high swelling ratio and improves water retention.

KEYWORDS

hydrogel, *poly*(AAm-*co*-AA), soils, swelling, water retention

1 | INTRODUCTION

Hydrogels are networks of cross-linked polymers capable of absorbing and retaining large quantity of water. Their structures are heterogeneous in different orders of micromorphology.[1] Many methods are available to prepare hydrogel materials. These polymers are mainly made from water-soluble monomers by radical polymerization reactions using a suitable cross-linking agent. The most popular water-soluble monomers include acrylic acid, acrylamide (AAm), and their derivatives. These monomers can be used to synthesize cross-linked homopolymers and copolymers consisting of different comonomers by aqueous solution or other polymerization methods. These monomers can also be grafted onto other materials such as polyethylene terephthalate fiber and certain natural macromolecules to obtain hydrogel materials. Such hydrogels can be used in the controlled release of drugs,[2,3] in biomaterials,[4] and as sensors.[5] In addition to these applications in various fields, these hydrogels are of particular interest in the agriculture domain. These materials are used as agents for reducing water consumption by reducing the amount of irrigation.[6] They also protect plants against dehydration[7] and stabilize fertilizers in the soil by preventing the active ingredient from escaping to groundwater, leading to improved plant growth.[8]

Eco-friendly materials,[9,10] polymeric materials derived from renewable sources, reduce reliance on fossil fuels, bio-based materials, including semi-permeable membranes[11] and blend polymers.[12] Eco-friendly materials aim to use green renewable resources to protect the environment from negative effects.[13–15] Bio-based polymers are natural and are derived from renewable resources and often require less energy.[16] In recent years, and especially in the period of falling oil prices, many countries, particularly Algeria, have moved toward the development of agriculture as an alternative to the oil economy. In this context, Algeria has adopted a development policy for Saharan agriculture. Moreover, some wilayas of southern Algeria, such as the wilaya of El-oued or the wilaya of Biskra, have shown very satisfactory results in some sectors of agricultural production. As such, an international agricultural research center, Kapia Algeria, was inaugurated on December 4, 2011, in Algiers, at the National Institute of Agronomic Research of Algeria (INRAA). The mission of this center is to carry out three projects: increasing the productivity of wheat and barley, installing a multi-chapel greenhouse equipped with solar energy and intended for research and development of the so-called hydroponic above-ground crops, and establishment of a pilot demonstration unit for the industrial production of vegetable seeds.

However, the main problem faced in these regions is the unavailability of water. The latter is an essential factor, which limits agricultural production. Indeed, during the dry season, there is a shortage of water, while in the rainy season, these regions cannot benefit from water resources due to the lack of managerial skills. A suitable solution to this problem is to use products (polymers) that can retain water and release it in a controlled manner. Polyacrylamide (PAAm) hydrogels and their derivatives are the subject of numerous studies,[17,18] which have demonstrated their capacity of water absorption and biocompatibility with physiological body fluids. The application of PAAm hydrogels in the field of agro-chemistry has been studied.[19–22]

Water scarcity in agriculture is very common in many countries around the world. In some areas, it is almost impossible to cultivate without irrigation. Even for regions that receive large amounts of precipitation during the rainy season, crops often experience severe droughts during the dry seasons.

Today's agriculture aims to optimize the use of all farm resources and seeks to make more sustainable use of the resources available for water retention. Water retention, harvesting, and storage practices are very important in agriculture. Indeed, in agriculture, it is above all essential to work in such a way as to improve the retention of water on the plots and to promote its infiltration into the soil. Polymer hydrogels are used to aid plant establishment and improve plant growth in dry soils.[23–27]

They have the potential to absorb water many thousands of times their weight, retain it, and supply it to plant soils during water stress, increasing plant survival and growth.[28–32] The application of hydrogels in soils makes it possible to increase the quantity of water retained by the soil.[33–36] It also makes it possible to retain certain useful cations for plant growth.[37] Thus, the introduction of hydrogels into soils can improve the conditions for plant establishment and growth.[37,38]

The objective of this work concerns, in a first part, the synthesis of a copolymer based on AAm and acrylic acid (AA) and its analyses by various characterization techniques (Fourier transform infrared or FTIR, x-ray diffraction or XRD, and viscosity measurements). The second part will focus on the study of the swelling behavior of the copolymer material in an aqueous medium and the determination of the factors influencing its swelling (temperature, presence of metallic cations, etc.). The third part is devoted to an application relating to the study of water retention in two sandy loam soils in the presence of these copolymers.

2 | EXPERIMENTAL

2.1 | Materials and methods

Acrylamide (AAm; >99%, Sigma-Aldrich), acrylic acid (AA; >99%, Sigma-Aldrich), ammonium peroxodisulfate (APS; >98%, Merck), *N,N,N′,N′*-tetramethyl ethylene diamine (TEMED; >98%, Sigma-Aldrich), glutaraldehyde (GA; >98%, Sigma-Aldrich), and ethanol (purity >99%, Sigma-Aldrich) used in the synthesis of the copolymers were used without prior purification except for AAm and AA monomers, which were purified prior to use. The AAm monomer was recrystallized in hot ethanol. The resulting crystals were separated by filtration and then dried under vacuum at 30°C. The AA monomer was distilled under vacuum at a temperature of around 40°C to eliminate the hydroquinone monomethyl ether, used as a polymerization inhibitor.

In the perspective of a future application of our copolymer material in agriculture, our application is devoted to the study and evaluation of water retention in two sandy loam soils (A and B) of southern Algeria (the region of Ghardaïa). Table 1 gives the two forms and the analysis results of the studied soils.[39]

The FTIR transmission was recorded over the range 400–4000 cm^{-1} using Shimadzu FTIR-8400S spectrometer with a resolution of 2 cm^{-1}. The samples were thoroughly mixed with spectroscopic KBr in a proportion of 1/100, crushed, and compacted into pellets under a pressure of 2 kbar.

XRD spectra were recorded using Rigaku Miniflex 600 instrument, anticathode: Cu (K_α). The step size was 0.02°, while the speed scan was 4°/min. The XRD spectra were recorded as a function of 2θ between 10 and 70°.

The viscosity was measured in $NaNO_3$ solution connected to a thermostated water bath (JULABO) at (25.0 ± 0.1°C) using Ubbelohde viscometer (Schott-Geräte AVS type 531) having the following characteristics: 0.36 mm capillary diameter, 10 mL bulb volume, 7.7 cm capillary length, and 838 s efflux time for water alone. The synthesized non-cross-linked copolymer was dissolved in $NaNO_3$ (1 M) solution, and the viscosity was measured for four concentrations.

