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POLYACRYLAMIDE HYDROGEL PROPERTIES FOR HORTICULTURAL APPLICATIONS

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Polyacrylamide (PAAm) hydrogels are commonly employed to ensure soil hydration in horticulture, but studies have shown that they have a minimal effect on crop life and quality. The reasons for this poor performance are not understood since the commercial hydrogels have not been adequately characterized. PAAm hydrogels were synthesized and their properties were measured along with those of commercial hydrogels. Hydrogel swelling, density, and SEM analyses showed that the commercial hydrogels were most likely a derivative of PAAm with ionic groups and they were able to retain moisture for only a few hours.

Keywords**:** Dehydration rate; Hydrogel degradation; Ionic hydrogel; Mechanical properties; Polyacrylamide gel

INTRODUCTION

There is a strong need for plant growth media with increased water and nutrient holding capacity.[1] In addition to reducing the frequency of irrigation during crop production, such hydration strategies are essential for minimizing postproduction crop loss in retail outlets. Polymer hydrogels are a commonly available product for ensuring adequate hydration as they have the ability to absorb large quantities of water. Among these hydrogels polyacrylamide (PAAm) is one of the most popular, having also been used to reduce water runoff and increase infiltration rates in field agriculture,[2,3] in addition to increasing water holding capacity for horticultural and agricultural applications.[4–6]

Despite their promise, PAAm hydrogels have a mixed record in horticulture. Several studies have suggested that these hydrogels can delay wilting and extend crop life and quality.[7,8] However, other studies have reported little or no improvement at

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best or detrimental ones at worst.[9] In most of these studies the hydrogels have not been fully characterized so it is not easy to determine if these variations are due to different properties of the hydrogels employed or to other factors. If such hydrogels are to be widely accepted for horticultural uses it is important that they show a clear enhancement in water retention and a corresponding improvement in plant performance when employed in growth media.

A recent study of the effect of PAAm hydrogels on bedding plant production highlights these mixed results. Unlike in previous studies, the plant growth and development and media characteristics of plants were simultaneously investigated on a daily basis throughout plant production.[10] For some plants the presence of hydrogels did increase flower production, but not for others. Similarly, the hydrogels seemed to increase root growth and decrease irrigation frequency initially for some plants, but showed little benefit over the entire production period. Overall, the benefits to plant production were minimal for any plant.

The key to understanding and improving the performance of the PAAm hydrogels is their properties, which depend on their molecular structure. These gels are made from acrylamide (AAm) monomers, whose structure consists of a carbon double bond and the $-CONH_2$ group, as well as suitable amounts of a cross-linker. Depending on the application, their properties can be varied by adjusting the synthesis conditions, by copolymerization with other monomers, and through chemical modification of the synthesized hydrogel.[11–13] For example, in high pH solutions, the $-CONH_2$ groups become hydrolyzed, causing repulsion between the resulting charged carboxylate groups, which will extend the polymer chains. This creates large spaces within the gel to absorb water molecules and increase swelling.[14] While such high water absorption may be desirable, it causes the gel to become extremely fragile and break up easily when deployed in growth media. This breakup in turn can cause the water retention ability of the gel to diminish significantly. This suggests that for their use in growth media appropriate PAAm hydrogels need to be synthesized with an optimal balance between the water absorption and retention properties.

In this study we seek to understand the properties of commercial PAAm hydrogels developed for agricultural applications, particularly their water absorption and retention properties. As the exact composition of these gels is not known, we will synthesize PAAm hydrogels of different compositions and compare their properties. These properties will be studied under simulated plant growth conditions to determine if these hydrogels perform as expected.

MATERIALS AND METHODS

Acrylacmide (AAm: ultra pure) and N,N′-methylenebisacrylamide (MBAAm, 99% purity) were purchased from Polyscience and used without purification. Ammonium persulfate (APS, 98% purity), hydrochloride acid (HCl, 37% purity), N,N,N′,N′-tetramethylethylenediamine (TEMED, 99.5% purity), and triethylamine (TEA, 99.5% purity) purchased from Sigma-Aldrich were used without further purification. Sodium hydroxide (NaOH, 99.5% purity) was purchased from Fisher Scientific. Commercial hydrogels were obtained from agricultural suppliers.

