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Water sorption behaviour of highly swelling (carboxy methylcellulose-g-polyacrylamide) hydrogels and release of potassium nitrate as agrochemical

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Abstract

Novel types of highly swelling hydrogels have been prepared by grafting crosslinked polyacrylamide chains onto carboxymethylcellulose (CMC) via a free radical polymerization method. The hydrogels were characterized by IR spectral analysis and by evaluating various network parameters such as average molecular weight between crosslinks (M_c), crosslink density (q) and the number of elastically effective chains (V_c). The hydrogels showed enormous swelling in aqueous medium and displayed swelling characteristics which were highly dependent on the chemical composition of the hydrogel and pH and ionic strength of the medium in which the hydrogel was immersed. The kinetics of water uptake and the water transport mechanism were studied as a function of the composition of the hydrogel and the pH of the medium. The hydrogels were also loaded with potassium nitrate as a model agrochemical and their potential for controlled release of potassium nitrate was judged by measuring conductivity. Various kinetic parameters such as the diffusion coefficient and swelling exponents were also calculated.

Keywords: Hydrogel; Carboxymethylcellulose; Polyacrylamide; Release; Potassium nitrate

1. Introduction

Hydrogels are a unique class of polymeric materials which imbibe enormous amounts of water when left in a water reservoir for long times (Peppas, 1987). The underlying property for this unusual behaviour of hydrogels is their transition from a glassy to a rubbery state when contacted with thermodynamically compatible solvents (Colombo, 1993). This water sorption property of hydrogels accounts for a great number of biomedical and technological applications such as artificial implants (Williams, 1990), contact lenses (Peppas & Yang, 1980), enzyme immobilization (Moustafa, Kahil, & Faizalla, 2000), etc. Other fields for hydrogel applications cover the area of controlled release technology which is being intensively used in pharmaceuticals and agriculture (Akelah & Moet 1990; Dutta, Vishwanathan, Mimrot, & Ravikumar, 1997; Kim, Bae, & Okana, 1992; Knop, 1996; Kydonieus, 1980; Saraydin, Karadag, & Guven, 1998). Controlled release polymer matrix systems offer a number of potential advantages over the conventional means of applications (Graham, 1990). The principal advantage is that these systems allow much less active agent to be used.

Agrochemicals are used to improve production of crops. Conventional application of agrochemicals can result in ground water contamination (Kenawy & Refale, 1997) which is further intensified by over-application and point source contamination. There is a need for a more controlled application of agrochemicals to reduce amounts of active ingredients without diminishing efficiency. The replacement of conventional agrochemical formulations by controlled release systems not only helps to avoid treatment with excess amounts of active substances, but also offers, the most suitable technical solution in special fields of application (i.e. in aquatic weed control and pest management in paddy fields). Controlled release formulations are used to maintain the local concentration of active ingredients in the soil and to reduce run off.

Chemically modified polysaccharides such as starch, guar or cellulose (Colombo, Bettini, & Peppas, 1999a,b; Gabrielli & Gatenholm, 1998) have been used in hydrogel preparations,

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however, the use of carboxymethylcellulose (CMC), an important modified polysaccharide, in gel formulations has not been extensively reported. The present paper reports a study on the water sorption dynamics of a novel type of hydrogel prepared by grafting crosslinked polyacrylamide chains onto CMC backbone by free radical polymerization.

2. Experimental

2.1. Mateials

CMC was obtained from the Loba Chemie (India) in the form of sodium salt (Molecular weight 5×10^4 and viscosity of 5% solution, 140 mpa s) and used as such. Acrylamide (AM) (Loba Chemie, India) was freed from the inhibitor by crystallization and dried under vacuum for a week. N,N′-methylene bis acrylamide (MBA) (Loba Chemie, India) was used as a crosslinking agent without any pretreatment. Other chemicals were of analytical grade and doubly distilled water was used throughout the experiments.