The pH of solutions was measured with a pH-meter (type Panpeha) combined with glass electrode (Schott), preliminarily calibrated with two buffer solutions.

2.2 | Synthesis of copolymers

Free-radical copolymerization was carried out with water according to the procedure cited in the literature.[30] In a three-necked flask containing 90 mL of distilled water, a quantity of AAm and AA, in four different compositions, was poured. After complete dissolution of the monomers, a quantity of TEMED, as an accelerator, was transferred to a three-necked flask, which is equipped with a stirring system and a condenser at reflux. The flask containing the reaction mixture is kept in a water bath at a temperature of 60°C with moderate stirring for 20 min. Then, APS, previously dissolved in 10 mL of distilled water, was added to the flask to start the polymerization process. After 30 min, a quantity of GA was slowly added to the flask to cross-link the chains formed. Heating was stopped after 2 h of reaction. After cooling, the contents of the flask were poured into glass containers. Table 2 gives the different quantities of reagents and the different formulations used in the synthesis of the copolymer materials.

2.3 | Swelling behavior of copolymer materials

The swelling percent of the synthesized copolymer materials and the amount of water absorbed were measured as a function of time. The copolymer materials were placed in 100 mL of distilled water in a 200-mL beaker. At regular time intervals, the swollen copolymer material was removed from the beaker, dried superficially with filter paper and with a dryer to remove water bound to the

TABLE 1 Analysis results of the soil profiles A and B of the Ghardaïa region (southern Algeria).

Characteristics		**Soil A**	**Soil B**
Color		**7,5 YR 6/3 (light brown)**	**7,5 YR 4/6 (strong brown)**
pH_{water}	Soil/water ratio: 1/5	8.51	8.47
pH_{KCl}	Soil/KCl solution ratio: 1/5	8.15	7.55
EC[a] (dS/m)	Soil/water ratio: 1/5	0.19	0.13
Humidity (%)		0.09	0.09
Total limestone (%)		1.60	1.60
Organic matter (%)		0.09	0.06
Granulometry (%)	SG (200–2000 μm)	17.99	17.12
	SF (50–200 μm)	80.79	81.47
Textural class	(USDA[b])	Sandy loam	Sandy loam
Dominant cation		Magnesium	Magnesium
SAR[c]		2.47	3.31

[a] Electrical conductivity at 25°C.
[b] United States Department of Agriculture.
[c] Sodium adsorption ratio.

TABLE 2 Composition and formulation of *poly*(AAm-*co*-AA) copolymer materials.

Samples	**Composition (%)**	**AAm (g)**	**AA (g)**	**APS (g)**	**GA (g)**	**TEMED (mL)**
poly(AAm)	AAm—100%/AA—0%	5.0	0.0	0.08	0.25	0.48
poly(AAm-co-AA)—90/10	AAm—90%/AA—10%	4.5	0.5	0.08	0.25	0.48
poly(AAm-co-AA)—70/30	AAm—70%/AA—30%	3.5	1.5	0.08	0.25	0.48
poly(AAm-co-AA)—50/50	Aam—50.%/AA—50%	2.5	2.5	0.08	0.25	0.48

surface, and weighed and then quickly replaced in the same beaker. The swelling percent S% of the copolymer materials was determined from the following equation (Equation 1):

$$S\% = \frac{w_s - w_d}{w_d} \times 100, \tag{1}$$

where w_d is the dry weight of the copolymer at time zero and w_s is the swollen weight of the copolymer at time t.

3 | RESULTS AND DISCUSSION

3.1 | Characterization

3.1.1 | Fourier transform infrared spectroscopy

Figure 1 depicts the FTIR spectra of the *poly*(AAm-*co*-AA) copolymer material. The analysis of the IR spectra confirms the formation of copolymer of AAm and AA by (1) the presence of the bands in the range of 3100–3500 cm^{-1} (O-H and N-H stretching) in the *poly*(AAm-co-AA) copolymer and (2) the absence of the peak at 1642 cm^{-1}, indicating that the vinyl group (C=C) of monomers (AAm and AA) has taken part in the

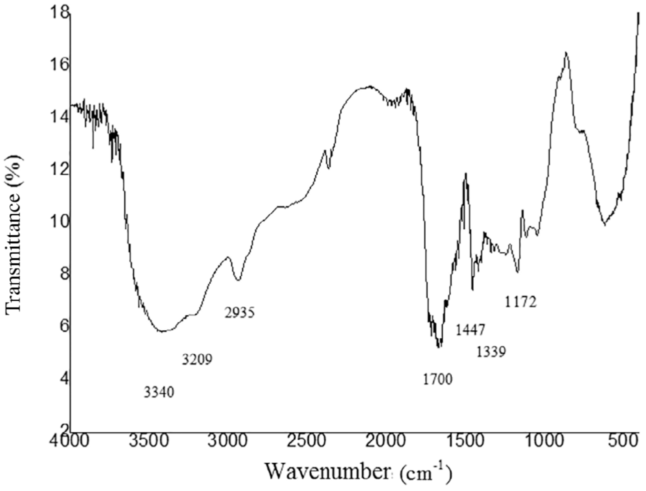

FIGURE 1 The FTIR spectra of the cross-linked copolymer of acrylamide and acrylic acid.

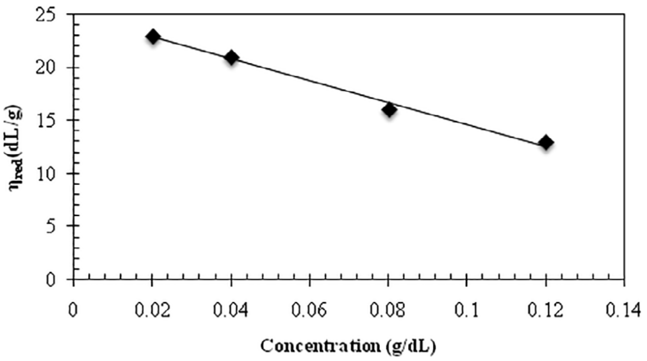

FIGURE 2 Reduced viscosity as a function of concentration of *poly*(AAm-*co*-AA) copolymer in $NaNO_3$ solution at 25°C.

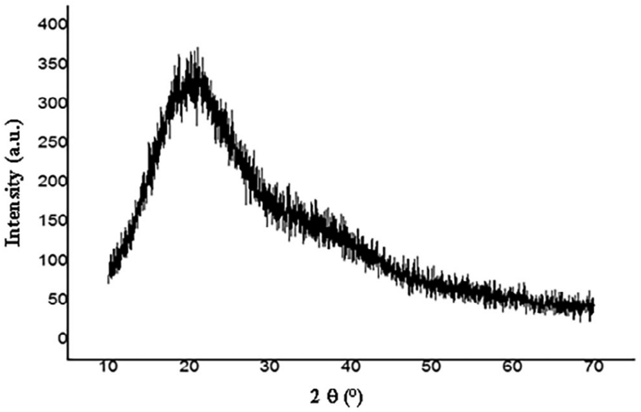

FIGURE 3 XRD pattern of the *poly*(AAm-*co*-AA) copolymer material.