Polyacrylamide Hydrogel Synthesis and Functionalization

Hydrogel synthesis. For a typical PAAm hydrogel synthesis AAm (2 g, 28.1 mmol) and MBAAm (1 mg, 0.0065 mmol, 0.023 mol %) were mixed in a dry 50 mL filter flask to which 20 mL deionized (DI) water was added. The monomer solution was gently swirled with a magnetic bar on a stirring plate until all reactants were completely dissolved. The monomer solution was deoxygenated for 15 min to prevent the reaction between oxygen and the initiators. Following this, 60 µL of APS solution (10% (w/v)) and 10 µL of TEMED were added to initiate polymerization, and the solution was swirled gently five or six times by hand to mix all reactants and poured into a polypropylene petri dish under nitrogen atmosphere. The solution was polymerized at room temperature for 2 h. The synthesized gel was immersed in deionized (DI) water for five days, with water changes three times a day to remove any unreacted monomers.

Hydrogel functionalization. Using HCl, NaOH, TEA, and DI water, different solutions at pH 3, 4.8, 6.8, 10, and 13 were prepared, with the pH measured by a pH meter. For each solution, 150 mL was prepared, and it was evenly divided into three 100 mL beakers. One round sample (diameter: 15.84 mm, thickness: 7 mm) cut by a cork borer was immersed into each beaker at room temperature. The weight of each sample was measured at least twice a day until it attained a constant value. The increased weight of hydrogels is an indication of their degree of ionization.

Swelling of Hydrogels

The hydrogel samples were placed in a plastic dish in DI water at room temperature. With DI water changed at least three times per day for the first five days, the weight of the samples was measured. After the fifth day, the DI water was changed only once per day. Before the samples were weighed on a digital balance, they were wiped to remove excess water on the surface. The samples were weighed at least twice a day until an equilibrium was reached. The samples were then dried at 80°C in a vacuum oven for three days to obtain their dried gel weight. The commercial hydrogels were obtained in the dried state and were weighed before immersion in DI water. The extent of gel swelling was measured by the swelling ratio, which is defined as:

$$\text{Swelling ratio} = \frac{\text{Equilibrium wet weight of gel}}{\text{Weight of dried gel}}$$

SEM Analysis

Samples of commercial and synthesized hydrogels were hydrated with reverse osmosis (RO) water, frozen in liquid N_2, and lyophilized overnight. Dried samples were then coated with 40 nm gold, fractured with a razor blade, then coated again with 20 nm gold. Prepared samples were then viewed with a scanning electron microscope (SEM, Hitachi model 3500 and model S-4700). Beam voltage was 15 kV for all magnifications up to 3,000×.

Mechanical Property Measurement

The hydrogels at equilibrium swelling were cut into cubes with a razor. For each hydrogel three samples were prepared, and the results of compressive strength were calculated from their mean value. The dimensions of the samples were measured with a digital caliper. The samples were gently wiped to remove excess water and prevent movement during measurement. The circular flat head of the tensile tester (Instron TTC M1) compressed each sample at a speed of 2 inches per minute, during which the maximum load was measured. The compressive strength was defined by:

$$\text{Compressive strength} = \frac{\text{Maximum load on gel}}{\text{Cross sectional area of gel}}$$

Dehydration Experiments

The dehydration time of commercial and our synthesized gels was measured by employing a simple filter paper (Whatman Schleicher & Schuell No. 2) test at a temperature of 50°C and relative humidity (RH) of 37% in an incubator (Fisher Scientific Isotemp Standard Lab Incubator 11-690-637D). The hydrogel samples were cut with a cork borer (diameter: 15.84 mm) to produce samples of the same size. The thickness of all samples was 7 mm. The equilibrium swollen gel, after excess water was wiped off, and the filter paper were weighed separately. The gel was placed on the filter paper, and then in the incubator. Their weight was measured at regular time intervals until the weight of a sample and a filter paper was unchanged. The weight of the filter paper was subtracted from the total weight to obtain the gel weight. All data of dehydration ratio resulted in the mean value of three samples for one kind of hydrogel. The dehydration ratio was defined by the formula:

$$\text{Dehydration ratio} = \frac{\text{Weight of gel at time } t}{\text{Weight of initial wet gel at } t = 0}.$$

Polyacrylamide Degradation

The degradation characteristics of the commercial hydrogels was measured by following their concentration in solution. Hydrogel samples were broken into small pieces and dispersed in water. Samples from this stock solution were placed in flasks and subjected to three different conditions for up to 73 days: with sunlight at ambient conditions, with no light at ambient conditions, and with no light in a refrigerator at ~3°C. Three flasks were set up for each of these conditions and they were kept stoppered at all times. Three aliquots were taken periodically from each flask and run through a liquid chromatography-mass spectrometry system (Varian 1200 L triple stage quadrupole mass spectrometer) to determine the PAAm concentration. Two separate runs were carried out for each of the three conditions.