2.2. Hydrogel preparation

The method of preparation of the graft polymer was the same as described in our earlier communications (Bajpai & Shrivastava, 2000). In brief, into a petridish (5 cm diameter, Corning) were mixed CMC (0.40 g), acrylamide (10.5 mM), MBA (0.13 mM) and KPS (0.037 mM) and the mixture was homogenized. The petridish was then kept at 80 °C for 3 h so that the contents changed into a gel like mass. The gel so obtained was dried at 60 °C for 18 h and a thin brittle film was obtained. The film was equilibrated with water for 72 h so that the unreacted monomer and other chemicals were washed out. The gels were dried at room temperature and stored in sealed containers.

2.3. Mechanism of grafting

The reaction scheme proposed for the grafting of crosslinked polyacrylamide chains onto CMC is shown in Fig. 1.

The percentage and efficiency of grafting were calculated using the following equations,

$$\%\ \text{grafting} = \frac{W_1 - W_0}{W_0} \times 100 \tag{1}$$

$$\%\ \text{efficiency} = \frac{W_1 - W_0}{W_0} \times 100 \tag{2}$$

where W_1, W_0 and W_2 denote, respectively, the weight of the grafted CMC, the weight of native CMC taken, and the weight of acrylamide (AM) used.

The various samples of prepared by taking different amounts of CMC, monomer (AM), crosslinker (MBA) and polymerization initiator (KPS) in the feed mixtures are listed in Table 1. The %grafting and %efficiency are also presented in this table.

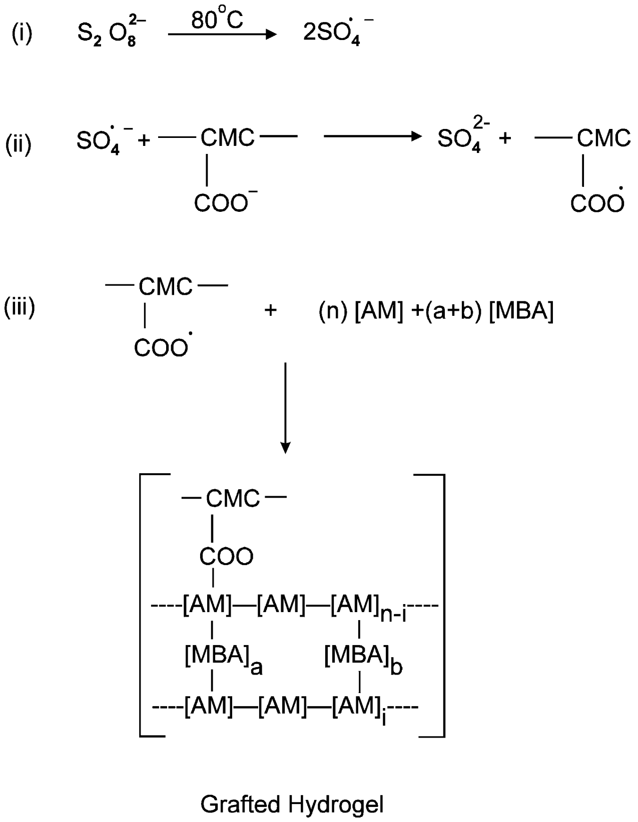

Fig. 1. An illustrated scheme of reactions for the graft copolymerization of crosslinked PAM chains onto CMC.

2.4. Swelling kinetics

Conventional gravimetric procedure (Vazquez, Roman, Peuiche, & Cohen, 1997) was adopted for studying the swelling kinetics. In brief, preweighed (0.04 g) pieces of hydrogels were immersed in bidistilled water, taken out at desired time intervals, freed from excess water by pressing them between two blotting papers, and weighed. The swelling ratio was calculated from the following relationship.

$$\text{Swelling ratio (SR)} = \frac{\text{Weight of the swollen gel}}{\text{Weight of the dry gel}} \tag{3}$$

The kinetic results were analyzed using the following two equations (Masaro & Zhu, 1999)

$$\frac{W_t}{W_\infty} = kt^n \tag{4}$$

and,

$$\frac{W_t}{W_\infty} = 4\left(\frac{Dt}{\pi l^2}\right)^{1/2} \tag{5}$$

where the terms involved have their usual significance. It is worth mentioning here that Eqs. (4) and (5) are valid only when $W_t/W_\infty < 0.6$ and $W_t/W_\infty < 0.8$, respectively.

