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Lignocellulosic biomass for the preparation of cellulose-based hydrogel and its use for optimizing water resources in agriculture

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ABSTRACT: Recently, efforts have been devoted to find ways in utilizing biomass as feedstocks for the production of organic chemicals. This is because of its abundance, renewability and worldwide distribution. Lignocellulosic agricultural waste materials are regarded as abundant, inexpensive, and readily available natural resources for both chemical and paper industries. Hydrogels are polymeric materials that vary in their origin and composition and can absorb large amount of water without dissolving. In our study, cellulose-based acrylic acid hydrogel was synthesized starting from rice straw as a source for the lignocellulosic material, where cellulose was first isolated after alkaline-acid pulping treatment followed by bleaching step with sodium hypochlorite resulting of 90.8% holocellulose. The cellulose-based acrylic acid hydrogel was synthesized applying heterogeneous reaction and shows a swelling ratio more than 3000%. The resulting hydrogel was further characterized with FT-IR and SEM. On the other hand, comparison between the rice straw-based hydrogel and the commercially available acrylamide hydrogel was studied for improving maize production in salt affected soil as well as in the growth promoters of maize under water stress. The experimental results demonstrated that the yield parameters were increased with increasing irrigation rates. Both types of hydrogels introduce positive and significant effect compared to the one without adding hydrogels. Also, acrylamide hydrogel was effective for improving almost yield parameters more than applying rice straw-based hydrogel. Generally, the addition of hydrogel increases the nutrient concentration, uptake, and both of water and nutrients use efficiency. © 2015 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2015**, *132*, 42652.

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INTRODUCTION

Because of increasing demand for fresh water supplies with the depletion of water sources, farmers are forced to use either efficient irrigation systems or considered deficit irrigation practices. Therefore, researchers, farmers, and governments should come together to study possible effect of reduced irrigation practices. El-Hendawy and Schmidhalter[1] reported that, every country in North Africa and Middle East are suffering from insufficient water supply for irrigation.

According to this, the interest in using superabsorbent hydrogels in agriculture is increased due to the need to reduce water consumption and optimize water resources in agriculture and horticulture, and thus play a role in changing the human habit and culture towards water to be treated as a benefit to save and not as an excess to waste.[2,3] The water is absorbed by the hydrogel during watering the cultivation, which then releases water and nutrients to the soil in a needed amounts that led to keep the soil humid over long periods of time. This process allows a high saving of water and a redistribution of the water resources available for cultivation in other applications. A further advantage in using hydrogels is related to their swelling effect on the soil, where they can absorb irrigation and rain water helping in reducing deep percolation by using gravitational water as well as capillary water.[4] Thus, researchers took great steps towards obtaining novel hydrogels, based on synthetic, natural or hybrid polymers, with high swelling properties and/or biocompatibility, and bioactivity.[5–7] Both natural and synthetic polymers and monomers are used for the preparation of hydrogels. Among all the natural polymers, cellulose is of special interest. Due to the large availability of cellulose in nature, the intrinsic degradability of cellulose, and the smart behavior displayed by some

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cellulose derivatives, cellulose-based hydrogels are extensively investigated.

Plant cell wall is built up of lignocelluloses, which is a complex material containing cellulose (linear glucose homopolymer), hemicellulose (branched sugar heteropolymer), and lignin (three-dimensional crosslinked phenylpropanoid). The efficient utilization of lignocelluloses requires the fractionation and separate utilization of these components to produce valuable products. Agricultural waste, which is a source of the lignocellulosic material, is a promising alternative for nutrient recycling. The critical factor is not only to increase crop yields but also to sustain long-term productivity. This can be made through the use of renewable resources easily and cheaply available on the farm. Rice is an important crop in many areas of the world that yields a large amount of rice straw residue,[8] which is produced in large quantities as an agricultural by-product. Rice straw is a lignocellulosic material that contains about 77% holocellulose which can be further utilized to produce several useful compounds.[9,10]

Many research papers have shown an interest in hydrogels, but not many farmers have shown an interest in using hydrogels in their farms. Hydrogels were developed to increase water holding capacity and have been used to aid plant establishment and growth in dry soils. Jhurry[11] has mentioned that crosslinked polyacrylamides hold up to 400 times their weight in water and release 95% of the water retained within the granule to growing plants. Thus, one of the objectives of this study was to synthesis cellulose-based hydrogel using rice straw as the starting lignocellulosic material and the second objective was to compare between the synthesized hydrogel with the commercially available acrylamide based hydrogel as a growth promoters of maize production under water stress, as well as the study of the reflection of these materials on nutrient uptake and water use efficiency.

EXPERIMENTAL

Materials and Methods

Acrylic acid (AA) was purchased by Acros. *N,N*-methylenebisacrylamide (MBA) used as crosslinking agent of analytical purity was purchased from Fluka and potassium persulfate (KPS) used as initiator was purchased from s.d. Fine-Chem. Cellulose was laboratory isolated from rice straw after applying chemical pulping and bleaching method. Commercially acrylamide hydrogel (HA) was kindly supplied by Prof. Dr. Omar El-Hady, Soil and Water Utilization Department, National Research Center, Egypt. The HA was purchased from Badische Anilin-und Soda-Fabrik (BASF).

Isolation of Cellulose

Chemical Pulping. Alkaline–acid pulping was applied as chemical pulping method for the rice straw in which two acids, namely sulfuric and acetic acids were used as the acid source after using sodium hydroxide (NaOH) in the first stage. Briefly, 100 g of rice straw was pulped with 5% NaOH (wt/wt) related to the biomass in liquor to fiber ratio of 10 : 1. The fibers were then cooked at 170°C for 2 h. The pressure was released and the pulped fiber was subjected for washing with water till neutrality then moisture content was determined and the pulp was remarked as R_A. The resulting pulp, R_A, was further applied for acid pulping, where sulfuric acid (H_2SO_4) was used in a percent of 5% (wt/wt) to the pulped raw material, and liquor to fiber ratio was 10 : 1. The fibers were then cooked at 170°C for 2 h. At the end, the pressure was released to atmosphere and the pulped fiber was subjected for washing with water till neutrality then left to dry in air. The pulp from both treatments was marked as R_{ACH}.

Another alkaline–acid pulping process was carried out where 10% (wt/wt) NaOH was used in the first stage and 10% (wt/wt) acetic acid for the second stage. The process was carried out as mentioned above. At the end, the pressure was released to atmosphere and the pulped fiber was washed with water till neutrality then air dried and was marked as R_{ACA}.

Bleaching. Bleaching was carried out for the treated lignocellulosic fibers, R_{ACH} and R_{ACA}, by the hypochlorite bleaching method. The treated lignocellulosic fibers were bleached as described by Ibrahim and El-Zawawy[12] using sodium hypochlorite solution that equivalent to 60% of the chlorine requirement for 2 h at 40°C. The liquor to fiber ratio was 10 : 1 and the pH was maintained at pH 9.0 during the bleaching process.[12] The bleached cellulosic fibers, i.e., bleached R_{ACH} and R_{ACA}, were washed with water till neutrality then air dried.

