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Synthesis and Swelling Characterization of Carboxymethyl Cellulose -g- Poly(Acrylic acid- co –Acrylamide) Hydrogel and their Application in agricultural field

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Abstract : Carboxymethyl cellulose (CMC) modification by grafting polymerization of Acrylic acid (AAc) and Acrylamide (AAm) were prepared by means of free radical polymerization to prepare a super absorbent hydrogel. The effect of CMC: AAc: AAm ratios, crosslinking agent N,N-methylene bis acylamide (MBAm) dose and initiator potassium persulphate (KPS) concentration on preparation of a hydrogel were investigated. Changing the crosslinking density has been utilized to achieve the desired higher swellability and best cohesive property of the hydrogel. The swelling characteristics and its kinetics of the so prepared hydrogel were additionally considered. The formation of super absorbent hydrogel (CMC -g- AAc /AAm) was affirmed by Fourier transform infrared spectroscopy (FTIR). Green peas plants were grown in pots containing mixture of sand and SAH (0.7 %) and were subjected to drought (75% of water requirements) comparing to control treatment whereby grown green peas in pots filled with sand soil and subjected to full irrigation or 75% of water requirements.

Key words : *arboxymethyl cellulose, superabsorbent hydrogel, Cultivation.*

Introduction

The past few decades have seen a passion increase in the search for water absorption resins or hydrogels. Hydrogels are a group of copolymers with cross-linked networks. The hydrogels are skilled to gather a lot of water, keeping the three-dimensional structure that relies upon the hydrophilicity of the polymers. Hydrogel swell by absorbing fluid and vice versa on drying. Hydrogel can assimilate and hold a lot of water as well as fluid containing electrolytes even under weight contrasted and customary sponges such as cotton, sponge, wipe and so forth. Hydrogels on the other hand are intriguing materials, as an aftereffect of low toxicity and high biocompatibility properties, along these lines; they turn into the focal point of consideration for a developing enthusiasm of examination, including hygienic items, agribusiness and cultivation, wastewater treatment, drug-conveyance framework, biomedical, and pharmacobiological and so on [1]. All these properties of the hydrogels have made them a fundamental part in an assortment of modern applications and purchaser things too [2, 3]. Both common and engineered polymers have been utilized as a model substrate for the hydrogel planning [4]. Several techniques are available for the preparation of the hydrogels. They are for the most part produced using water-dissolvable monomers by means of free radical polymerization responses. Acrylic acid, acrylamide and their derivatives are the most prevalent water-dissolvable monomers utilized for this reason. These monomers likewise can be joined onto some normal polymers to get hydrogel materials. Another approach to synthesize hydrogel materials is to prepare interpenetrating polymer network (IPN) composites from water-soluble monomers with different materials [5]. Modifying natural polymeric compounds can be considered as a promising way for preparing new. Graft copolymerization of vinyl monomers onto natural polymers is a convincing approach to manage achievement of these materials. Development of superabsorbent hydrogels was achieved for agricultural purpose through grafting of acrylonitrile onto hydrolysed starch[6].

Many studied done to apply superabsorbent hydrogel in agricultural field. Using super absorbent hydrogel increases soil ability to hold water, so plant growth increases and lateness the period to wilting point in drought stress [7-9]. Ataei and Ghorbani [10] reported that during warm season, each 1 m² soil needs about 14 to 18L water per day, this amount was reduced to half by adding 100 g hydrogel to each1m²soil. Also, adding superabsorbent hydrogel to soil improves plants through supplying the plant roots with water, providing soil with potassium ion which is important to retain a K^+/Na^+ homeostasis and components of polymers held the ions of salt in the drying soil [10]. On the other hand, Abedi- Koupai and Mesforoush[11] reported that adding high doses of the hydrogel causes bad results because of filling most of empty spaces of soil causing sever soil ventilation.

The present work is undertaken with view of studying the carboxymethyl cellulose (CMC) alteration by grafting polymerization of Acrylic acid (AAc) and Acrylamide (AAm) utilizing potassium persulphate (KPS) as initiator. The so prepared CMC /AAc/AAm hydrogel graft copolymer was further crosslinked with N,Ǹ methylene bisacrylamide (MBAAm) to acquire the sorbent material as hydrogel. Experimental parameters such as CMC: AAc: AAm ratios, concentration of the initiator and crosslinker dose were varied to acquire hydrogels with higher water uptake. Swelling behavior and kinetics of the so prepared hydrogels were evaluated. The study was reached out to incorporate a serious active investigation of the swellability of the prepared hydrogel and the reliance of the swell ability on CMC: AAc: AAm ratio. Test parameters, for example, CMC: AAc: AAm ratio, grouping of the initiator and crosslinker dosage were changed to acquire hydrogels with higher water uptake. Swelling conduct and swelling energy of the so got hydrogels were assessed. It is worth mentioning that the hydrogel under investigation will be applied in agricultural field

Materials and Methods

Materials

Carboxymethyl cellulose sodium salt (CMC) purchased from El-Nasr Pharmaceutical Chemicals Co. Acrylic acid (AAc) in the monomeric form produced by Sisco Research Lab. Pvt. Ltd., India, potassium hydroxide (KOH) supplied by Sigma–Aldrich, Inc., N,N-methylenebisacrylamide (MBAAm) as a crosslinking agent and potassium persulfate (KPS) as initiator. These reagents were used as laboratory grade chemicals and were employed without further purification.