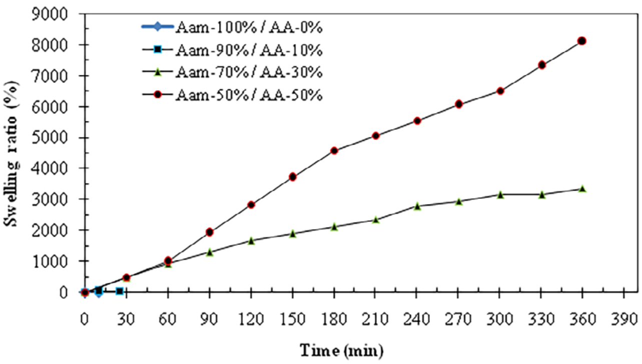

FIGURE 4 Swelling ratio over time for different compositions of AAm and AA in distilled water.

polymerization process. The intense peak at 1700 cm^{-1} is attributed to carbonyl group (C=O) bond stretching vibration.[40] Peaks due to N-H stretching of amide groups and O-H stretching are at 3200 cm^{-1} and at 3340 cm^{-1}, respectively, in spectra of the *poly*(AAm-co-AA) copolymer.[40] The doublets at 1447 and 1400 cm^{-1} are assigned to -CH_2- deformation and bending vibrations of ⟩CH-CO-group.[41] Asymmetrical stretching of C-H is found at 2935 cm^{-1}. The absence of the peak around 1642 cm^{-1} indicates that the vinyl group (C=C) of monomers has taken part in the polymerization process.[42]

3.1.2 | Estimation of viscometric molecular weight

Figure 2 shows the reduced viscosity versus concentration plot at 25°C. The intrinsic viscosity [η] of *poly*(AAm-*co*-AA) was obtained by extrapolating the straight line of the plot of reduced viscosity vs. the concentration to infinite dilution using the linear least squares method. The viscosity average molecular weight of *poly*(AAm-*co*-AA) copolymer could be estimated using the empirical Mark–Houwink–Sakurada equation (Equation 2),[43]

$$M_w = \left[\frac{[\eta]}{6.31\times10^{-3}}\right]^{1/0.80}. \qquad (2)$$

The estimated value of the viscosity average molecular weight, M_w, of the copolymer is 3.1292×10^4 g/mol.

3.1.3 | X-ray diffraction (XRD)

XRD analysis was performed to examine the crystalline nature of the *poly*(AAm-*co*-AA) copolymer material. Figure 3 shows its XRD pattern. A single broad peak, between 10° and 30°, was observed on the x-ray pattern of the *poly*(AAm-*co*-AA) copolymer. This result confirms that the structure of the said copolymer is completely amorphous, which is very similar to that obtained by Hassan M.F. et al.[40]

3.2 | Swelling study

3.2.1 | Effect of added acrylic acid on swelling

After having synthesized these four copolymer materials at different percentages of AAm and AA, we tested their swelling capacities in distilled water. Figure 4 gives the swelling ratio as a function of time of the copolymer materials for different AA compositions. As shown by these results, the swelling ratio increases dramatically with the addition of AA. Indeed, this ratio increases from 48% to 8107% for a percentage in AA, which increases from 10% to 50%. It is well known in the literature that the phenomenon of water sorption by polymers depends mechanically

TABLE 3 Maximum swelling ratio capacity of *poly*(AAm-*co*-AA) at different compositions of acrylamide and acrylic acid.

Materials	*poly* (AAm)	*poly*(AAm-*co*-AA) 90/10	*poly*(AAm-*co*-AA) 70/30	*poly*(AAm-*co*-AA) 50/50
Maximum swelling percentage (%)	9	48	3351	8107
Swelling time (min.)	10	20	360	360

on the diffusion of water molecules inside the gel matrix and a relaxation of the macromolecular chains of the hydrogel.[44] In an aqueous solution, AA is able to form hydrogen bonds and rigid frameworks. Therefore, the interaction of AA with water molecules involves several stages. The first consists in a strong diffusion of water between the chains of the copolymer by producing hydrated macromolecules, followed thereafter by their relaxations. In the last step, the copolymer solution exhibits structurization.[45] In this study, the increase in the swelling percentage with the increase in the quantity of AA in the copolymer can be attributed to the increase in the number of -COO$^-$ groups along the macromolecular chains, which induces an increase in free H$^+$ ions (counter ions) along said chains, and this causes an increase in the osmotic pressure of the swelling. These two factors result in a high swelling capacity of the copolymer. The carboxyl groups in AA exhibit a high capacity to hold water molecules compared to the amide of AAm.[46–49] Table 3 gives the effect of added AA on swelling behavior of copolymer materials.

It should be noted for the *poly*(AAm) and *poly*(AAm-*co*-AA)-90/10 polymers that after this swelling time, that is, 10 and 20 min (Table 2), respectively, they undergo a degradation.

From Table 3, the *poly*(AAm-co-AA) copolymer material containing 50% AAm and 50% AA exhibits the best swelling results in distilled water, which gave a swelling ratio of 8107. This copolymer will be used later for the study of the influence of some parameters such as the quantity of the material, temperature, and the presence of cations on the swelling ratio. This copolymer material will be denoted as *poly*(AAm-50-*co*-AA-50).

3.2.2 | Effect of the amount of *poly*(AAm-50-*co*-AA-50) on swelling behavior

The swelling kinetics of the copolymer material *poly*(AAm-50-*co*-AA-50) for three different quantities is shown in Figure 5. A very rapid increase in the rate of swelling during the first 150 min is observed for the sample with a mass of 0.03 g. After that, a degradation of the material is observed. On the other hand, for the two other materials (0.05 g and 0.07 g), an almost linear and slow increase in the swelling ratio is obtained. They reach their maximum swelling ratios after 360 min.