Table 1
Data showing the yields of grafts hydrogels of various feed compositions

CMC (g)	AM (mM)	MBA (mM)	KPS (mM)	Percent grafting	Percent grafting efficiency	Equilibrium swelling ratio
0.2	10.5	0.13	0.037	110.6	40.2	52.8
0.3	10.5	0.13	0.037	137.4	46.6	64.2
0.4	10.5	0.13	0.037	167.5	53.6	92.6
0.4	7.03	0.13	0.037	178	62.6	110
0.4	14.06	0.13	0.037	142.2	70.0	78.4
0.4	21.1	0.13	0.037	104.8	86.2	46.6
0.4	10.5	0.064	0.037	140.4	94.3	66.8
0.4	10.5	0.26	0.037	102.2	70.6	48.6
0.4	10.5	0.39	0.037	90.4	66.2	42.2

2.5. Loading of potassium nitrate

In the present work the loading was performed by equilibrating preweighed pieces of the dry gel in the aqueous solution of KNO_3 of known concentration ($1\% w/v$) and thereafter drying and weighing again.

The percent loading was calculated by the following formula,

$$\%\ \text{Loding} = \frac{m_1 - m_0}{m_0} \times 100 \qquad (6)$$

where m_1 and m_0 are the weights of KNO_3 in the loaded and dry hydrogels, respectively.

2.6. Determination of potassium nitrate release

To study the release of KNO_3, the loaded gels of known weights were placed in measured volume (25 ml) of distilled water (release medium) under unstirred condition. The released amount of KNO_3 at different time intervals (M_t) was determined by measuring the conductivity of the release medium using a conductivity meter (Systronics, Model No.303, India) at desired time intervals. This was related to the amount of KNO_3 using a calibration plot. Similarly, the equilibrium release (M_∞) of the KNO_3 was determined by measuring the conductivity of the release medium after nine days. The release data was analyzed kinetically using Eqs. (4) and (5), where W_t/W_∞ is replaced by M_t/M_∞ in both of the above equations.

3. Results and discussion

3.1. IR Spectral analysis

The IR spectra of unloaded and loaded graft polymers are depicted in Fig. 2. The spectra clearly marks the presence of amide group at 3420 cm^{-1} (N–H stretching) and 1680 and 1660 cm^{-1} (NH_2 bending), carboxymethyl cellulose unit bearing carboxylate ion at 1600 cm^{-1} (strong asymmetrical