RESULTS AND DISCUSSION

The polyacrylamide (PAAm) hydrogels synthesized for this study had significantly different properties than the commercially available PAAm gels. The properties were investigated in more detail to determine the reasons for this difference. This investigation was also needed to understand the fragility of these gels and their poor performance as growth media for plants.

Swelling Behavior of Commercial and Synthesized PAAm Hydrogels

Numerous acrylamide samples with varying conditions were synthesized in an attempt to match the high swelling of the commercial acrylamide gels. While it was possible to increase the extent of swelling somewhat, the synthesized gels did not come close to matching the swelling of the commercial gels. Table I represents among the highest swelling that could be achieved for the synthesized PAAm gels by varying their synthesis composition.

The swelling ratios of a few of the synthesized gels were higher than that of the gel in Table I. These gels had lower cross-linker concentrations, which made them extremely fragile and difficult to handle in the swollen state, more so than the commercial hydrogels. As a result representative synthesized gels in this study were limited to those that had a minimum level of strength and retained their shape in the swollen state, such as that listed in Table I.

The large difference in swelling ratios shown in Table I clearly suggests that the commercial hydrogels are significantly different from the PAAm hydrogels synthesized here. There are two possible reasons for this: (1) the commercial hydrogels are PAAm hydrogels but were chemically modified to enhance their swelling, or (2) the commercial gels are regular PAAm hydrogels but their synthesis conditions were modified to facilitate greater swelling. The common approach to enhancing the swelling of hydrogels by chemical modification is by the addition of ionic groups to the gels following synthesis. Similarly, modifying the gel synthesis conditions, either by decreasing the cross-linker concentration or by using porosity generators to create superporous gels, can increase swelling. We will investigate these possibilities for the commercial hydrogels next.

Synthesis of Ionic Hydrogels

Similarly charged ionic groups can greatly enhance the swelling of hydrogels through repulsion and increased binding of water molecules.[14,15] One of the easiest

Table I. Comparison of swelling of typical samples of commercial and synthesized gels

	Commercial gel	Synthesized gel
Composition	Unknown	2 g AAm, 1 mg MBAAm, and 20 mL DI water
Swelling ratio	326.2	71.9

Table II. Effect of reaction with NaOH and TEA solutions to form ionic groups on the swelling of synthesized gels

		Swelling ratio		
Gel composition	Texture	Unmodified	NaOH hydrolysis	TEA hydrolysis
4 g AAm, 100 mg MBAAm, and 10 mL water	Hard	13.8	75.2	47.2
2 g AAm, 1 mg MBAAm, and 20 mL water	Soft	71.9	349.3	409.7

approaches for adding ionic groups to PAAm gels is through the hydrolysis of the amide group in acrylamide in high pH solutions:

$$CH_2{=}CH{-}CO{-}NH_2 + H_2O \rightarrow CH_2{=}CH{-}COO^- + H^+ + NH_3$$

Two different hydrolysis reactions were carried out here: the first with a NaOH solution and the second with triethylamine (TEA), as described in the materials and methods section. The equilibrium swelling of the resulting hydrogels is shown in Table II.

Hydrolysis was carried out for two different synthesized hydrogel samples, one with high cross-linker concentration producing a harder gel and the other the same soft hydrogel in Table I. The hydrolysis process transformed both of these gels into softer ones, with the latter gel becoming extremely fragile, similar to the commercial hydrogel. The swelling of both hydrogels increases dramatically following hydrolysis, with that of the soft hydrogel slightly exceeding that of the commercial one. This similarity in swelling, as well as in their texture, is a good indication that the commercial hydrogel and the soft synthesized gel are similar. This suggests that the commercial hydrogels are a slightly more cross-linked version of the soft hydrogel that have been functionalized with ionic groups.

While the above increased swelling in the synthesized gels can be attributed to the hydrolysis of the amide groups in acrylamide, it is necessary to verify that this was not caused merely by the change in pH of the hydrolysis solution. The swelling ratios of the gel at various pH values were measured and the results are shown in Table III. It can be seen that the pH does have a modest effect on the swelling of this gel, as reported by other investigators for PAAm hydrogels.[16,17] However, this pH effect cannot account for the dramatic increase in swelling observed following treatment with the hydrolysis solution, which can only be achieved by the hydrolysis of the amide groups in the hydrogel.