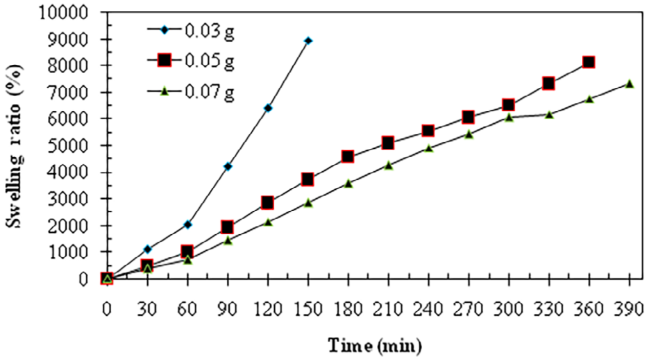

FIGURE 5 Effect of the amount of *poly*(AAm-50-*co*-50-AA) on the swelling ratio in distilled water as a function of contact time.

The quantity of copolymer material used thereafter is 0.05 g since, among these three quantities, it is the one that gives the best results in terms of maximum swelling ratio and duration of swelling.

3.2.3 | Effect of temperature

Temperature plays an important role in the absorbance capacity and strength of polymers. The effect of temperature on the swelling ratio of *poly*(AAm-50-*co*-AA-50) copolymer in distilled water was studied under three different temperatures (25, 45, and 65°C). Figure 6 gives the results of the variation in the swelling ratio of the copolymer as a function of time. This variation is almost linear for the three temperatures studied. The *poly*(AAm-50-*co*-AA-50) copolymer exhibits a slight increase in its swelling ratio at 45°C and at 65°C compared to room temperature (25°C). Indeed, the maximum swelling ratio increases from 4583% to 5590% when the temperature increases from 25 to 65°C during the first 180 min. Previous studies have shown that the effect of temperature on the structural strength of cross-linked polymers for improved swelling ratio is directly related to the amount of cross-linking agent used in the preparation of the polymer. The experimental results of Ben Ali et al.[50] showed that at a temperature of 75°C, PPGs with a low amount of cross-linking agent, such as PPG1 and PPG2, lose some of the water and decrease the swelling capacity; the PPGs with a medium amount of cross-linking agents, such as PPG3 and PPG4, continue to swell. In contrast, PPG5 and

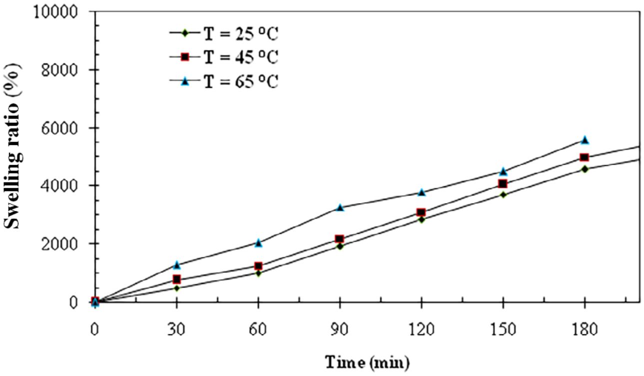

FIGURE 6 Effect of temperature on the swelling ratio of the *poly*(AAm-50-*co*-AA-50) copolymer in distilled water as a function of contact time.

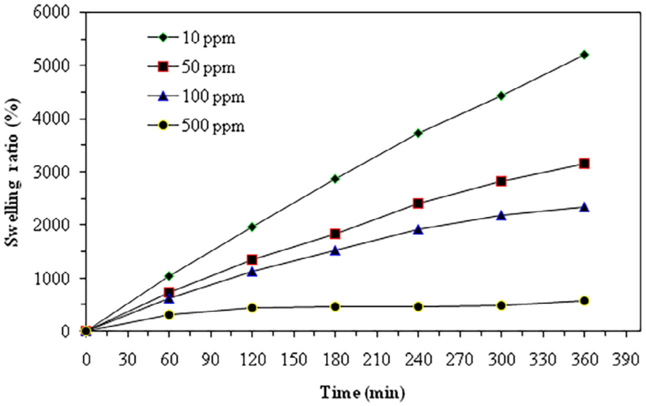

FIGURE 7 Effect of the concentration of Cu^{2+} ions on the swelling ratio of *poly*(AAm-50-*co*-AA-50) as a function of contact time.

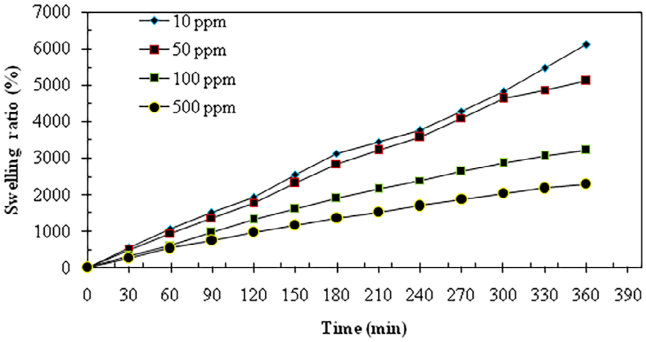

FIGURE 8 Effect of the concentration of Zn^{2+} ions on the swelling ratio of *poly*(AAm-50-*co*-AA-50) as a function of contact time.

PPG6 can resist the increase in temperature up to 100°C as they keep the swelling almost stable due to the high amount of cross-linkers they contain. Above these temperatures, all polymers undergo a collapse of the network structure and therefore a viscous state of the material. Another work carried out by Bai et al.[51] proposed that the increase in polymer swelling at higher temperatures (60°C or more) can be attributed to the hydrolysis of amino groups (–$CONH_2$) present on the polymer chains to carboxylate groups ($-COO^-$). This last hypothesis seems the most favorable for our case since our *poly* (AAm-50-*co*-AA-50) copolymer already has 50% of the amino groups on the chains of the copolymer.

3.3 | Effect of concentration of metal cation on the swelling ratio of the copolymer material

3.3.1 | Effect of concentration of Cu^{2+} ions

According to the results obtained during these experiments, which are shown in Figure 7, it can be seen that the swelling ratio decreases when the concentration of Cu^{2+} ions in the supernatant liquid is increased. Indeed, the swelling ratio of the gel in a Cu^{2+}–solution at 500 ppm is three times lower compared to that obtained under the same experimental conditions in distilled water without the metal cation. This decrease may be due to the insertion of Cu^{2+} ions.

3.3.2 | Effect of concentration of Zn^{2+} ions

The experimental results of the swelling ratio as a function of the concentration of Zn^{2+} ions are presented in Figure 8. According to these results, we notice that the swelling ratio also decreases when the concentration of Zn^{2+} ions increases in the supernatant liquid. So, a similar behavior as that of Cu^{2+} ions is observed. At this time, the swelling ratio of the copolymer in a zinc solution at 500 ppm is four times lower compared to that obtained under the same experimental conditions in distilled water without the metal cation.