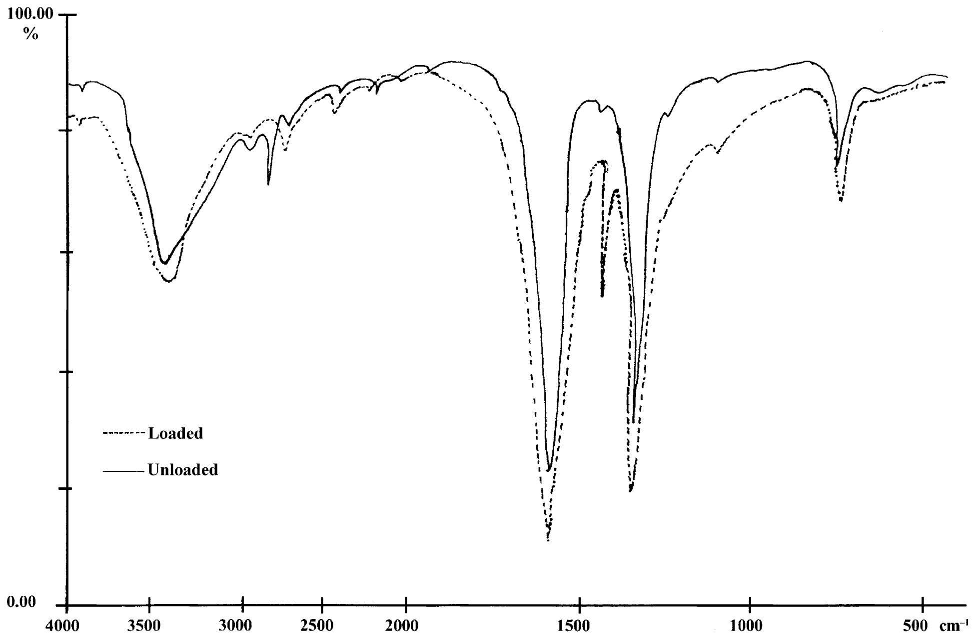

Fig. 2. IR spectra of unloaded and loaded gel.

stretching band) and 1450 cm^{-1} (O–H bending of carboxylate ion). The entrapment of KNO_3 is evident from vibrational frequency of nitrate ion at 1380 cm^{-1}. The broad bends from 1200 to 1000 cm^{-1} were due to sugar ring absorption (Bellamy, 1975).

3.2. Network studies

One of the most important structural parameter characterizing crosslinked polymer is M_c, the average molar mass between crosslinks which is directly related to the crosslink density. According to the theory of Flory and Rehner, for a perfect network,

$$M_c = -V_l d_p \frac{(V_s^{1/2} - V_{s/2})}{\ln(1 - V_s) + V_s + \chi V_s^2} \tag{7}$$

where M_c is the number average molar mass of the chain between crosslinks. V_1 is the molar volume of water (ml mol^{-1}), d_p is the polymer density (g ml^{-1}), V_s is the volume fraction of polymer in the swollen gel, χ is the Flory–Huggin's interaction parameter between solvent and polymer.

The swelling ratio is equal to $1/V_s$. Here, the crosslink density, q is defined as the mole fraction of crosslinked units.

$$q = M_o/M_c \tag{8}$$

where M_o is the molar mass of the repeating unit.

Other authors defined a crosslink density, V_e, as the number of elastically effective chains, totally included in a perfect network, per unit volume. V_e is simply related to q since

$$V_e = d_p N_A / M_C \tag{9}$$

The values of V and χ were taken from related literature. The density d_p of the gel was measured as 1.10 g cm^{-3} by pyknometry.

The value of M_c, q and V_e of the networks have been calculated and summarized in Table 2 for varying crosslinker contents.

3.3. Effect of hydrogel composition on swelling

3.3.1. Effect of CMC on swelling

CMC is a polysaccharide (Fig. 3) that contains different type of hydrophilic functional groups. When the amount of CMC in the feed mixture increases in the range 0.2–0.5 g, it is observed that the swelling ratio increases in the concentration range 0.2–0.4 g while it decreases beyond 0.4 g (Fig. 4). The results can be explained by the fact that increasing the proportion of hydrophilic polymer in the gel increases the affinity for water thus resulting in a greater swelling ratio. However, at much higher concentration of CMC (beyond 0.4 g) the density of network chains increases so much that both the diffusion of solvent molecules and relaxation of macromolecular chains are reduced. This explains the fall in the swelling ratio of the hydrogel. Similar type of results have also been obtained by other workers in swelling studies of hydrogels containing hydrophilic polymers (Gravier, Hyon, & Ikada, 1995).