Table III. Effect of various pH solutions on the swelling of one of the synthesized gels (2 g AAm, 1 mg MBAAm, and 20 mL water) in Table I

	pH value				
	3	4.8	6.8 (DI water)	10.0	13.0 (Hydrolysis solution)
Swelling ratio	62.5	66.8	71.9	76.9	349.3

Microstructural Analysis of Hydrogels

As shown earlier in Table III it is possible to increase the swelling of PAAm hydrogels by decreasing the cross-linker concentration during synthesis. However, this is insufficient to account for the large swelling ratios observed in commercial hydrogels. Another approach to increasing hydrogel swelling is to use porosity generators (porogens) during synthesis to produce superporous hydrogels.[18] It is possible that the commercial hydrogels were produced in this manner.

A simple approach to verify if the commercial hydrogels are superporous is to compare their densities with those of the synthesized gels. In their dried state superporous hydrogels can be expected to have much lower densities than the regular hydrogels. To measure the density of gels of both types, the synthesized hydrogel samples were first dried in an oven at 50°C for 10 h. The dried gels were hardened with liquid nitrogen, ground evenly in a grinder, and sieved into different sized particles. The pellet size distribution of dried commercial hydrogel was 1–2 mm, and synthesized hydrogel particles of the same size distribution were used to compare the densities of both. The pellets were placed in micro centrifuge tubes and their weights were obtained by subtracting the weight of the tubes from the total weight, as well as the volume occupied by the pellets. The density of commercial hydrogel samples was found to be 0.70 g/cm^3, and that of synthesized hydrogels was found to be 0.65 g/cm^3. The comparable densities of the samples clearly suggest that the commercial hydrogels are not superporous ones.

A more definitive method to verify if the commercial hydrogels are superporous is by using SEM analysis of the samples. Typical SEM images of the commercial and synthesized hydrogel samples in the swollen state are shown in Figure 1. These images suggest that the microstructures of both gels are similar. Most significantly, they both have similar sized pores of the order of 100 μm. This is much smaller than the expected pore sizes of superporous gels of between 300 and 1000 μm.[19,20] Although not shown here, SEM images were also collected of the gels in the dried state and the pore sizes were found to be unchanged. In addition, open pores

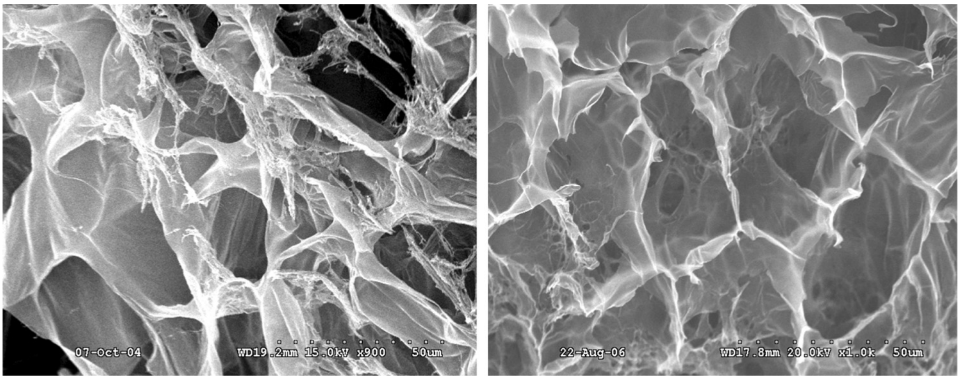

Figure 1. SEM images of swollen commercial and synthesized hydrogels showing their pore structure: (a) commercial hydrogel, and (b) low cross-linked synthesized hydrogel (2 g AAm, 3 mg MBAAm, and 20 mL water). Scale in the lower right of each picture indicates 50 μm. The swelling ratios of commercial and synthesized hydrogels are 326.2 and 36.6, respectively.

characterize superporous gels. While some open pores can be observed in the commercial hydrogel sample, most of the pores are closed ones. It should be noted that all the synthesized hydrogel samples we examined showed almost no open pores, such as the one shown in Figure 1.