3.3.3 | Effect of concentration of Fe^{3+} ions

For the results of the effect of the concentration of Fe^{3+} ions, given in Figure 9, it is noted that the maximum swelling ratio decreases considerably when the concentration of Fe^{3+} ions increases from 10 to 500 ppm. So, a same behavior as Cu^{2+} and Zn^{2+} ions is obtained.

Figure 10 gives the variation of the maximum swelling ratio as a function of the concentrations of the three metallic cations (Cu^{2+}, Zn^{2+}, and Fe^{3+}).

According to these results, when the metal cation concentration increases, the maximum swelling rate drops and this drop is more marked for the trivalent

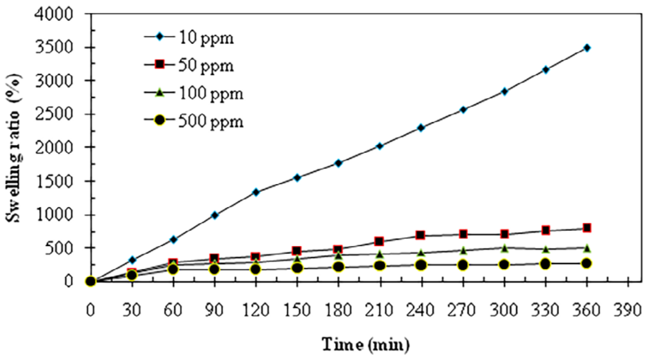

FIGURE 9 Effect of the concentration of Fe^{3+} ions on the swelling ratio of *poly*(AAm-50-*co*-AA-50) as a function of contact time.

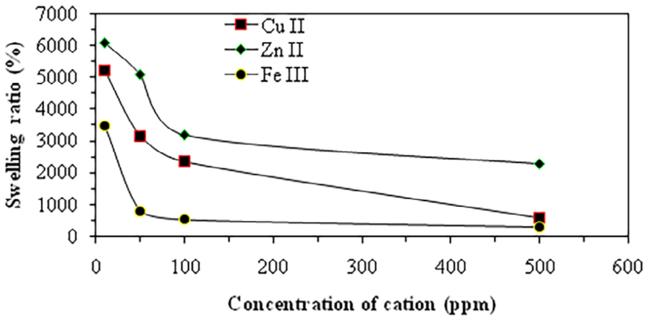

FIGURE 10 Effect of metal valence on the maximum swelling ratio as a function of the concentrations after a contact time of 360 min.

cations (Fe^{3+}) than for the divalent cations (Cu^{2+} and Zn^{2+}). The swelling mechanism at different concentrations of metal cations is shown in Figure 11. At low metal cation concentrations, electrical repulsion forces will separate the molecules of the hydrogel (the copolymer) and create more space for the fixation of water molecules. However, when the copolymer is swollen in water with a high concentration of metal cations, the negatively charged groups of hydrogel will be fixed by the cations and thus limit further water absorption.[51] The fixing bonds between the negatively charged groups located on the copolymer and the metal cation are coordination bonds where the carboxylate functions (COO^-) play the role of ligand, which form with the metal cations (Cu^{2+}, Zn^{2+}, or Fe^{3+}) monodentate, bidentate, or multidentate complexes depending on the valence of the metal cation studied. Bassaid et al.[52] showed the formation of very stable complexes between the *poly*(acrylic acid-co-crotonic acid) copolymer and the metal cations M(II) (M = Pb, Cd, and Hg) with high formation constants ($K_2 \sim 10^9$). The stability order is the following: Pb(II) > Cd(II) ~ Hg(II).

3.4 | Study of retention water using two types of soils in the presence of *poly*(AAm-50-*co*-AA-50) copolymer material

3.4.1 | Effect of quantity and position of *poly* (AAm-50-*co*-AA-50) copolymer material on water retention by the two soils

This study was carried out at room temperature for two positions of the copolymer material in each of the two soils (below forming a single layer and in a mixture with soil) and with different mass percentages of this copolymer material (0.25%, 0.5%, 0.75%, and 1%). All experiments were performed for a 50 g quantity of soil. Table 4 gives the different amounts of the copolymer material as well as their mass percentages relative to the mass of the soil used.

Into each plastic container, a quantity of 50 g of the soil was introduced, and the appropriate quantity of the copolymer material was added for two different positions (below forming a single layer and in a mixture with soil). Using a graduated burette, suspended above the container, water was gradually added until a thin layer of water appears on the surface of the soil. Then, using a filter paper, the excess water remaining on the surface was removed. The maximum water retention ratio τ_{H_2O} was determined from the following relationship (Equation 3):

$$\tau_{H_2O} = \frac{(m_2 - m_1)}{m_1}.100, \quad (3)$$

where, m_1 is the dry sample mass and m_2 is the mass of the sample saturated with water.

Table 5 gives the results of the maximum water retention ratio obtained for the soil A for different quantities of the copolymer material and for two different positions (below and in the mixture).

Figure 12 illustrates the variation of the maximum water retention ratio of the copolymer material–soil A system for each percentage and for each position of the copolymer material with respect to the soil.

Table 6 gives the results of the maximum water retention ratio obtained for soil B for different quantities of the copolymer material and for two different positions (below and in the mixture).

Figure 13 illustrates the variation in the maximum water retention ratio of the copolymer material–soil B system for each percentage and for each position of the copolymer material with respect to the soil.

According to these results, we notice that the maximum percentage of the copolymer material in the soil is for 1% in a composition with the two studied soils.

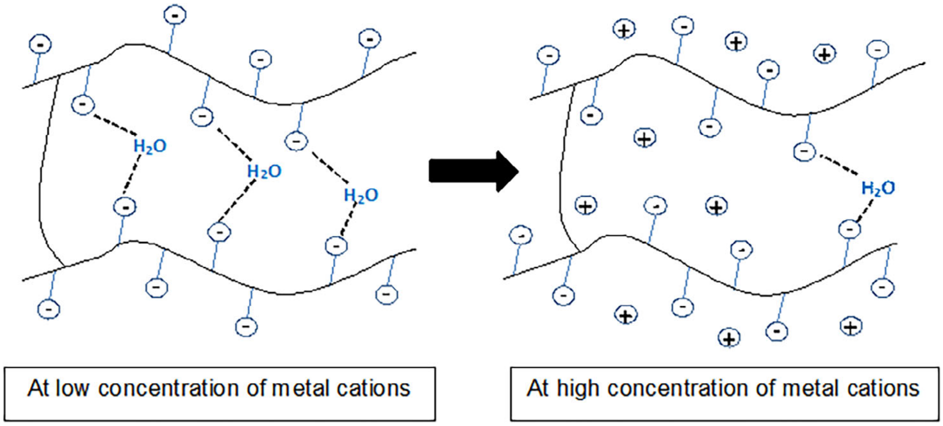

FIGURE 11 Swelling mechanism of *poly*(AAm-50-*co*-AA-50) copolymer in solution containing metal cations.