Preparation of CMC-g-poly(AAc/AAm) superabsorbent hydrogel.

CMC solution has been prepared by mixing CMC powder to distilled water and heated to 80°C. The calculated amount of initiator KPS was added and kept at 80°C for 10 min to generate radicals. After cooling the reactants to 40°C, the mixture of AAc, AAM and MBAA was added to the mixture, and then pH was adjusted by KOH solution. The temperature was risen to 80°C and maintained for 2 h to complete reaction. The obtained products have been washed using methanol/water, dried and weighed. Figure 1 represents the block diagram of the preparation steps to produce CMC -g- poly(AAc /AAm) superabsorbent hydrogel.

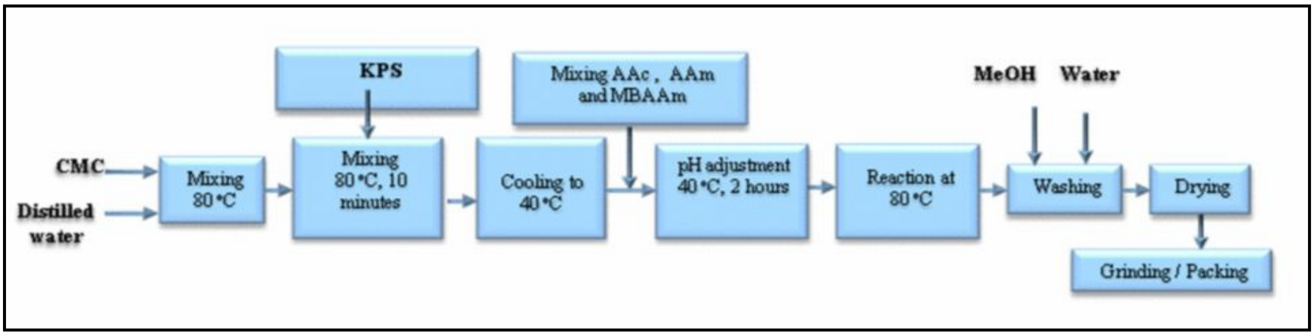


Figure 1: Block diagram for the production of CMC -g- (AAc /AAm) superabsorbent polymer

Analytical methods

Fourier Transform Infra Red (FTIR)

Fourier Transform Infra Red (FTIR), the preferred method of infrared spectroscopy. Unknown materials, the quality or consistency of a sample and the amount of components in a mixture were provided through FTIR – 6100 – JASCO, JAPAN. The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample.

Swelling studies

The swelling characteristics of the hydrogel prepared were measured gravimetrically[12]. One gram of the dried hydrogel samples were placed in 100 ml of distilled water at room temperature. The swelling of the prepared hydrogel was tested at different pH values (from 3 to 10) by adjusting the pH using 0.1 N sulphuric acid and 0.1N NaOH using pH meter (Jennway - model 3051). The swelling properties of the prepared super absorbent hydrogel (SAH), such as the swelling ratio (S) and equilibrium swelling ratio (S_{eq}) were determined and calculated with the following equations [12]:

$$S\left(\frac{g}{g}\right) = \frac{W_t - W_o}{W_o} \qquad (1)$$

$$S_{eq}\left(\frac{g}{g}\right) = \frac{W_{eq} - W_o}{W_o} \qquad (2)$$

where W_o, W_t and W_{eq} are the weights of the samples in the dry state, the swollen state at a certain time, and the completely swollen state, respectively. The equilibrium swelling of the gels was determined as follows: gels were dried at 80°C. After the weight of the dried samples was determined, the samples were equilibrated in distilled water for 3 days at room temperature and then weighed again.