Compositional Analysis of Isolated Cellulose

The chemical composition of the isolated fibers was determined according to the following methods : holocellulose and α-cellulose (TAPPI T257 om-85), Klason lignin (TAPPI T222 om-88) and ash content (TAPPI om-85).

Hydrogel Preparation

Cellulose hydrogel was synthesized using bleached cellulosic fibers resulting from rice straw in heterogeneous reaction with acrylic acid.

Heterogeneous Reaction. ***Activation of cellulose fibers.*** The powdered cellulosic fiber was weighed and dipped in 2000 mL beaker with sodium hydroxide solution of 20% for activation. The aqueous suspension was heated at 80°C and stirred for 4.5 h. After the desired time, the aqueous suspension was filtered and washed with ethanol solution of 95%. After that, the activated cellulose fibers were air dried and stored for further reaction.

Cellulose-g-poly(acrylic acid) super-absorbent hydrogel preparation. A series of samples of activated cellulose, *N,N*-methylenebisacrylamide (MBA), potassium per sulfate (KPS), and neutralized acrylic acid (AA) were prepared by the following procedure. Appropriate amount of activated cellulose (0.5 g) was immersed in 30 mL distilled water in a 500 mL three-neck flask, equipped with a mechanical stirrer, a reflux condenser and a funnel. After being purged with nitrogen for 30 min to remove the oxygen, certain amount of KPS was introduced in the mixture to initiate the activated cellulose to generate radicals, and a solution of 5.00 g AA after neutralization, certain amount of MBA and 10 mL distilled water was added. The water bath was kept at 70°C for 3 h to complete polymerization.

The resulting product was dried to a constant weight at 70°C till no weight variation was observed.

Water Absorbency Measurement. A weighted quantity of the super-absorbent hydrogel, i.e., 1 g, was immersed in distilled water, i.e. 100 mL, at the room temperature to swell equilibrium. When the super-absorbent hydrogel reached water saturation, the swollen samples were separated from the unabsorbed water by filtration over a screen to allow the separation of the excess water, i.e., the water that had not been absorbed.[7] The water absorbency (Q_{H_2O}) of the prepared super-absorbent hydrogel was determined by weighing the swollen samples and was calculated as grams of water per gram of sample (g/g) using the following equation:

$$Q_{H_2O}=\frac{w_2-w_1}{w_1} \quad (1)$$

where w_1 and w_2 were the weights of the dry sample and the water-swelling sample (g), respectively.

Structural Analysis. The FTIR spectra of samples were taken in KBr pellets using JASCO FT/IR 6100 Instrument in the range of 4000–500 cm^{-1}. The surface morphology of the gels were examined by scanning electron microscopy (SEM) using a JEOL JXA-840A electron microprobe analyzer (JOEL USA, Peabody, MA), on aluminum stubs and coated with a thin layer of palladium gold alloy.

Field Experiment

Field experiment was carried out in Ismailia governorate (Latitude: 30° 35 Longitude: 32° 16 Elevation: 11.2) to compare between commercially available hydrogel; namely, acrylamide hydrogel (HA), and cellulose-acrylic acid hydrogel, prepared from rice straw, which named as rice straw-based hydrogel (HS), as growth promoters of maize plants (*Zea mays* L. Single cross 129 white) under different irrigation levels and frequencies. Whereas field affected with salinity (7.5 dS/m) was parted into two main plots separated by 2 m and contained two irrigation frequency (F_1 = irrigation every second day or short frequency and F_2 = irrigation every 3 days or long frequency). The number of irrigations varied from 35 for the lowest frequency (F_2) treatment to 53 for the highest frequency treatment (F_1). Each main plot was divided into three subplots, where they consisted of three levels of irrigation water ($I_1 = 100$, $I_2 = 85$, and $I_3 = 70$% of irrigation water requirements). Every subplot was divided into three subsubplots containing (i) no hydrogel (H_0), which means without any addition of hydrogel, (ii) HS treatment, which means with the addition of 2 g of rice straw-based hydrogel into plant hole, and (iii) HA, which means with the addition of 2 g of acrylamide-based hydrogel into plant hole. The hydrogel was then mixed, in the dry form, to the soil in the area close to the plant roots then the soil sample was air dried, crushed and sieved to pass through a 2-mm sieve.

Ammonium sulphate, with nitrogen content (N) of 20.6%, was added at a rate of 120 kg N/fed in three equal portions, i.e., before cultivation, after two weeks from cultivation and after 3 weeks from the second addition. Super-phosphate and potassium sulfate, with 15.5% P_2O_5 and 48% K_2O, respectively, were added, as mentioned in a method described in a previous field experiment,[13] before plantation at a rate of 200 and 50 kg/fed, respectively. The field was leveled to facilitate uniform distribution of the applied irrigation water and the fertilizer.[13] Plants were irrigated by drip irrigation system and the crop evapotranspiration (ETc) was calculated according to Food and Agriculture Organization of the United Nations (FAO-56) by the following formula:[13,14]

$$ET_c=K_c\times ET_0 \quad (2)$$

where ET_c = crop evapotranspiration in mm, ET_0 = potential evapotranspiration in mm/day, and K_c = crop coefficient.

The data of the water requirement was calculated by an average of 8 years of meteorological parameters using CROPWAT computer model (FAO, 1992), based on the calculation using Penman Monteith equation and the K_c values illustrated in FAO-56.[13,14] The calculated quantities of I_1, I_2, and I_3 treatments were shown to be 3202, 2722, and 2241 m^3/fed.

The Irrigation water use efficiency, IWUE (kg/m^3), was calculated for each treatment according to eq. 3,[1,15–17] where it is expressed in gross weight of product (kg) per water supplied (m^3).[13]

$$\text{IWUE}=\frac{\text{Grains, ears or stover yield(kg/fed.)}}{\text{Total water applied }(m^3/\text{fed.})} \quad (3)$$

Measurements

The percent of the coarse sand, fine sand, silt, clay, electrical conductivity of the soil (EC), as well as the pH and both soluble cations and anions were analyzed.[18,19] Abou-Baker *et al.*[13] mentioned that fertilization with nitrogen, phosphorus, and potassium were carried out according to Ministry of Agriculture recommendations. On the other hand, IWUE, ear yield (kg/fed), grain yield (kg/fed), biological yield (kg/fed), and root length (cm) were calculated at the end of the season.

Moreover, nitrogen, phosphorus, and potassium were analyzed by Kjeldahl, ascorbic acid, and flame-photometery methods, respectively[20] after digesting a portion of a dried grain as described by Chapman and Pratt (1978).[21]

Furthermore, the total nutrients in percent, i.e., phosphorus use efficiency (PUE), nitrogen use efficiency (NUE), and potassium use efficiency (KUE) were calculated as described by Malhi *et al.* (2001)[22] according to the following equation:

$$\text{Total nutrients (PUE or NUE or KUE)} =\frac{(\text{DMY in } H \text{ treated soil} - \text{DMY in control } H_0)}{\text{Rate of applied P or N or K (kg P or N or K/fed.)}} \quad (4)$$

The experimental design was Split–Split plot (SSP) in three replicates. According to Abou-Baker *et al.*, (2012)[13] the data were statistically analyzed through analysis of variance (ANOVA) and least significant difference (LSD) at 0.05 probability level was applied as described in Gomez and Gomez (1984).[23]

Also, the swelling ratio for the hydrogel was measured by weighing samples before and after their immersion in distilled water for about 24 h. The swelling ratio (SR) is calculated according to the following equation:[24]

$$\text{SR}=(W_s-W_d)/W_d \quad (5)$$

where W_s is the weight of the swollen hydrogel and W_d is the weight of the dried sample.