Table 2
Network parameter of the hydrogel at varying crosslinker content

Crosslinker concentration chains (mM)	Average molecular wt. (M_c) ($M_c \times 10^{-5}$)	Crosslink density ($q \times 10^5$)	Elastically effective chains ($V_e \times 10^{-18}$)
0.064	4.08	17.3	1.62
0.13	7.18	9.8	0.92
0.19	2.61	27.1	2.53
0.26	2.32	30.5	2.84
0.39	1.8	39.4	3.68

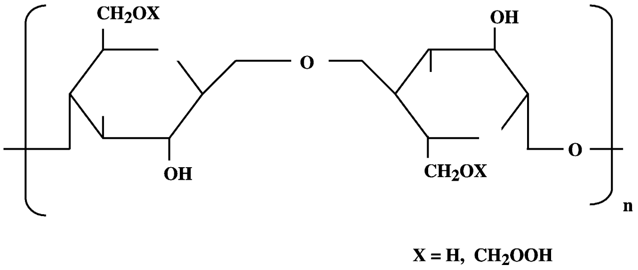

Fig. 3. Structural unit of CMC.

3.3.2. Effect of acrylamide

The effect of acrylamide content on the swelling behaviour of the hydrogel was studied by varying acrylamide concentration in the range 7.03–21.1 mM in the feed mixture. The results are presented in Fig. 5 which reveal that the swelling ratio continuously decreases with increasing concentration of the acrylamide in the feed mixture. The results can be explained by the fact that increasing content of the monomer in the feed mixture will result in a greater number of crosslinked polyacrylamide chains in the hydrogel network which decreases swelling.

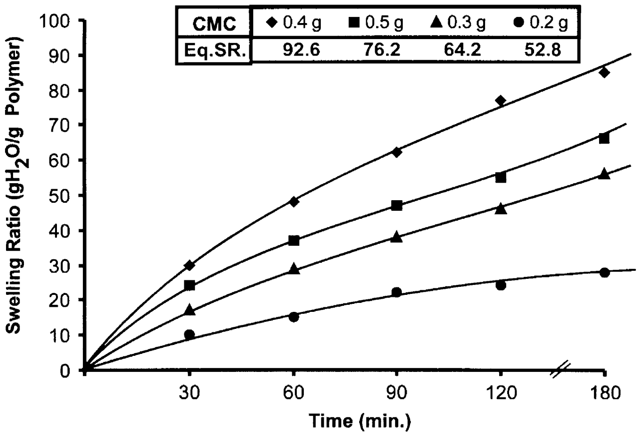

Fig. 4. Effect of CMC content of the hydrogel on its swelling behaviour.

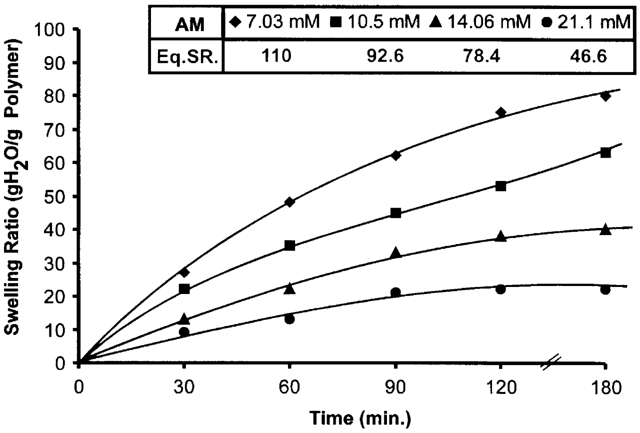

Fig. 5. Effect of acrylamide content of the hydrogel on its water sorption behaviour.