Finally, superporous hydrogels are characterized by relatively rapid swelling kinetics, with hydrogel samples reaching their equilibrium swelling in several minutes to a few hours because of their large pores and open structure. However, the dried commercial hydrogel samples took over 9 h to reach their equilibrium swelling when immersed in DI water. This means that the commercial PAAm hydrogels do not have any of the typical characteristics of superporous hydrogels. This strongly suggests that these are primarily ionic hydrogels, such as the hydrolyzed PAAm hydrogels that were synthesized in this study. Strictly speaking, such ionic hydrogels should not be labeled as PAAm gels, which have quite different swelling properties, as we have shown.

Mechanical Property Measurements

The ability to absorb water many times their weight makes hydrogels susceptible to breakage when subjected to even small forces. Such breakage, in turn, can cause the gels to dehydrate quickly. This becomes an issue when such gels are placed in soils that may be compacted. For their successful deployment in soils it is necessary for the gels to possess sufficient mechanical stability; this was investigated by measuring the mechanical properties of the same commercial and non-hydrolyzed synthesized hydrogel samples shown in Figure 1.

The measured compressive strengths of these hydrogels are shown in Table IV. The results indicate that the compressive strength has an inverse relationship with the gel swelling ratio. The reduced swelling ratio of synthesized hydrogels was compensated for by their superior mechanical strength. In addition, the mechanical strengths of commercial hydrogel and synthesized hydrogel were expected from their microstructure. Figure 1 shows that the synthesized hydrogels have a more closed pore structure, which contributes to greater mechanical stability, compared with the partially open pore structures of the commercial hydrogel. These results suggest that it is necessary to strike a careful balance between the swelling property and the mechanical strength of the hydrogels to ensure optimum performance for horticultural applications.

Dehydration Rate Measurements

While swelling and mechanical strength are significant properties for hydrogels in soil media, it is the ability to slowly release moisture that is most important for

Table IV. Measured compressive strengths of commercial and synthesized hydrogel samples

	Gel composition	Compressive strength (N/cm^2)
Commercial hydrogel	Unknown	4.8
Synthesized hydrogel	2 g AAm, 3 mg MBAAm, and 20 mL water	13.0

their success. However, this property is difficult to estimate as it is depends strongly on many other factors, such as the temperature, humidity, hydrogel sample size, and the wicking ability of the soil. As a result, understanding the relationship between the dehydration rate of the hydrogel and these parameters will require a comprehensive analysis that is outside the scope of this study. Here we will focus on how the water retention properties of the commercial and synthesized hydrogels measure up under conditions similar to those expected for horticultural applications.

To ensure constant conditions the dehydration experiments were carried out in an incubator with a controlled temperature of 50°C and a controlled relative humidity of 37.5%. To simulate the wicking action of soils, the fully hydrated hydrogels were placed on filter paper for the dehydration measurements. A recent study confirmed that the wicking action of filter paper correlates well with that of soil.[21]

The resulting dehydration behavior for both gels is shown in Figure 2, and they clearly show similar behavior. It should be noted that unlike the controlled conditions of these experiments, in soils the extremely fragile commercial hydrogels are likely to break up easily. As a result, under those conditions the commercial hydrogels are likely to dehydrate faster than the synthesized ones. The more significant observation, however, is the rapidity of the dehydration rates for both gels. In less than 10 h both gels have lost almost all their available water. For purposes of reducing irrigation during crop production or minimizing postproduction crop losses in retail stores, this performance is quite inadequate. This is most likely the reason why these hydrogels delivered limited improvement for horticultural applications.

The microstructure of these hydrogels with their relatively large pore sizes is one of the likely causes of these high rates of dehydration. These large pores ensure that there is very little resistance to water transport across the gel volume. Another possible reason is the small sample sizes of the hydrogels used for these measurements. The higher surface-to-volume ratio of the smaller samples ensures that the effect of the wicking action will be stronger in these. Finally, as mentioned earlier,

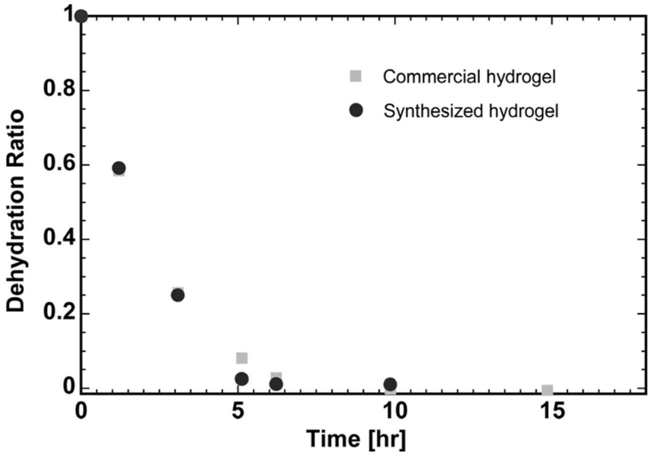

Figure 2. Variation of dehydration ratios with time for a commercial hydrogel and a synthesized hydrogel. The composition of the commercial hydrogel is unknown, while that of the synthesized hydrogel is 2 g AAm, 1 mg MBAAm, and 20 mL water.