RESULTS AND DISSECTION

Fiber Composition

Cellulose is the most important renewable natural resource on earth as it is a long chain of 1→4-linked anhydro-D-glucose molecules that gives wood its remarkable strength. It is the main component of plant cell walls, and the basic building block for many textiles, paper and for many industrial purposes. The purpose of the pretreatment is primarily to open up the structure of the material to facilitate access to the cellulose structure, where a prerequisite to the cellulose reaction is the release of the cellulose portion from the tightly woven lignocellulosic structure. For this, different pretreatment process was carried out with a variety of methods, as mentioned in the experimental part. The lignocellulosic raw material used in this study, i.e. rice straw contains holocellulose of 68.09% and lignin of 14.55%.

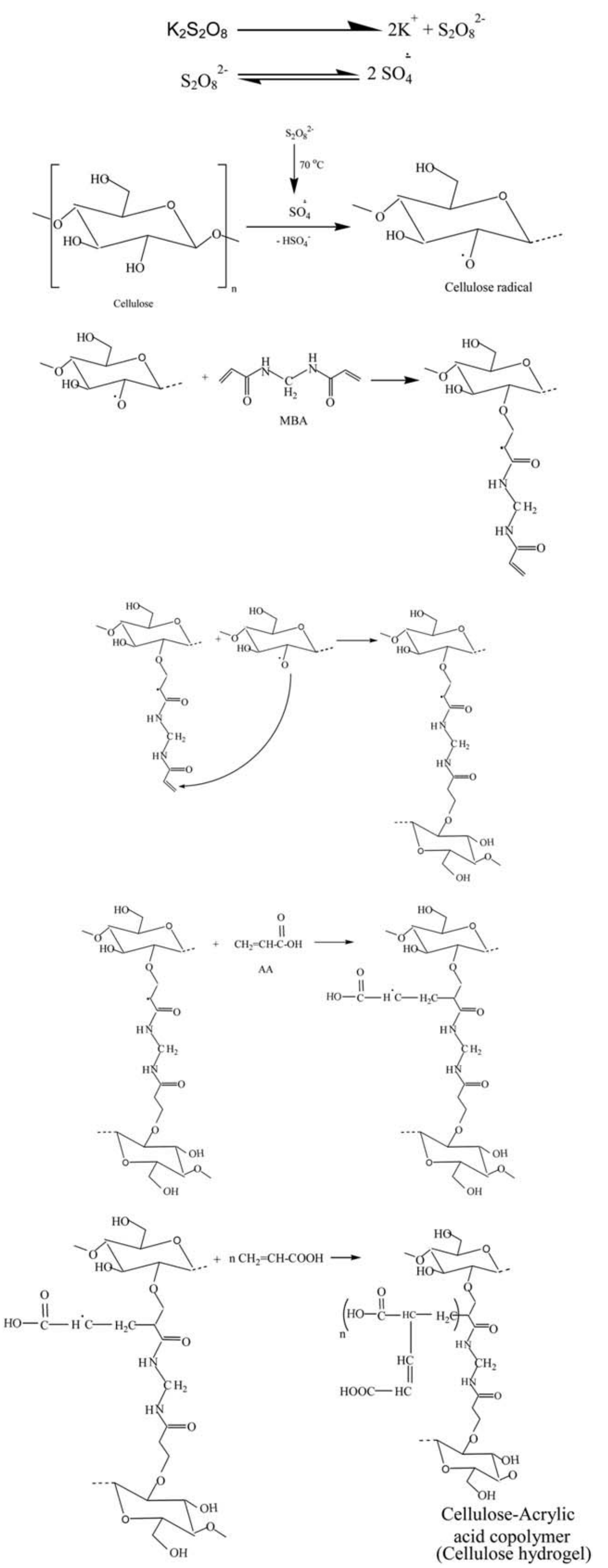
The selection of the pretreatment method was to be compatible with the need of our objective. The pretreatment was made to delignify the raw material and to produce cellulosic fiber. The effect of the pretreatment on the composition of the biomass, rice straw, indicated that the holocellulose for the alkaline–acid pulping in case of using sulfuric acid (R_{ACH}) is higher than those of using acetic acid (R_{ACA}), 90.8 and 87.65%, respectively, and the lignin content was 5.09% for R_{ACH} compared to 7.14% for R_{ACA}.

Since the objective of our current project is to produce cellulosic fiber, thus bleaching was carried out for the pretreated rice straw in order to remove the lignin and thus increase the cellulose content, where bleaching is another treated process for the removal of the lignin and it is necessary in case of the need of cellulose material. Thus, the results for the bleached rice straw after the pretreatment process show an α-cellulose, which is the pure cellulose present in the sample, of 64.70% in case of the bleached R_{ACH} and 65.74% in case of the bleached R_{ACA}. Also, it was noticed that the lignin content decreased during bleaching to reach 2.22% in case of the bleached R_{ACH} and 2.12% in case of the bleached R_{ACA}. This means that the higher percent reached for the α-cellulose and lower percent for the lignin is needed for our study to prepare cellulose hydrogel.

Cellulose Hydrogel Preparation

In the current study, cellulose hydrogel was prepared to be used as superabsorbent polymer gel (SAPs), where they can absorb a large amount of water, considerably more than their dry mass. Pretreated bleached rice straw was used as the starting cellulosic material. Activation with sodium hydroxide was carried out first for the cellulosic material. After that the cellulosic material was grafted with acrylic acid in heterogenous reaction as described in the experimental part via free-radical polymerization initiated by potassium persulfate (KPS).

The mechanism of grafting acrylic acid onto cellulose using potassium persulfate as initiator is represented in the Scheme 1, where first the persulfate initiator is decomposed under heating to generate sulfate anion radical. After that the radical abstracts hydrogen from the hydroxyl group of the cellulose to form alkoxy radicals on the substrate. According to that this persulfate-saccharide redox system results in an active center on



Scheme 1. A brief proposed mechanism of grafting of cellulose with acrylic acid via for KPS initiator.