TABLE 4 The different quantities (in mass and in %) of the copolymer material used relative to the mass of soil.

Quantity of copolymer material (g)	0.125	0.250	0.375	0.500
Mass percentage of copolymer material (%)	0.25	0.50	0.75	1.00

TABLE 5 Maximum water retention ratio obtained for soil A for different quantities of the copolymer material and for two positions (below forming a single layer and in a mixture with soil).

	Below forming a single layer		In mixture with soil	
Amount of copolymer material added (%)	**Water retention ratio (%)**	**Improvement contributed (%)**	**Water retention ratio (%)**	**Improvement contributed (%)**
0.00 (soil A alone)	17.7	/	17.7	/
0.25	19.9	2.2	20.6	2.9
0.50	23.2	5.5	24.9	7.2
0.75	26.4	8.7	27.4	9.7
1.00	28.6	10.9	29.9	12.2

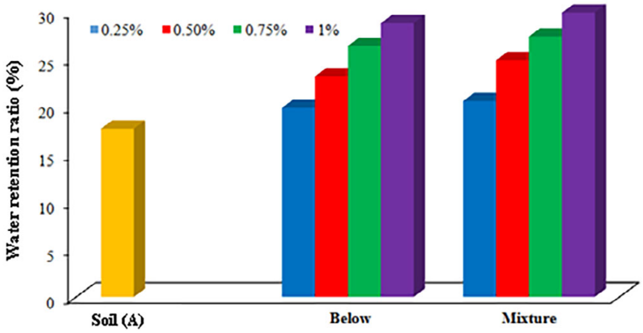

FIGURE 12 Water retention ratio of the copolymer material—soil A system as a function of the percentages of the copolymer material and its position with respect to the soil.

As expected, it can be seen that the presence of the copolymer material in the soil makes it possible to increase the water retention ratio of the soil. Indeed, soil A alone can retain 17.7% of water, and soil B alone can retain 25.2% of water. These values increase considerably in the presence of the copolymer material as the percentage thereof increases, regardless of its position (below forming a single layer and in a mixture with soil). For a percentage of copolymer material of 1% and in the mixture position, the maximum water retained is 29.8% for soil A and 33.1% for soil B, respectively. This gives us an improvement of 12.2% for soil A and 7.9% for soil B. This can be explained by the mass effect and the distribution of the copolymer material in the soil. Indeed, when the copolymer material is placed below with the soil forming a single layer, there is an insufficient intensity of the repulsions of an electrostatic nature between the macromolecular chains, responsible for the swelling, to repel the grains of the soil and allow the material to swell freely. However, when the copolymer material is mixed with the soil, there is a homogeneous distribution of this material throughout the soil, which allows all the grains of this material to swell in a homogeneous way and all contribute to the retention of some water. Thus, the water retention ratio of the copolymer–soil system increases.

TABLE 6 Maximum water retention ratio obtained for soil B for different quantities of the copolymer material and for two positions (below forming a single layer and in a mixture with soil).

	Below forming a single layer		In mixture with soil	
Amount of copolymer material added (%)	**Water retention ratio (%)**	**Improvement contributed (%)**	**Water retention ratio (%)**	**Improvement contributed (%)**
0.00 (soil B alone)	25.2	I	25.2	I
0.25	25.6	0.4	29.2	4.0
0.50	27.5	2.3	30.2	5.0
0.75	30.8	5.6	31.5	6.3
1.00	31.9	6.7	33.1	7.9

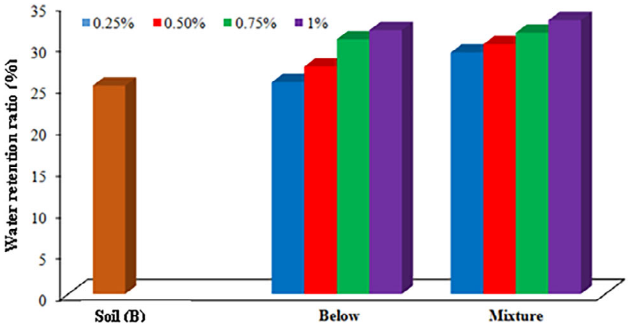

FIGURE 13 Water retention ratio of the copolymer material—soil B system as a function of the percentages of the copolymer material and its position with respect to the soil.

Certainly, *poly*(AAm-50-*co*-AA-50) copolymer has good water retention capacities when mixed with soils A and B, but the percentage of 1% by weight relative to the soil seems to be high compared to those found in the literature. Indeed, the work of Chen et al.[1,34] showed that the addition of 0.1% by weight of a superabsorbent polymer (SAP), prepared by copolymerization by grafting AA and AAm onto starch, to soil and sand samples significantly improves their water retention. This is due to the effect of various polymer synthesis parameters (monomer concentration, γ-radiation dose, monomer to starch ratio, and poly(ethylene glycol) content). Research by Andrey V. Smagin et al.[53] confirms the possibility of combining various technological properties in a single gelling polymer material, capable of significantly improving the water retention of the soil. This is due to the optimal ratio of acrylic copolymers and the filling of the polymer matrix with components amphiphiles and trace elements and biocides in the form of silver ions and nanoparticles or suitable organic fungicides. The use of small percentages (0.1%–0.3%) of these new polymeric gelling materials can increase the retention of water and the dispersity of sandy substrates.

In the experimental work of Jahangir et al.,[54] the use of 0.8% by weight of PR3005A hydrogel with sandy loam significantly increases the water content in the soil. This can be explained by the chemical structure of the hydrogel in question. The presence of two functional groups produces a hydrogen bonding with water, that is, NH_2 of AAm and COOH of AA in PR3005A hydrogel. In the work of Shaukat Ali Shahid et al.,[55] a sandy-loamy soil was amended with different concentrations (0.1%–0.4%) of *poly*(acrylamide-co-acrylic)/$AlZnFe_2O_4$-synthesized superabsorbent hydrogel nanocomposite (SHNC). The results of the study show an increase in the water retention in the soil with the increase in the concentration of SHNC, and the highest moisture retention value was obtained with the addition of 0.4 wt% SHNC to the soil. The experimental results of Al-Darby et al.[56] demonstrate that the addition of 0.2%, 0.4%, and 0.6% of AquaSorb to sandy soil increases the amount of water retained to 8.5%, 17.4%, and 31.6%, respectively. In the light of what was said above, it can be concluded that the percentage of 1% of *poly*(AAm-50-*co*-AA-50) copolymer hydrogel used in this study is 2.5 times higher than that found in the literature. This is probably due to the low quality of this hydrogel because all the polymers used in the studies cited above are either polymers synthesized under well-chosen operating conditions for these applications in agriculture or commercial polymers carefully manufactured for the retention of water in the soil.