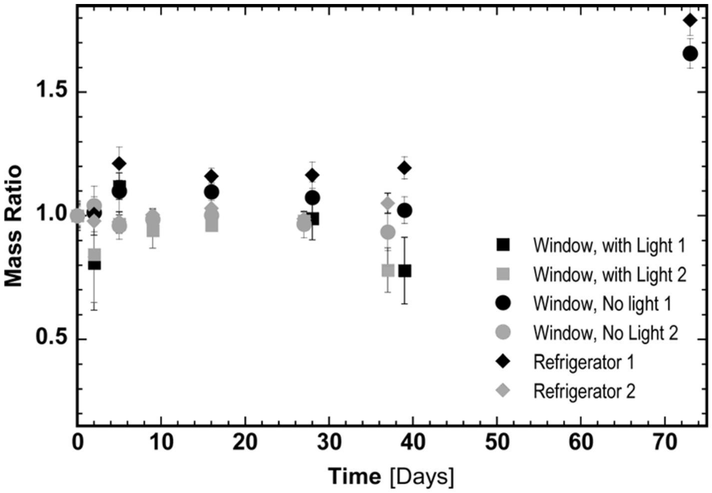

Figure 3. Variation of the mass ratio of different PAAm samples measured over several days, starting with a mass ratio of 1, subject to three different conditions. The samples from the two different runs are labeled 1 and 2.

the mechanical strength of these hydrogels has to be carefully balanced with their water-absorbing property. A stronger gel may have a reduced capacity to absorb water, but this could be compensated for by a reduced dehydration rate.

Polymer Degradation Measurements

The final tests that were carried out measured the degradation of the hydrogels. This was done to verify if poor performance of the hydrogels was due to their degradation over time. Samples of commercial PAAm dispersed in water, at an initial concentration of ~0.5 ppm, were subjected to conditions similar to those expected for growth media in a greenhouse: normal sunlight for hydrogel samples on the surface, darkness for buried samples, and dark, near-freezing conditions for samples kept in cold storage. Two separate runs were carried out for each of these conditions. Also, the concentration measurements were carried out over periods of up to 73 days to study the effect of a short growing season.

The results of the degradation measurements are shown in Figure 3. These are presented in terms of the mass ratio, which is the ratio of the concentration on a given day to the starting concentration of the polyacrylamide. While there is some variation in concentration between the samples there is no discernible trend that would suggest hydrogel degradation. The variations are most likely caused by non-uniformities in hydrogel distribution in the aliquots. This stability of polyacrylamide is an expected result and has been observed by other investigators as well.[22]

CONCLUSIONS

Commercial PAAm hydrogels commonly used for horticultural applications swell extremely well, much more than the PAAm hydrogels synthesized in this study.

This high swelling was also matched by their extremely fragility. This behavior could be due to the commercial hydrogels being superporous. However, density and SEM analysis showed that their microstructure was no different than that of the synthesized hydrogels, consisting mostly of closed micron-sized pores, with some open pores. When the synthesized hydrogels were modified to incorporate ionic groups their swelling behavior matched that of the commercial hydrogels. The gels also became very fragile, again matching the commercial hydrogels, as measured by their compressive strengths. This suggests that the commercial hydrogels are not pure PAAm hydrogels, but modified ionic ones.

The commercial and synthesized hydrogels also behaved similarly when subjected to controlled dehydration conditions similar to those encountered in plant growth in soils. However, both showed poor water retention abilities, losing most of their moisture within a few hours. This explains their poor performance in growth media and is likely caused by the small sizes of the hydrogels resulting from their extreme fragility and by the large pores in them. Degradation measurements confirmed that the poor performance was not due to the degradation of polyacrylamide. In order to improve the performance of these gels in growth media gels with improved properties need to be developed.

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