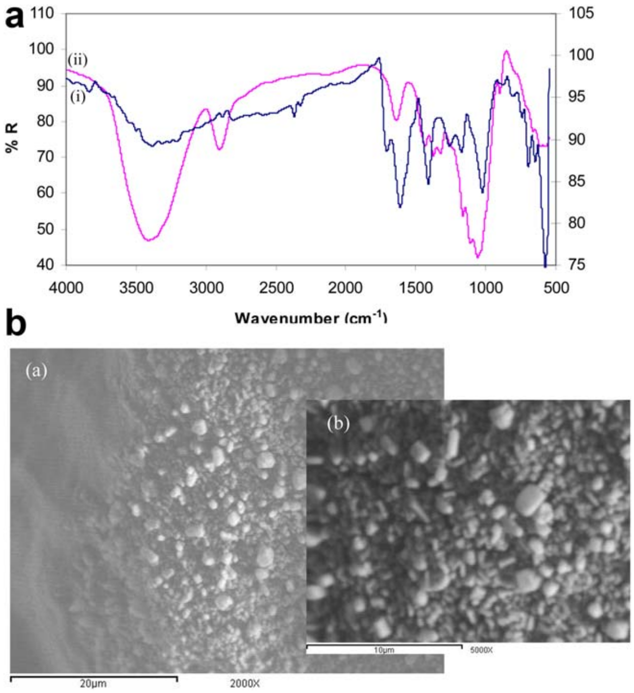


Figure 1. (a) FT-IR for (i) cellulosic material from bleached rice straw unactivated and (ii) cellulose grafted with acrylic acid in heterogenous reaction, i.e., cellulose-g-acrylic acid hydrogel. (b) SEM for cellulose-g-acrylic acid resulting from grafted bleached rice straw in heterogenous reaction. (a) ×2000 and (b) ×5000. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

the substrate that radically initiate polymerization of acrylic acid crosslinked by MBA via free-radical polymerization led to a graft copolymer to obtain the hydrogel.

$$K_2S_2O_8 \rightarrow 2K^+ + S_2O_8{}^{2-}$$

$$S_2O_8{}^{2-} \Longleftrightarrow 2SO_4$$

The FT-IR was studied and illustrated in Figure 1(a). Compared with the cellulosic materials and cellulose-g-acrylic acid hydrogel, Figure 1(a), new absorption bands at $\sim$ 2300 cm^{-1}, 1720 and 1524 cm^{-1}, 1420 and 1229 cm^{-1} were observed represents a bands for amide group stretch, amide group characteristic absorption, methylene shear vibration in both carbonyl and methylene and carboxyl in ethers, respectively. According to Liu *et al.*, (2009)[25] this indicate that the acrylic acid has been grafted on the cellulose chain of the copolymer net work and thus we can conclude that the grafting copolymerization between cellulosic material and acrylic acid monomers has taken place during the reaction with potassium persulfate as the initiator and *N,N′*-methylenebisacrylamide as the cross-linker. On the other hand, the SEM microscope for the cellulose-g-acrylic acid hydrogel, Figure 1(b), shows a surface structure with droplets.

Swelling Properties of the Hydrogels

The influence of the cellulosic material on the swelling behaviour of cellulose-g-acrylic acid hydrogel in distilled water at 25°C was studied. A higher swelling ratio was noticed and the maximum swelling ratio was more than 3000%. The photos of the cellulose-g-acrylic acid hydrogel are shown in Figure 2(a). The appearances of the hydrogels were transparent and the swollen hydrogel was with large shrinkage. To have a high swelling ratio is important for biodegradable materials for wide application.

Furthermore, Figure 2(b,c) show the SEM images of the cross-section of the freeze-dried hydrogel samples. The cross-sections of the samples exhibited macropores architecture. Moreover, it can be seen that the size of pores are increased, leading to a more open and loose structure. According to Chang *et al.* (2010)[26] this suggest that an electrostatic repulsion occurred causing enlargement of the space in the networks of hydrogels. Interestingly, the pore size of the hydrogels was very large, therefore, the numerous water molecules could easily diffuse into hydrogels to form the large pores, leading to the higher swelling ratio.

Field Experiments

The study was carried out at Ismailia Governorate, where the sandy soil was first characterized for its physical and chemical properties as well as the properties of the hydrogel under investigation and the results were gathered in Tables I and II. Moreover, the water requirements for drip irrigated bean grown were illustrated in Table III for the period of the study from May to September.

Ear Yield. From the statistical analyses of the measured ear yield one can noticed that there was no significant effect of irrigation frequency (F) on ear yield, but it affected significantly by irrigation rate (I), hydrogel (H) application, and the interaction between them [Table IV]. It was noticed that reduction in irrigation water amount lowers ear yield, although, the addition of both HS and HA increases it by 18.6 and 22.6% compared to the control (H_0), respectively.

The second interaction between I and F treatments was significant, in contrast of most growth parameters. Reducing irrigation frequency from F_1 to F_2 with adding high irrigation rate ($I_1 \times F_2$) produces significant increase in ear yield compared to ($I_1 \times F_1$), under the specific condition of the study area. This may be referred to the frequent applications, i.e. one every 2 days (F_1), which led to the remaining of water near the soil surface to subsequently evaporate. But the decrease in frequency, i.e., one every 3 days (F_2), causes an increase of water stored in root zone. This result was in agreement with a conclusion to Mermoud *et al.* (2005)[27] Lowering irrigation rate from I_1 to I_2 and I_3 puts the plants under high stress, so it affects negatively with decreasing frequency.

As for the interaction between H and I, it was also significant, where no significant different was between HA $\times$ I_1, HA $\times$ I_2, and HA $\times$ I_3. This result means that using HA could decrease the irrigation rate to 85 or 70% of maize water requirements, and it was noticed that there are high significant difference between (HS $\times$ I_1), (HS $\times$ I_2), and (HS $\times$ I_3), whereas ear yield decreases with decreasing irrigation rate, significantly.

Concerning the second interaction between H and F, it was significant and the treatments were arranged as follows: HA $\times$ $F_1 >$ HA $\times$ F_2 for acrylamide hydrogel and HS $\times$ $F_2 >$ HS $\times$ F_1