3.4.2 | Study of the effect of the copolymer material on some soil properties

In this second part, we try to highlight the effect of the presence of the same copolymer material, used in the first part of this application, on some soil properties such as

- the water retention and filtration capacity of a soil,
- the infiltration capacity of water in a soil (its permeability),
- the amount of air in a soil.

TABLE 7 Volumes and percentages of water retention by soils alone and soils mixtures with 1% copolymer.

	Volume of water retained (mL)		% water retention	
Soil type	**Soil alone**	**Soil with 1% copolymer material**	**Soil alone**	**Soil with 1% copolymer material**
A	8	10	33.3	41.7
B	8.8	9.5	36.7	39.6

In these tests, the *poly*(AAm-50-*co*-AA-50) copolymer material is in a mixture position in each of the two soils (A and B) and with a composition of 1% by weight.

Measurement of the water retention and filtration capacity of a soil

In a clean beaker, 5 drops of a dye, bromothymol blue (BBT), is mixed with 300 mL of water. A filter paper is placed in a funnel placed above an empty graduated beaker; the funnel, containing the filter paper, is filled with 30 g of the soil alone or of the soil mixed with *poly*(AAm-50-*co*-AA-50) copolymer material. A small hole (1 cm deep) is dug in the center of the funnel, and 24 mL of colored water was gently poured into it. For these tests, two control beakers are made by pouring 24 mL of colored water into the first and 24 mL of colored water into the second containing a funnel with a filter paper without the soil.

According to the measurements carried out, the beakers do not contain the same amount of water after filtration. The two soils therefore do not retain water in the same way. Table 7 gives the results of the volumes and percentages of water retention by the soils alone and the soil mixtures with 1% *poly*(AAm-50-*co*-AA-50) copolymer material. Addition of 1% of *poly*(AAm-50-*co*-AA-50) copolymer material increases the percentage of water retention in the two soils. Indeed, it increases from 33.3% to 41.7% in soil A and from 36.7% to 39.6% in soil B. It is evident that the addition of *poly*(AAm-50-*co*-AA-50) copolymer material substantially increased the water retained by the sandy soil. The amount of water remaining in the soil at equilibrium is a function of sizes of water-filled pores. For example, in work of A. M. Al-Darby et al.,[56] the addition of AquaSorb to sandy reduced the pore median diameter (d_m) from 46 to 23 μm when the amount of AquaSorb increased from 0.0% to 0.6%. Consequently, there was an increase in a volumetric soil water content (θ) from 4.8% to 31.6%.

In addition to that, a more accentuated coloring is observed in the beaker of the soil (A) alone than that of the soil (B) alone. As regards the beakers of the soils containing *poly*(AAm-50-*co*-AA-50) copolymer material, no coloration is observed. Thus, the ability to filter and purify water differs depending on the type of soil.

Measurement of water infiltration capacity in soil (its permeability)

In this test, we cut three plastic containers to the same height (2.5 cm). We pierce, with the help of the point of the compass, two small holes at the bottom of each of the three recipients using a spoon; the three containers are filled with 30 g of the soils alone or of the soils mixed with *poly*(AAm-50-*co*-AA-50) copolymer material. Pack the soil well with a finger in the first container and a little less into the second container by tapping the bottom on the table, and do not compact the soil in the third container. We place three identical small pieces of filter paper on each container. We fill a flat plate with water, and then, we place the three containers in the water at the same time. The time during which the three filter papers become wet is noted.

After a while, the pieces of filter paper are wet: it is as if the soil sucks the water upward. This force that drives the water is the capillarity force. For the water to rise quickly, the soil must be loose: The water is then close to the grains to which it clings. If the soil is too packed: the holes are too tight, the path to be covered then becomes too long. To have a good capillarity, the grains of the soil should not be too tight. This is how plants continue to drink through their roots when the top layer of soil contains little water. Table 8 summarizes the durations of water infiltration in the two soils.

The presence of *poly*(AAm-50-*co*-AA-50) copolymer material (1%) in soil B does not hinder the rise of water in the soil. This result is confirmed by the durations of infiltration, which are close to each other (Table 8). On the other hand, the presence of *poly*(AAm-50-*co*-AA-50) copolymer material in soil A considerably increases the duration of infiltration. For example, the duration of infiltration of unpacked soil containing 1% of the polymeric material is multiplied by a factor of 17.22. This can be explained by the large space that exists in soil A, which is occupied by the copolymer material, which takes a considerable time to swell and pass water. Thus, after soil watering, the hydrogel copolymer mixed with the soil absorbs water by swelling and releases it slowly as the soil dries off by diffusion. Irrigation water losses and evaporation losses are, thus, avoided.[57]

TABLE 8 Duration of infiltration in soils alone and in soils mixed with the copolymer material (1%).

	Infiltration time (s)			**Infiltration time (s)**		
	Soil (A) alone	**Soil (A) +1% copolymer**	**Report**	**Soil (B) alone**	**Soil (B) +1% copolymer**	**Report**
Soil condition	**t_1(s)**	**t_2(s)**	**t_2/t_1**	**t_1(s)**	**t_2(s)**	**t_2/t_1**
Well packed	105	489	4.66	155	336	2.17
Half packed	44	259	5.88	129	150	1.16
Not packed	9	155	17.22	80	104	1.30

TABLE 9 Volume and percentage of air contained in soils.

	Soil A only	**Soil A +1% copolymer**	**Soil B only**	**Soil B +1% copolymer**
V_f (ml)	49	43	50	44
V_a (ml)	11	16.5	10	15.5
% air	36	55	33	52

Measurement of the amount of air in a soil

30 mL of soils alone or of soils mixed with *poly*(AAm-50-*co*-AA-50) copolymer material is deposited in a graduated cylinder, and the soil is stirred to obtain a roughly flat surface, and then, 30 mL is read on the graduation of the cylinder; 30 mL of water is poured into the test tube containing the soil. The whole thing is gently stirred with the help of a rod, and then, the final volume reached by the soil and the water is read. The volume of air contained in the soil sample is calculated from the following equation (Equation 4):

$$V_a = (V_s + V_W) - V_f, \tag{4}$$

where, V_a is the volume of air contained in the soil, V_s is the volume of soil solids, V_W is the volume of water added, and V_f is the final volume read on the test tube.