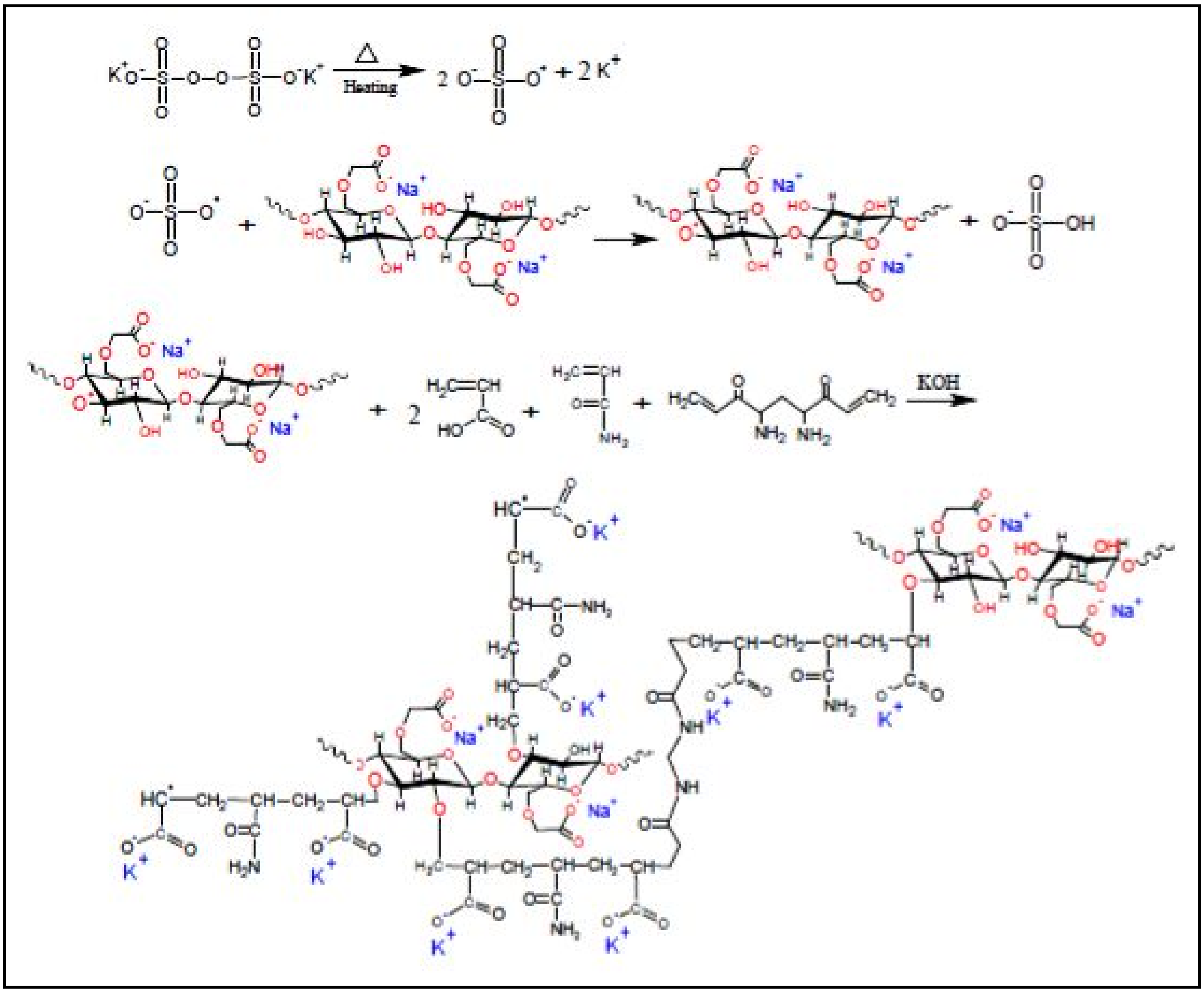
Cultivation of Green Peas plant

Seeds of green peas plants CV master B were sown in pots 25 size filled with mixture of sand soil and 0.7 % of hydrogel. The grown plants subjected to drought, so irrigated with 75% of water requirement. In addition to control treatment whereby plants were sown in sand soil without hydrogel and divided into two parts, the first was irrigated with full water requirement (100 % WR) meanwhile the second was irrigated with 75% of water requirement. Dose of SAH was selected according to our previous study[13]. At the stage of flowering onset (after about 40 days from sowing), total chlorophyll content (as SPAD readings) of leaves was recorded using TYS-A chlorophyll Meter (Zhe Jang Top Instrument Co. LTD., China) as well as some vegetative and yield parameters.

Results and Discussion

Mechanism of CMC/AAc/AAm hydrogel formation

The superabsorbent hydrogel (SAH) under investigation was prepared according to a procedure involved graft copolymerization both AAc and AAm on CMC under the free radical initiating effect of KPS and MBAAm as crosslinker. The suggested reaction mechanism for preparation of cross linked grafted CMC can be represented as shown in the following scheme: The persulphate initiator decayed to generate anionic radical upon heating. This anionic radical can abstracts hydrogen from the hydroxyl group of CMC to form macro-radicals which initiate the monomers polymerization, resulting to the formation of the grafted chains.



Factors affecting SAH efficiency

Sodium carboxymethyl cellulose (CMC) amendment by grafting polymerization of AAc and AAm onto CMC were prepared via free radical polymerization to prepare a super absorbent hydrogel [CMC -g- (AAc /AAm)]. The impact of CMC: AAc: AAm ratios, crosslinking agent (MBAAm) dose and initiator concentration on hydrogel preparation were explored.

Effect of CMC/AAc/AAm ratios

As appeared in Figure 2 it is observed that water absorption of cross-linked [CMC -g- (AAc /AAm)] hydrogel tend to increment with increasing AAc ratio in the prepared hydrogel. It might be because of the presence of carboxylic group gatherings. Equal ratios of CMC, AAc and AAm show good water absorption. On the other hand these prepared hydrogels characterized by good robust texture. While as the other ratios namely 2:1:2, 1:1:2 and 1:2:2 are almost equal with less robust texture.