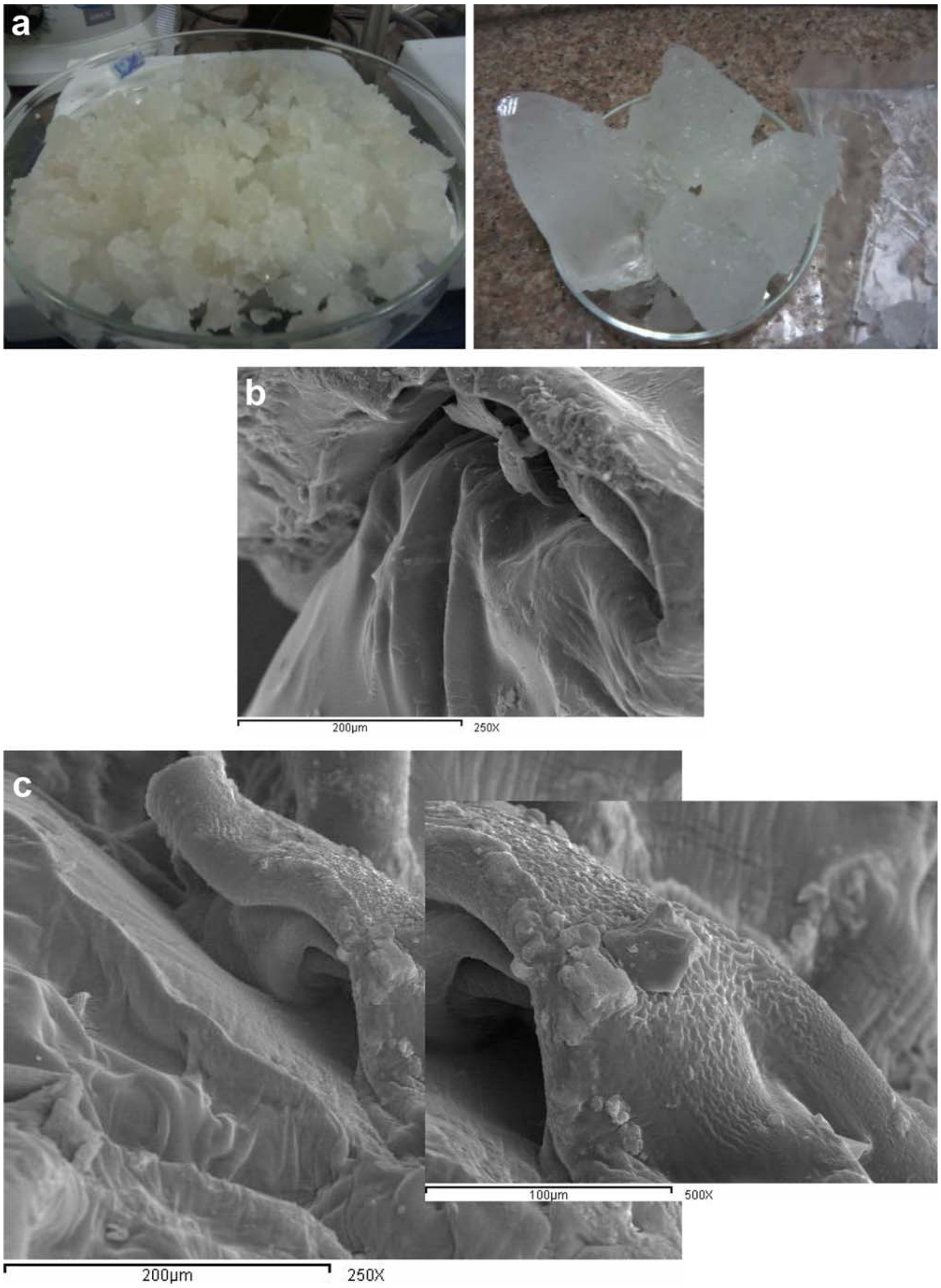


Figure 2. Photographs of cellulose-g-acrylic acid hydrogel (a) before and (b) after swelling in distilled water. (b) SEM for cross-section of cellulose-g-acrylic acid hydrogel. (c) SEM for cross-section of cellulose-g-acrylic acid hydrogel after swelling. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

for rice straw-based hydrogel. This data means that, HA absorbs high amount of water but it losses water quickly. In contrast, HS absorbs small amounts of water and reserves it causing slow release of water. On the other hand, the third interaction effect between *F*, *I*, and *H* was not significant.

Biological Yield (kg/fed). Ekebafe *et al.* (2013)[28] mentioned that the okra yield, plant height, stem diameter, leaf area, biomass accumulation and relative water content as well as protein and sugar contents in the fruits increased significantly by the addition of the hydrogel. The data listed in Table IV demonstrated that the biological yield of maize plants increases significantly by increasing irrigation rate and hydrogel application, while no significant effect was noticed for both frequency effect and the second interaction, i.e., $I \times F$. On the other hand, F_1, I_1, and HA were noticed to give the highest biological yield.

As shown in $H \times I$ data, the biological yield increases by increasing the irrigation rate under H_0, and the I_2 treatment produces higher biological yield compared to the I_1 with the

Table I. Physical and Chemical Properties of the Studied Soils

Characteristics	Value
pH (soil : water ratio is 1 : 2.5)	8.1
EC (soil paste extraction) d Sm^{-1}	7.5
Soluble cations (m.e./100 g soil):	
Calcium	13.6
Magnesium	7.5
Potassium	0.9
Sodium	45.5
Soluble anions (m.e./100 g soil):	
Carbonate	–
Bicarbonate	3.2
Chloride	65.5
Sulphate	6.3
Physical properties (%):	
Coarse sand	33.6
Fine sand	39.9
Silt	18.7
Clay	8.5
Textural class	Sandy/loam

Table II. Properties of the Studied Sources of Hydrogel

	Total nutrients (%)					
Hydrogel	N	P_2O_5	K_2O	EC (dS/m) 1 : 500	pH	SR
HA	11.76	0.02	4.80	0.317	7.1	247.6
HS	1.94	0.06	8.64	0.326	5.7	215.0

addition of hydrogels, irrespective of its source. On the other hand, the treatments which received hydrogel are arranged as follows: HA $\times$ I_2 > HA $\times$ I_1 > HS $\times$ I_2 > HS $\times$ I_1. In another mean, the hydrogel application reserves about 15% of maize water requirement.

As for $H \times F$ interaction, the addition of HA with irrigation every 2 days (F_1) increases the biological yield, while using of HS increases its biological yield with irrigation every 3 days (F_2). This finding may support the previous claim where HS releases water slowly.

Root Length (cm). The data of the root length as affected by *I*, *F*, and *H* treatments was gathered in Table IV and showed that the short frequency period (F_1) increases the root length compared to the long frequency period (F_2) without significant deference between them. Raising irrigation rates from I_3 to I_2 and I_1 increases the root length significantly, but there was no significant difference between I_1 and I_2 [Table IV]. Opena[29] reported that the irrigation significantly increases the root length density of potato plants in the second season and without any difference in the first season. By the application of HA, the maize root length was higher than that in case of using HS. Under short frequency (F_1), the root length increases with the decreasing of the irrigation rate and the addition of HA and HS. In contrast, the root length increases with the increasing of the amount of water in case of the control treatment (H_0).

Grain Yield (kg/fed.). The effect of studied factors, i.e., *F*, *I*, and *H*, and their interactions, i.e., $I \times F$, $H \times F$, $H \times I$, and $H \times I \times F$, on maize grain yield were significant as they can be seen in Figure 3. The results revealed that, short frequency irrigation (F_1) contributed for the enhancement of maize grain yield. This may be referred to: (1) the properties of sandy soils such as high infiltration rate, low water holding capacity, cumulative evaporation, weak structure, and high permeability;[30] and (2) under F_2 treatment, plants are suffering from higher water stress than F_1 treatment. As irrigation interval increased (F_2), bell paper yield and taro offshoots were decreased.[17,31] The maize grain yield was increased in the ranking of $I_1 > I_2 > I_3$, respectively. Thus, the improvement of grain yield with applying high water rate could possibly due to: (1) the solubilization effect of high water rate upon soil nutrient; (2) the amount of water is more suitable to exporting produced compounds to grains resulting in more grain filling;[32] (3) the maize is very sensitive to water stress as reported by El-Hendawy and Schmidhalter (2010)[1]; and (4) the high salinity of the studied soil (EC = 7.5 dS/m). The average maize grain yield was obtained when 100% of irrigation water requirement was applied (2992 m^3/fed) compared to 85 or 70% of irrigation water requirement (2918 or 2897 m^3/fed), irrespective of frequency and hydrogel treatments. It was reported by Dogan *et al.* (2007)[33] that, under semiarid climatic conditions any

Table III. Water Requirements for Drip Irrigated Bean Grown at Ismailia Governorate

Month	May		June	July						August			Sept.
Period	15–31		1–30	1–31						1–31			1–12
ET_0 (mm/day)	5.87		6.25	6.05						6.32			5.21
K_c		0.53			0.88			1.09				0.72	
ETc (mm/day)	3.11		3.31	5.5		5.32	6.60		6.89		4.55		3.75
Eu							90%						
L_r							10%						
IR m^3/season/fed. (I_1)		573.38			858.31			1054.83				715.05	

ET_0 = reference evapotranspiration, K_c = crop coefficient, Eu = application uniformity, L_r = leaching requirements; IR = irrigation requirements, I_1 = 100% of water requirements (3202 m^3/fed.), I_2 = 85% of water requirements (2722 m^3/fed.), I_3 = 70% of water requirements (2241 m^3/fed.).