It is well known that the amount of air in a soil depends on two very important properties: its texture (grain size) and its structure (variety of pore sizes in which air and water circulate). After mixing, the water poured onto the soil samples (soils alone and in the presence of *poly*(AAm-50-*co*-AA-50) copolymer material) will take the place of the air between the particles. The results of the calculations of the volumes of air contained in the soils are given in Table 9.

In general, the water retention changes strongly with the air entry value (h_b). Due to the larger pore geometry in sandy loam soils, water is released under lower matric suction. Indeed, in the study carried out by J. Abedi-Koupai et al.,[54] the value of h_b increases from 0.68 for the sandy loam soil without the hydrogel to 1.54 (more than double) for an amount of 0.8% hydrogel PR3005A added, which allows the reduction of the largest pores in the soils, and the pressure required for water expulsion is increased. Therefore, adding hydrogel to these soils may open the medium, by forcing soil particles apart, thereby increasing air penetration.

According the literature, the main factors contributing to the changes in soil properties after adding the copolymer material are pH, electrical conductivity (EC), and cation exchange capacity (CEC) as these factors have a direct effect on plant growth.

Prabha et al.[58] showed that soil pH can be affected by adding SAPs. The results obtained show that the addition of 0.5% of SAP to a sandy clay loam soil, undergoing two watering intervals over a 2-week study period, reduced the soil pH from 6.04 to 5.8 and this decrease was attributed to the carboxylate ions present in the SAP.

Prabha et al.[58] also showed that the CEC can be influenced by the addition of SAPs. For example, in sandy clay loam soil, the control soil had an initial EC of 1.5 dS m^{-1}, and after 13 weeks, the EC of the control soil treated with 0.5% of SAP increased to 5.3 dS m^{-1}. The increase in EC was associated with acids and ions present in the SAP.

Just as SAPs can alter soil pH and EC, they can change CEC. For example, addition of 0.2% SAP to sandy loam soil increased the CEC from 6.2 to 8.2 cmol kg^{-1}, but no change was observed at an application rate of 0.07%.[59] Similar to changes in soil pH and EC, changes in CEC are associated with the composition of SAPs.

4 | CONCLUSION

In this work and in a first part, we synthesized copolymer materials with various compositions of AAm and AA

($2.5 < AAm < 5.0$ g and $0.0 < AA < 2.5$ g) by radical polymerization reaction in aqueous solution using APS as initiator, TEMED as activator, and GA as cross-linking agent.

In order to confirm the completion of the polymerization reaction, we analyzed our copolymer materials obtained by different characterization techniques such as FTIR spectroscopy and XRD. The IR spectra of the copolymer sample obtained clearly confirm the formation of copolymer of AAm and AA by the presence of the bands in the range of 3100–3500 cm^{-1} (O–H and N–H stretching) in the *poly*(AAm-co-AA) copolymer. The absence of the peak located at 1642 cm^{-1}, which characterizes the C=C double bond, confirms that this vinyl group has participated in the polymerization process. The XRD spectra of the *poly*(AAm-*co*-AA) copolymer material give a single broad peak, between 10° and 30°, which confirms that the structure of said copolymer is completely amorphous. The average molecular weight, M_w, of the *poly*(AAm-*co*-AA) copolymer material was estimated by viscosity measurements in $NaNO_3$ solution at 25°C, using the Mark–Houwink equation; its value is around 3.1292×10^4 g/mol.

Then in a second part, we tested the swelling capacity of these copolymer materials in distilled water. The obtained results show that the swelling ratio increases significantly with the addition of AA in the copolymer material. The material containing 50% in AAm and 50% in AA has the best swelling ratio, which is 8107%. This material has been the subject of the study of the effect of some parameters on its swelling capacity. The increase in polymer swelling with temperature can be attributed to the hydrolysis of amino groups ($-CONH_2$) present on *poly*(AAm-50-*co*-AA-50) copolymer chains to carboxylate groups ($-COO^-$) because this copolymer already has 50% of the amino groups on the chains of the copolymer. The swelling capacities of *poly*(AAm-50-*co*-AA-50) copolymer material increase from 4583% to 5590% when temperature increases from 25 to 65°C, respectively. The presence of metallic cations causes a drop in the swelling ratio of the copolymer material. This effect is more accentuated in the presence of trivalent metallic cations. This material can form very stable inter-chain complexes with carboxylate groups, thus preventing the expansion of the polymeric network. The order of stability of these complexes is as follows: $Fe^{3+} > Cu^{2+} > Zn^{2+}$.

In the last part, we studied and evaluated the water retention in two sandy loam soils in southern Algeria (Ghardaïa region) in the presence of *poly*(AAm-50-*co*-AA-50) copolymer material for different percentages and for two different positions of the copolymer material in the soil. The results of this study show that the best composition of the copolymer material in the soil is for 1% with the two soils studied.

The experimental results obtained, for some properties of the soil in the presence of the copolymer material, revealed the following conclusions: An improvement in the water retention ratio of 12.2% for soil A, an improvement in the water retention ratio of 7.9% for soil B, duration of infiltration multiplied by a factor of 17.22 for unpacked soil A, duration of infiltration multiplied by a factor of 1.3 for unpacked soil B, and an improvement in the volume of air contained in the soil of 19% for the two soils.

The remarkable results of the study of the swelling ratio and the water retention capacity of this *poly*(AAm-50-*co*-AA-50) copolymer material, given by the values of the maximum swelling ratio and the very interesting improvements made in the two sandy loam soils studied, show that this *poly*(AAm-50-*co*-AA-50) copolymer material is a good water retention agent, and it can be used in plant protection and growth in agriculture field.

AUTHOR CONTRIBUTIONS

Khaled Djakhdane, Fadhila Chaibi, and Chahrazed Cheikh contributed to methodology, data collection, and interpretation. Salah Bassaid wrote the original draft and prepared the manuscript. Abdelkader Dehbi Ali Alsalme and Pietro Picuno conceived the study, and interpreted, reviewed, and edited the manuscript.

ACKNOWLEDGMENTS

The authors would like to acknowledge the Researchers Supporting Project (RSP-2023R78), King Saud University, Riyadh, Saudi Arabia.

CONFLICT OF INTEREST STATEMENT

The authors declare that they have no competing interest.

DATA AVAILABILITY STATEMENT

Data sharing not applicable to this article as no datasets were generated or analysed during the current study.

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How to cite this article: Djakhdane K, Chaibi F, Cheikh C, et al. Synthesis, characterization, and swelling behavior of a *poly*(acrylamide-*co*-acrylic acid) copolymer: Application to water retention in two sandy agricultural soils in sub-Saharan region. *Polym Eng Sci*. 2023;1-15. doi:10.1002/pen.26474