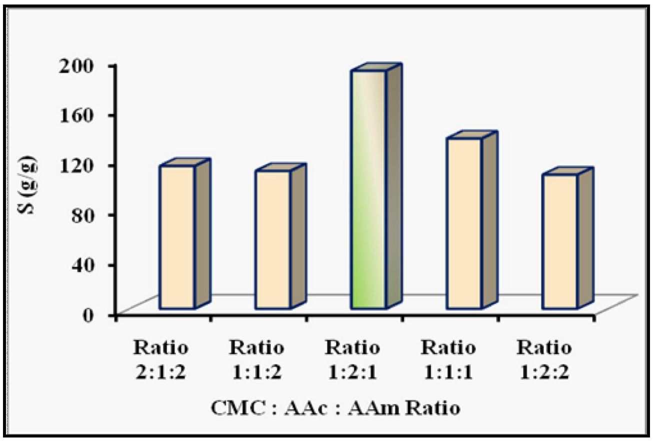


Figure 2: Effect of CMC: AAc: AAm ratio on swelling ratio of SAH

Effect of crosslinker density

Crosslinking density is a very important parameter in determination of swelling capacity of superabsorbent hydrogels. Changing crosslinking density has been utilized to accomplish the desired higher swellability. Distinctly the swellability of hydrogel increased by increasing MBAAm dose from 0.08g to 0.1g. Further increase in MBAAm causing a decrement in swellability. Figure 3 demonstrates the impact of MBAAm concentration on the swellability of hydrogels. It has been observed that the swellability of grafted hydrogels reached to a maximum value of 190 g/g of dry hydrogel upon using 0.1g of MBAAm. The amounts of space inside the hydrogels network are deciding the swelling capacity of a hydrogel. The use of higher concentrations of the crosslinker (MBAAm) leads to tighter network structure of the hydrogel and will swell less if compared to the same hydrogels with a lower crosslinking ratio. Despite the fact that, the higher crosslinking density results in stronger hydrogel, lower swelling ratio has been obtained. Expanded crosslinking density will build the resistive force to chain elongation consequently reducing the degree of equilibrium swelling rather than hydrogels with low crosslinking density. Subsequently, there is an optimum level of crosslinking [14].

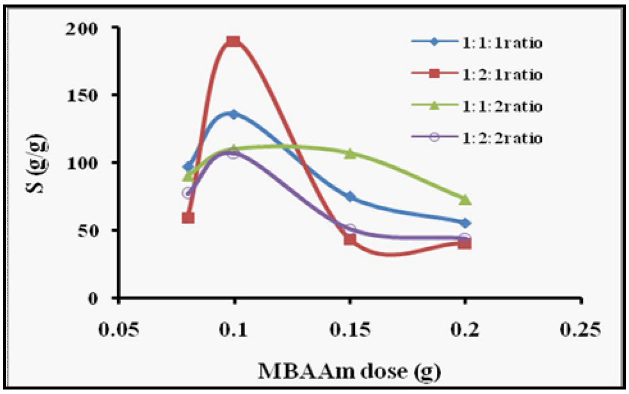


Figure 3: Effect of MBAAm dose on swelling ratio of SAH

Effect of initiator dose

In order to investigate the effect of KPS concentration on the prepared hydrogel swellability experiments were conducted at different KPS concentrations ranging from 0.1 g to 0.6 g. The results illustrated in Figure 4 revealed that increasing of KPS concentration from 0.1g to 0.5g increases the swelling proportion from 88.6 g/g to 190 g/g. Further, increase in KPS concentration bringing down the swellability of the prepared hydrogel. This is might be due to the presence of more free radicals which acts as a scavenger to the grafting response. Thus, using a concentration of 0.5 g KPS is employed at fixed MBAAm amount of 0.1 g and CMC:AAc :AAm ratio 1:2:1 a swelling ratio of 190 g/g is achieved.

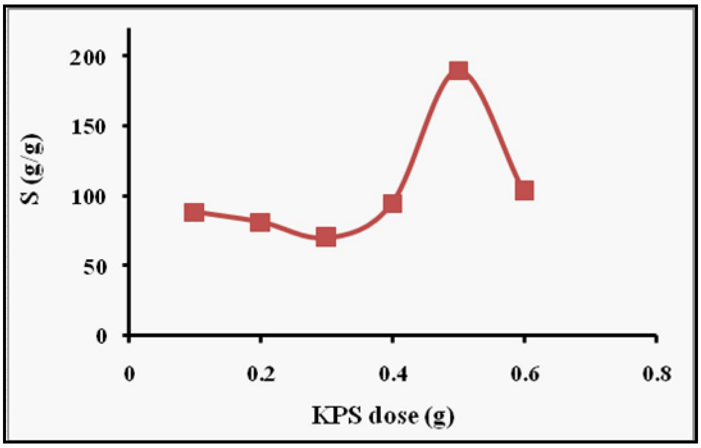


Figure 4: Effect of KPS dose on swelling ratio of SAH

Fourier Transform Infra Red (FTIR)

For the identification of the hydrogel, infrared spectroscopy was used. The basic changes of the grafting copolymerization were investigated by comparing the FTIR spectrum of the CMC substrate with that of the grafted copolymer. Infrared spectroscopy spectra of CMC were shown in Figure 5. The absorption bands in the range 4000 – 1500 cm^{-1} region identify the functional groups and also termed the region of the functional groups of the FTIR spectrum. In both spectra CMC and CMC-*g*-poly(AA-*co*-AAm), a broad band absorption between 3800-3000 cm^{-1}, correlated with bonded and free hydroxyl groups, was observed. The peak in wave number 1621 and 1425 cm^{-1} elucidate the presence of carboxymethyl elucidate the presence of carboxymethyl constituent. elucidate the presence of carboxymethyl components. The wave number 1621 cm^{-1} plot of C=O group stretching of acetyl or Carboxymethyl. The obtained data of peaks in FT-IR spectra of Carboxymethyl cellulose ingredients, was listed in Table 1. The dislodging may be due to the hydroxyl groups stretching band of the carboxymethylated in C_6. The substitution of the hydroxyl group in C_6 significantly changes that it raised carbonyl group (-C=O) in wavelength 1621 cm^{-1} and –CH_2 in wavelength 1425 cm^{-1}.