Table IV. (a) Ear Yield (kg/fed.), (b) Biological Yield (kg/fed.), and (c) Root Length (cm) as Affected by Irrigation Frequency, Rate, and Hydrogel Application

		Hydrogel Source			
Freq.	Irr. Rate	H_0	HS	HA	Mean
(a)					
F_1	I_1	3316	3744	3876	3645
	I_2	3110	3734	3911	3585
	I_3	3064	3699	3917	3560
	Mean	3163	3726	3901	3597
F_2	I_1	3325	3851	3876	3684
	I_2	3088	3739	3846	3558
	I_3	3056	3714	3825	3532
	Mean	3156	3768	3849	3591
Mean	I_1	3321	3798	3876	3665
	I_2	3099	3737	3879	3571
	I_3	3060	3707	3871	3546
	Mean	3160	3747	3875	
$LSD_{0.05}$		F = ns I = 19 H = 16 I × F = 27 H × I = 27 H × F = 22 H× I × F = ns			
(b)					
F_1	I_1	5839	6284	6542	6222
	I_2	5207	6343	6609	6053
	I_3	5091	6321	6615	6009
	Mean	5379	6316	6589	6095
F_2	I_1	5676	6365	6593	6211
	I_2	5150	6380	6531	6020
	I_3	5073	6317	6454	5948
	Mean	5300	6354	6526	6060
Mean	I_1	5758	6325	6568	6217
	I_2	5179	6362	6570	6037
	I_3	5082	6319	6535	5978
	Mean	5339	6335	6557	
$LSD_{0.05}$		F = ns I = 26 H = 24 I × F = ns H × I = 41 H × F = 34 H × I × F = 58			
(c)					
F_1	I_1	20.0	21.7	23.0	21.6
	I_2	19.2	21.7	24.3	21.7
	I_3	13.6	24.3	24.7	20.9
	Mean	17.6	22.6	24.0	21.4
F_2	I_1	18.1	23.3	24.0	21.8
	I_2	19.5	22.7	24.3	22.2
	I_3	13.3	18.0	22.3	17.9
	Mean	17.0	21.3	23.5	20.6
Mean	I_1	19.1	22.5	23.5	21.7
	I_2	19.3	22.2	24.3	21.9
	I_3	13.5	21.2	23.5	19.4
	Mean	17.3	22.0	23.8	
$LSD_{0.05}$		F = ns I = 0.51 H = 0.52 I × F = 0.72 H × I = 0.90 H × F = ns H × I × F = 1.27			

reduction in irrigation amount would result in small plant height, less biomass and especially reduced soybean yield. It was noticed that the most increase in grain yield was obtained by HA application followed by HS. This could be due to the high swelling ratio of the HA, 247.6, compared to that for the HS, 215.0. The addition of the hydrogels decreases the differences between treatments and the decline caused by water stress became smoother.

On the other hand, there is a high depression in grain yield values under control treatments (H_0), which may be refer to the studied soil that is affected with salinity and hydrogel and have great potential for use in alleviating salinity stress on plant growth and growth parameter by reducing soil electricity, conductivity and electrolyte leakage of plant.[34]

The suitable matching between irrigation rate and frequency is $I_1 \times F_1$, which helps to achieve maximum grain yield by exerting positive effects on water balance, especially in root zone. In contrast, the combination between $I_1 \times F_2$ (high irrigation rate with low irrigation frequency) can cause water stress. This may be due to the amount of water applied at each irrigation event which can be higher and possibly excessive than the soil–water storage capacity and thus increases the amount of water and nutrients that moves below the root zone.[1] Generally, HA > HS and $F_1 > F_2$, while the combination between H and F revealed that HA $\times F_1 >$ HA $\times F_2$, as the same in case of the control treatment $H_0 \times F_1 > H_0 \times F_2$ and in opposite for using HS, i.e., HS $\times F_2 >$ HS $\times F_1$. This could refer to the effect of the rice straw-based hydrogel, i.e., HS has an effective slow release water material especially under stress condition.

As for the third interaction between F, I, and H, the highest grain yield was obtained by $F_1 \times I_2 \times$ HA, 3198 kg/fed, followed by $F_1 \times I_1 \times$ HA, 3185 kg/fed. The application of HA under short irrigation frequency shows lower yield in case of I_1 treatment compared to that in I_2 treatment. This may be referred to the higher moisture in the treated soil with HA over plant needs and increasing soil microporosity on the expense of

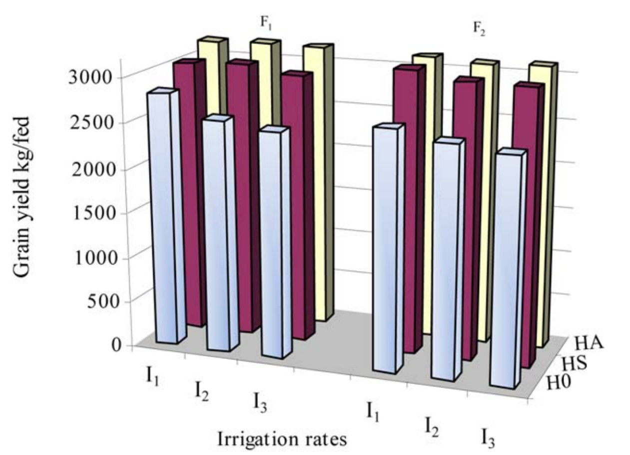


Figure 3. Grain yield (kg/fed.) as affected by irrigation frequency, rate, and hydrogel application. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