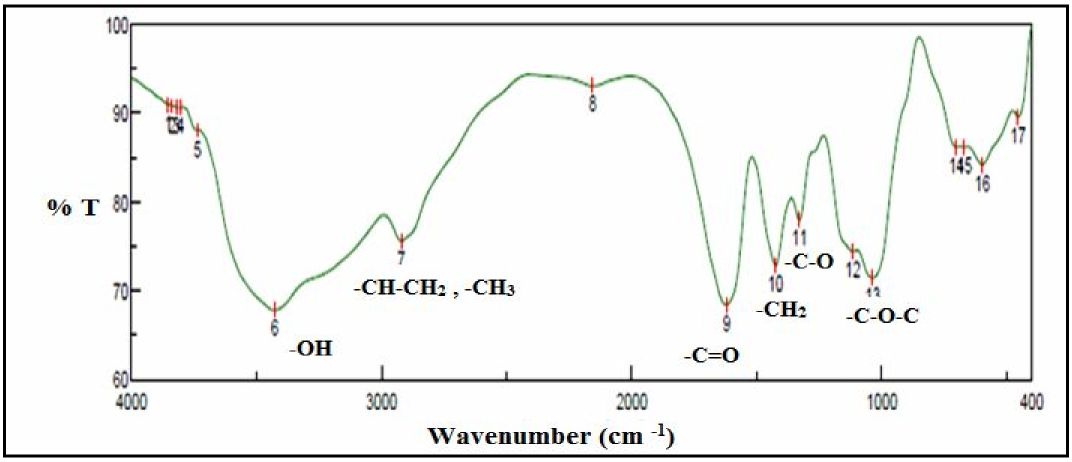


Figure 5: Typical FTIR spectrum showing functional group and fingerprint regions of CMC

Table 1: Assignment of main absorption bonds in CMC and SAH

Wave number (cm^{-1})	
CMC	**Assignment**
3427	– OH stretching
2920	–CH stretching CH_2 and CH_3
1621	– C = O region
1425	– CH_2 bonding
1329	–OH in plane bonding
1036	– C– O – C asymmetry stretching alcohl

Infrared spectroscopy spectra of CMC-*g*-poly(AA-*co*-AAm) were shown in Figure 6. The peak at 3432 cm^{-1} spotted in the IR spectrum of the prepared SAH is assigned to the NH stretching of the acrylamide unit, which overlaps the -OH stretching band of the carboxymethyl cellulose. The peak at 2924.91 cm^{-1} is attributed to the C—H stretching of the polymer structure. The absorption peak at 1744.68 cm^{-1} signified the carbonyl stretch of the carboxylic acid groups. The appearance of peak at 1595.23 cm^{-1} is due to the absorption bands of the amide moiety of the acrylamide unit. The sharp peak at 1559.23 cm^{-1} is caused by the C = O asymmetric stretching in the carboxylate anion that is reaffirmed another sharp peak at 1405.27 cm^{-1}, which is relevant to the symmetrical stretching of the carboxylate anion. Moreover, peaks at 1458 and 1104.43 cm^{-1} identifying with the –CO – O - and OH coupling interactions of the carboxylic group and C=N stretching vibrations can also be noticed. The absorption peak showed at 1055 cm^{-1}, attributed to the –CO – C - stretching vibration, can also be spotted. Comparing of the two IR spectra revealed that the absorption bands of AA and AM appear in the spectrum of the prepared SAH, and this assured the grafting copolymerization of AA and AM monomers onto the CMC backbone.