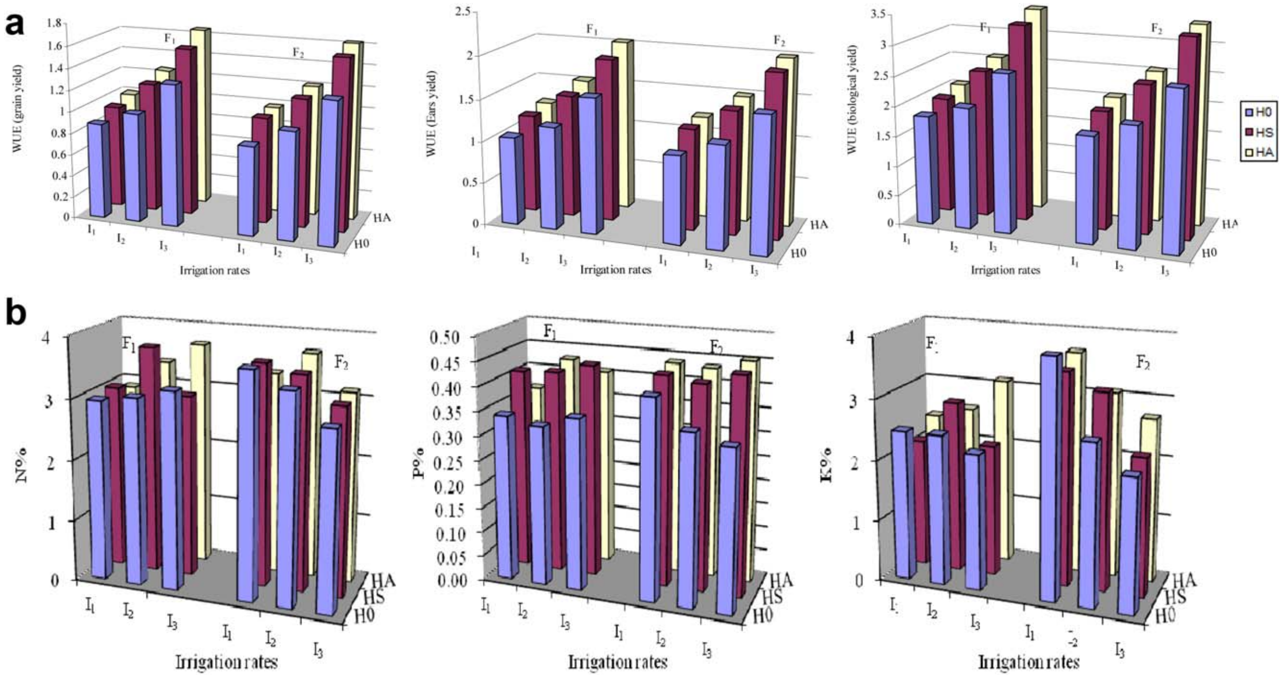


Figure 4. (a) Irrigation water use efficiency calculated by grains, ears and biological yield as affected by irrigation frequency, rate, and hydrogel application. (b) Nitrogen, phosphorus, and potassium concentrations as affected by irrigation frequency, rate, and hydrogel application. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

its macro-ones and in turn adverse effect on aeration of the root zone as discussed by El-Hady *et al.* (2001).[35]

Generally, we can conclude that almost 15% of irrigation water can be saved if farmers follow these practices, as well as, incorporation of 2 g hydrogel in squash plant pit, i.e., 20 kg/fed, that reduces the amount of irrigation water by 15%, which agreed with El-dewiny (2001)[36] results.

Irrigation Water Use Efficiency (IWUE). Optimizing water is a major challenge for improving crop productivity and maximizing water use efficiency. The IWUE is defined as biomass accumulation, i.e., grains, ears or biological yield, over irrigation water applied. It is also considered one of the parameters that used to evaluate the performance of agricultural production systems. Hassanli *et al.* (2010)[37] reported that IWUE can be increased by practicing deficit irrigation, improving irrigation technology, irrigation scheduling, and agronomic practices which led to yield increase. Figure 4(a) illustrated the values of IWUE which were calculated by grain, ears and biological yield. It was noticed that higher irrigation water use efficiency was recorded by F_1 followed by F_2 without significant difference between them. Sezen *et al.* (2006)[17] concluded that IWUE values decreases with increasing irrigation interval. Values of IWUE were greater at lowest rate and decreases by increasing irrigation amount. Under sandy soil condition, application of the hydrogel led to better water usage and less water losses. The three ways of IWUE calculated took the same trend, but it differs in values. One meter cubic of water can produce from 0.82 to 1.66 kg grains, 1.037 to 2.037 kg ears and 1.773 to 3.443 kg biological yield, respectively, which agree with Zwart and Bastiaanssen, (2004)[38] where they reported that maize crop water productivity values were measured ranging from 0.22 to a maximum of 3.99 kg m^{-3}.

A statistical analysis of the experimental treatments showed that IWUE was significantly affected by the addition of both hydrogel sources, i.e., HA and HS. Application of HA produces IWUE higher than those obtained by HS. These results emphasized that low yields, due to water stress, did not concomitant to low WUE values, and the increase in WUE did not refer to suitable or high water amount. This may be due to mathematically WUE calculated as yield (kg/fed)/total water applied (m^3/fed), hence increasing water amount tend to raise the denominator of equation and subsequently decreases the net result. Abou-Baker *et al.* (2012)[13] mentioned that, as for the viewpoint of the plant nutrition, the plant responses to first application unit, water or fertilizer, is higher than that after adding second unit. These findings were consistent with the results of Zhang and Yang (2005)[39] where they found that plants growing under water limited conditions have a higher WUE. It has been predicted that plants, generally, have the capability to optimize their water use in short term and maximize their chance of survival during drought in the long term.

Macronutrient Concentrations and Contents. Nitrogen, phosphorus, and potassium concentrations in maize grains, as affected by irrigation frequency, irrigation rate, and two different sources of hydrogel, were illustrated in Figure 4(b). In contradicted line of those obtained in yield data and its parameters, long intervals between irrigations (F_2) produces higher N, P, and K concentrations than short frequencies with significant differences in K concentration and without significant differences in N and P concentrations. Nitrogen concentration of maize grain decreases significantly by the order $I_2 > I_1 > I_3$, where this may be due to that I_2 is more suitable to protect nitrogen from leaching by the time.[32] Phosphorus concentration was not affected significantly by irrigation treatments, while, potassium concentration in maize grains was significantly affected with irrigation treatments, where it was increased in the order $I_1 > I_2 > I_3$. This trend confirms the results produced by maize grain yield as previously discussed.

Polymers do not only reduce the amount of water added to plants, but also, hydrogels are claimed to reduce leaching of fertilizers. This seems to occur through interaction of the fertilizer with the polymer. At research level, Jhurry (1997)[11] found the loading of N, P, and K fertilizers directly into crosslinked polyacrylamide gels received more attention. It was noticed that application of both HA and HS increases the N, P, and K concentrations in maize grains compared to the control one (H_0) and it was noticed that both nitrogen and potassium concentrations increased with HA application. In the opposite direction of those obtained in yield, N and K concentrations data, the addition of HS increment P concentration than that in HA treatment.

On the other hand, nitrogen, phosphorus, and potassium content in maize grains increases by the same order $F_2 > F_1$, Table V. The nitrogen content took the same line as its concentration, i.e. $I_2 > I_1 > I_3$, while, P and K took the trend of dry weight, i.e. $I_1 > I_2 > I_3$. Nitrogen and potassium contents were obviously higher in HA treatment than those in HS treatment. In contradict line with those obtained in N and K content, P increment by HS addition than HA. This is in agreement with that explained by Singh *et al.* (1997)[40] and Ali, (2011)[41] where they reported that the available N, P, and K in sandy soil increased with increasing the applied rates of carboxymethyl cellulose as a result of improving soil–water relations, as well as the availability of sufficient moisture around root zone that led to a greater proliferation of root biomass which results in higher absorption of nutrients subsequently and thus increasing production of biomass. Generally, El-Hady and Abo-Sedera (2006)[42] mentioned that the hydrogel has a positive effect on hydrophysical, chemical, and biological properties of the soil, where they reflect an increasing available and uptake of N, P, and K.

Macronutrients Use Efficiency. The average of N, P, and K use efficiency values increases significantly with increasing irrigation period, i.e., $F_2 > F_1$, and with decreasing irrigation rate, i.e., $I_3 > I_2 > I_1$. Application of HA tends to raise N, P, and K use efficiency by 25.5% compared to HS application, as seen in Table VI. The maximum value of N, P, and K use efficiency is associated with $F_1 \times I_3 \times$ HA, while the minimum value obtained due to the conjunction affected between $F_1 \times I_1 \times$ HS. The possible reasons for this finding were that high nutrient leaching with application of high amount of water in short frequency and addition of HA could have directly improved uptake by serving adequate water in root zone or by indirectly improving maize root development that happens lower under HS application. These results agree with those obtained by Ali, (2011)[41] who reported that, treating sandy soil with hydrogel increased fertilizer use efficiency by maize plants.

Table V. (A) Nitrogen, (B) Phosphorus, and (C) Potassium Uptake (kg/fed.) as Affected by Irrigation Frequency, Rate, and Hydrogel Application

Freq.	Irr. rate	Hydrogel source H_0	HS	HA	Mean
(A)					
F_1	I_1	83.6	92.3	92.4	89.4
	I_2	79.0	114.4	107.6	100.4
	I_3	81.0	89.2	117.8	96.0
	Mean	81.2	98.6	105.9	95.3
F_2	I_1	96.2	113.6	105.1	105.0
	I_2	85.9	106.2	115.0	102.3
	I_3	71.0	92.6	98.0	87.2
	Mean	84.4	104.1	106.0	98.2
Mean	I_1	89.9	103.0	98.7	97.2
	I_2	82.5	110.3	111.3	101.4
	I_3	76.0	90.9	107.9	91.6
	Mean	82.8	101.4	106.0	
$LSD_{0.05}$		F = ns I = 3.32 H = 2.70 I × F = 4.70 H × I = 4.68 H×F = ns H× I × F = 6.62			
(B)					
F_1	I_1	9.55	12.57	11.47	11.19
	I_2	8.42	12.76	13.64	11.61
	I_3	8.77	12.99	12.95	11.57
	Mean	8.91	12.77	12.69	11.46
F_2	I_1	10.69	13.44	13.77	12.63
	I_2	8.76	12.74	13.47	11.66
	I_3	8.05	13.39	14.18	11.87
	Mean	9.17	13.19	13.81	12.05
Mean	I_1	10.12	13.01	12.62	11.91
	I_2	8.59	12.75	13.56	11.63
	I_3	8.41	13.19	13.57	11.72
	Mean	9.04	12.98	13.25	
$LSD_{0.05}$		F = ns I = ns H = 0.34 I × F = 0.38 H × I = 0.59 H × F = 0.48 H × I × F = 0.83			
(C)					
F_1	I_1	69.77	64.87	76.43	70.36
	I_2	63.60	86.83	82.07	77.50
	I_3	56.10	64.47	98.73	73.10
	Mean	63.16	72.06	85.74	73.65
F_2	I_1	101.63	109.43	115.63	108.90
	I_2	66.50	98.07	95.37	86.64
	I_3	53.23	68.43	84.43	68.70
	Mean	73.79	91.98	98.48	88.08
Mean	I_1	85.70	87.15	96.03	89.63
	I_2	65.05	92.45	88.72	82.07
	I_3	54.67	66.45	91.58	70.90
	Mean	68.47	82.02	92.11	
$LSD_{0.05}$		F = 2.50 I = 2.16 H = 1.91 I × F = 3.05 H × I = 3.30 H × F = 2.70 H × I × F = 4.67			

Table VI. (A) Nitrogen, (B) Phosphorus, and (C) Potassium Use Efficiency as Affected by Irrigation Frequency, Rate and Hydrogel Application

		Hydrogel source		
Freq.	Irr. Rate	HS	HA	**Mean**
(A)				
F_1	I_1	1.85	3.04	2.45
	I_2	4.05	5.16	4.61
	I_3	3.92	5.67	4.80
	Mean	3.27	4.63	3.95
F_2	I_1	4.15	4.38	4.27
	I_2	4.23	4.86	4.55
	I_3	4.62	5.51	5.07
	Mean	4.34	4.92	4.63
Mean	I_1	3.00	3.71	3.36
	I_2	4.14	5.01	4.58
	I_3	4.27	5.59	4.93
	Mean	3.80	4.77	
$LSD_{0.05}$	$F=0.13$ $I=0.29$ $H=0.15$ $I\times F=0.41$ $H\times I=0.26$ $H\times F=0.21$ $H\times I\times F=$ ns			
(B)				
F_1	I_1	17.09	28.09	22.59
	I_2	37.36	47.67	42.52
	I_3	36.22	52.37	44.29
	Mean	30.22	42.71	36.47
F_2	I_1	38.35	40.41	39.38
	I_2	39.05	44.89	41.97
	I_3	42.66	50.90	46.78
	Mean	40.02	45.40	42.71
Mean	I_1	27.72	34.25	30.99
	I_2	38.21	46.28	42.24
	I_3	39.44	51.64	45.54
	Mean	35.12	44.06	
$LSD_{0.05}$	$F=1.21$ $I=2.67$ $H=1.37$ $I\times F=3.77$ $H\times I=2.37$ $H\times F=1.93$ $H\times I\times F=$ ns			
(C)				
F_1	I_1	11.11	18.26	14.68
	I_2	24.29	30.99	27.64
	I_3	23.54	34.04	28.79
	Mean	19.65	27.76	23.70
F_2	I_1	24.93	26.27	25.60
	I_2	25.38	29.18	27.28
	I_3	27.73	33.09	30.41
	Mean	26.01	29.51	27.76
Mean	I_1	18.02	22.26	20.14
	I_2	24.83	30.08	27.46
	I_3	25.63	33.56	29.60
	Mean	22.83	28.64	
$LSD_{0.05}$	$F=0.79$ $I=1.73$ $H=0.88$ $I\times F=2.45$ $H\times I=1.54$ $H\times F=1.26$ $H\times I\times F=$ ns			

CONCLUSIONS

From the results, we can conclude that irrigation by 100% of water requirement every 2 days with addition of HA gave the highest grain yield and save about 15% of irrigation water. While HS, which was prepared from rice straw as an agricultural residues, is the cheapest and environmentally friendly, where it is free of acrylamide and totally biodegradable and biocompatible. It is appropriate to declared that, under the condition of studied area, which was affected with salinity, high frequency irrigation is recommended. While under normal condition, irrigation every three days is the best.

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