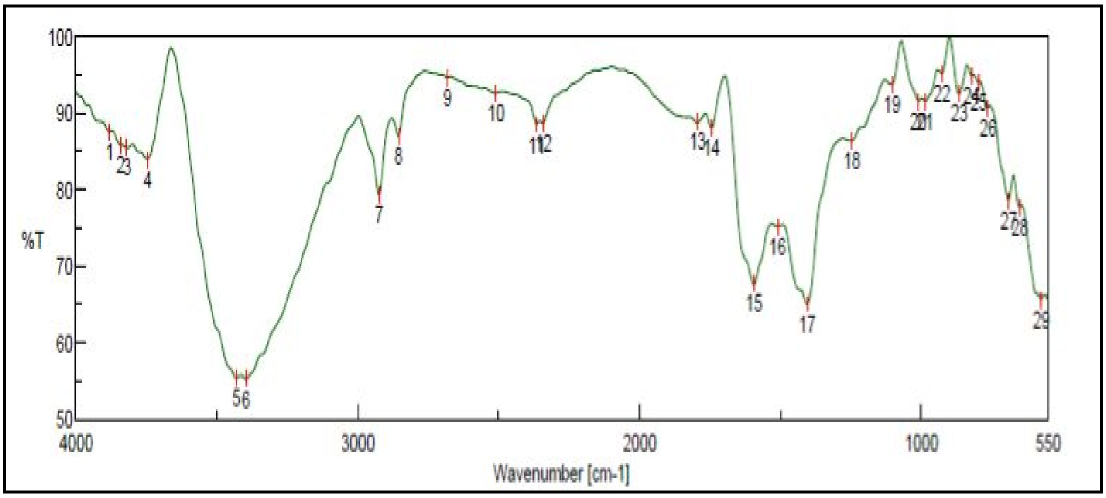


Figure 6: Typical FTIR spectrum showing functional group and fingerprint regions of SAH

Swelling behaviour

The swelling capacity of the prepared hydrogel was undertaken in distilled water at room temperature (25 ± 1°C). The polymers swelling property is reflected by the swelling water proportion (S) which is the proportion of the weight of the swelled hydrogel to the dry hydrogel weight. The perfect swelling property of hydrogels has the potential to conserve water. Figure 7 revealed that increase in the hydrogels swelling is directly correlated to the time of swelling. In general, water retention is increased with time. The Inclination of the prepared hydrogel to swell is faster at the first hour then is diminished as time tended to 24 hours.

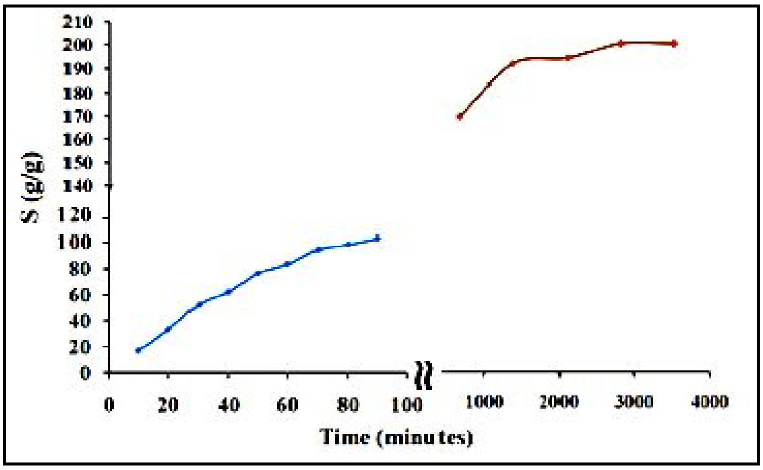


Figure 7: Effect of swelling duration on water retention of the prepared hydrogel

On the other hand the swelling tests were carried out on the three ratios at different pH (3, 5, 6.5, 8 and 10). The swelling behaviour of the prepared hydrogel is influenced by pH value. Figure 8 demonstrates that decreasing pH of the swelling media enhanced the swellability of the hydrogel under investigation and followed the order: pH 6.5 > pH 5 and pH 8 > pH 10 > pH 3.

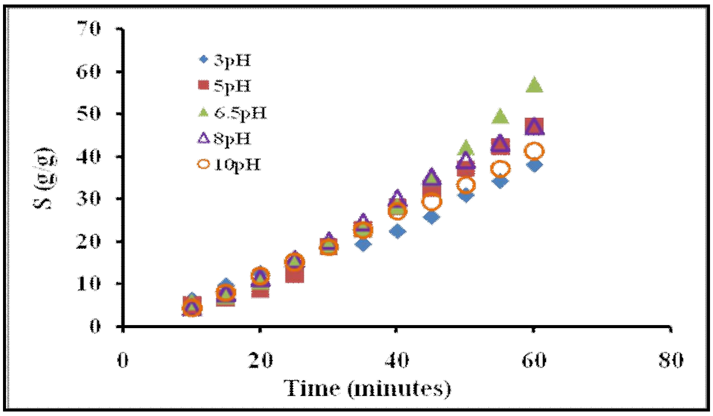


Figure 8: Effect of swelling pH on water retention of the prepared hydrogel

Swelling kinetics

The swelling behaviour was related to the absorption mechanism which is determined by the diffusion process. The kinetic study allows the hydrogel's swelling order to be determined: first order (Fick Model), second order (Schott Model), or if it adjusts to both at a particular time. The Fick study was applied for the first swelling times, due to the fact that for longer times there is a deviation in this behavior. The Schott Model was used for longer times than the one mentioned when the density of the sample has increased. The perception of a mechanism to control the swelling processes through kinetic models. The mechanism of the swelling processes was visualized by kinetic models. These models were used to analyze the data obtained from the experimental work. A simple kinetic analysis is referred by the given second-order equation[15]:

$$\frac{dS}{dt} = K_s\left(S_{eq} - S_t\right) \qquad (3)$$

where k_s is the swelling rate constant and S_{eq} is the swelling degree at equilibrium. After integration, when the initial conditions S = 0 at t = 0 and S = S at t = t, was applicable, equation (3) became as follows:

$$\frac{t}{S} = Bt + A \qquad (4)$$

where $B=1/S_{eq}$ is the reciprocal of equilibrium swelling and $A = 1/k_S(S_{eq})^2$ is the inverse of the rate of swelling at the initial state of the hydrogel. The kinetic models were considered by plotting **t/S** versus **t** for the prepared hydrogel. Initial swelling rate (r), k_S, and equilibrium swelling $[(S_{eq})_{max}]$ as swelling parameters were calculated from the equations of the straight lines shown in Figures 9 and 11.

The diffusion mechanism in swellable hydrogels solicits a considerable attention because of several applications in agriculture, biomedicine, environmental and pharmaceutical. The diffusion of water in hydrogel indicates that it could be assessed with the following equation [14]:

$$s\,({}^{g}/_{g}) = \frac{(W_t - W_o)}{W_o} = Kt^n \qquad (5)$$

where K and **n** are the swelling constant and the swelling exponent calculated from the slopes of the lines of Ln S versus Ln t plots (Figures 10 and 12). For Fickian diffusion, **n** = 0.45–0.50, whereas 0.50 < **n** < 1.0 indicates that the diffusion is non-Fickian type (anomalous diffusion) [5, 13]. From Figure 9, the value of **n** was found to be 0.826, and it is revealed that the diffusion was non-Fickian type. On the other hand the study of the swelling kinetics for longer times (> 720 minutes), the value of **n** was found to be 0.103. The calculated values of swelling parameters were set forth in Table 2. The experimental data was found to be in matching with swelling kinetic model, and for the first swelling times the diffusion of water into the hydrogels was the non-Fickian type. While as for longer swelling time the density of the sample has increased and causes a regular diffusion of water into the hydrogel.

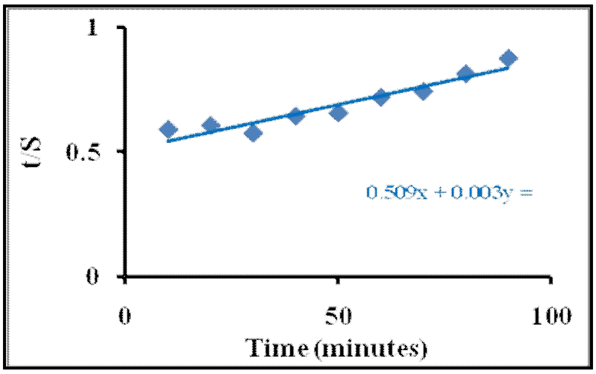


Figure 9: Swelling kinetic relations of prepared hydrogel at the first 90 minutes

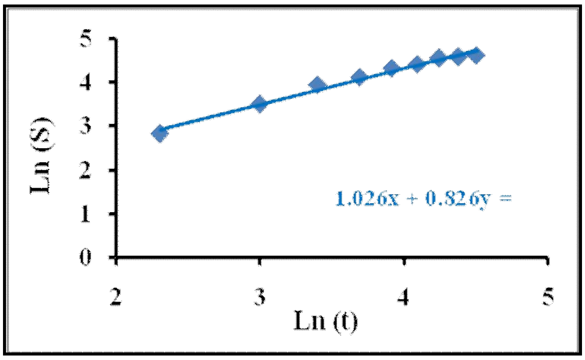


Figure 10: Swelling kinetic relations of prepared hydrogel at the first 90 minutes

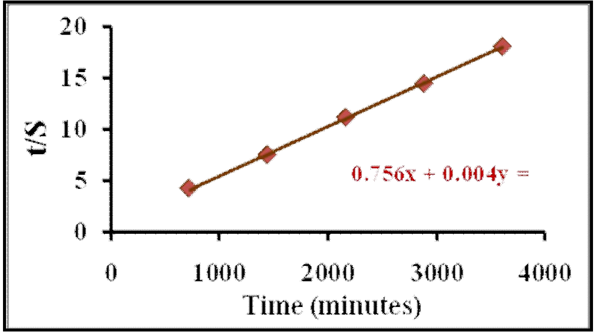


Figure 11: Swelling kinetic relations of prepared hydrogel at time >720 minutes to 3600 minutes

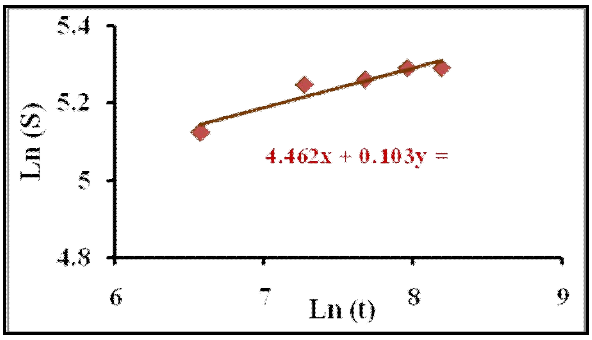


Figure 12: Swelling kinetic relations of prepared hydrogel at time >720 minutes

Table 2: Some swelling parameters of CMC-g-poly(AAc–co-AAM) superabsorbent hydrogel

Kinetic Model	Swelling parameters				
	$(S_{eq})_{exp}$	$(S_{eq})_{max.}$	K_s x 10^3	r	n
Fick Model	102.6	333.3	1.76817	1.96	0.826
Schott Model	190	250	2.1164	1.32	0.103

Cultivation o of Green Peas plant

Seeds of Green Peas plants were sown in sandy soil containing mixtures of sand and SAH in addition to two control treatments (grown in sand without hydrogel). Plants which sown in mixed sand soil and 0.7 % SAH and control one were irrigated at 75% water requirement (WR), while control two irrigated with 100% water requirement. Green peas plant is sensitive to water amount trapped in SAH and this enhancement of water holding capacity caused positive effects on the physiological changes, growth and productivity of green peas plants. While as another control one was watered at 100 % WR. Figure 13 shows the effect of adding SAH to sand soil and the amounmt of water requirement on plant yield and number of pods/plant. It is clear that both plant yield and number of pods/plant were increased by adding SAH to sand soil compared with either control at 75 % WR or control at 100 % WR and the same holds true for root and vegetative growth weights of green peas as listed in Table(3).

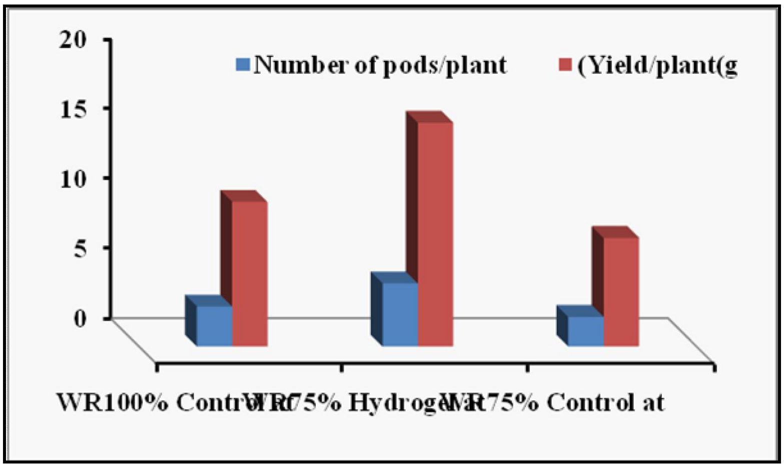


Figure 13: Effect of presence of SAH on productivity of green peas grown in sandy soil under drought stress.

Application of super absorbent hydrogen motivates higher total chlorophyll content of leaves and soil water potential compared to control plants. Figure 14 shows the effect of presence of SAH on some vegetative growth characters of green peas grown in sandy soil under drought stress such as root length, plant length, number of leaves and chlorophyll content. It is evident that presence of SAH improve the studied characters of green Peas plant.

Table 3: Effect of presence of SAH on root and vegetative growth weights of green peas grown in sandy soil under drought stress.

Treatment	Weight of fresh root (g)	Weight of dry root (g)	Weight of fresh Vegetative growth (g)	Weight of dry Vegetative growth (g)
Control at 100% WR*	3.03d	0.40d	18.72de	7.93d
Hydrogel at 75% WR	3.43c	0.47c	20.17c	5.30bc
Without treatments at 75% WR	2.73e	0.36e	16.84f	4.47e

Means within a column followed by the same letter are not significantly different (P = 0.05) according to Duncan's multiple range test.

*WR: Water requirement.

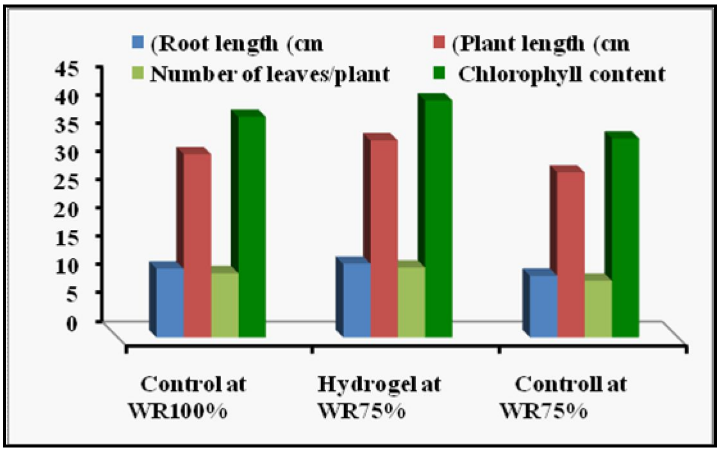


Figure 14: Effect of presence of SAH on some vegetative growth characters of green peas grown in sandy soil under drought stress.

Conclusion

CMC -g- poly(AAc –co-AAM) superabsorbent hydrogel were successfully prepared via a free radical copolymerization. Preparation factors such as CMC/AAc/AAm ratios**,** crosslinker density and initiator dose were also investigated**.** The swelling characteristics of the resulting hydrogels were evaluated. Swelling capacity was found to be related to swelling duration and pH. The obtained SAH has an optimum swelling ratio 190 g/g. At the first 90 minutes kinetic studies of swelling behavior was found to be in agreement with experimental data and the diffusion of these hydrogels was of the non-Fickian type. While as for longer swelling time the density of the sample has increased and causes a regular diffusion of water into the hydrogel. Vegetative growth and yielding of green peas plants grown in sandy soil amended with SAH were improved even under drought compared with that grown in sandy soil only under drought or full irrigation.

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