3.3.3. Effect of crosslinker

In the present study to investigate the effect of crosslinker on the swelling characteristics of the gel the concentration of MBA in the feed mixture was varied in the range 0.064–0.39 mM. The results are depicted in Fig. 6 which clearly indicate that the swelling ratio increases in the range 0.064–0.13 mM of MBA while beyond 0.13 mM the swelling ratio decreases. However, at higher concentrations of MBA the swelling is not much affected. The initial increase in the swelling ratio is because MBA is itself a hydrophilic monomer and, therefore, the swelling ratio increases with increasing concentration of MBA. However, beyond 0.13 mM the decrease observed is attributed to the fact that the increasing crosslinking density in the hydrogel lower the average molecular weight between crosslinks (Table I) and this curtails the free volumes accessible to the penetrant water molecules and thus the swelling ratio decreases. At higher amounts of MBA the hydrogel network chains become so inflexible that the swelling ratio is independent of MBA concentration. At higher concentration of MBA, the equilibrium swelling is attained earlier (120 min.) than for hydrogels containing lower amounts of MBA. This could be explained by the fact that at higher crosslink density the network chains are quite inflexible and, therefore, even at lower water uptake the hydrogel appears to have equilibrated. Similar observations have been noticed by us earlier (Bajpai, Bajpai, & Shukla, 2001).

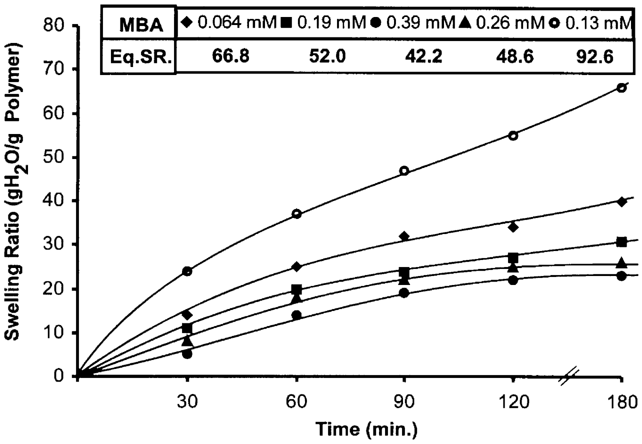

Fig. 6. Variation in the swelling ratio of the hydrogel with varying concentrations of the crosslinker (N,N′-methylenebisacrylamide).

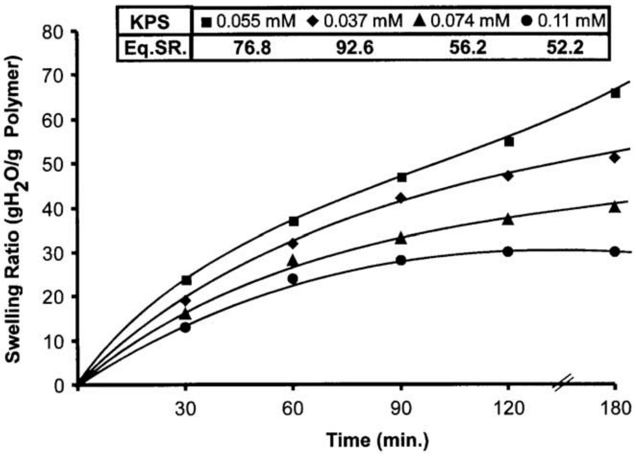

Fig. 7. Effect of initiator (potassium persulphate) concentration on the swelling ratio of the hydrogel.

3.3.4. Effect of initiator

In the present work the initiator has been varied in the feed mixtures in the concentration range 0.037–0.22 mM and the effect of this variation on the swelling ratio of the hydrogel has been studied. The results are shown in Fig. 7 which reveal that with increasing concentration of KPS, the swelling ratio decreases. The observed results expected since on increasing the KPS concentration the molecular weight of the resulting crosslinked PAM will decrease thus shortening the macromolecular chains and reducing the available free volumes within the hydrogel. This clearly brings about a decrease in the swelling ratio of the hydrogel.

3.4. Effect of pH

Most of the hydrogel systems are pH responsive (Seno, Lin, & Iwamoto, 1991) and, therefore, the pH of the immersion medium has direct control over the degree of swelling of the network. In the present work the pH of the external solution has been varied in the range 2.6–11.2 and the effect on the swelling ratio has been observed. The results are depicted in Fig. 8 which indicate that the swelling increases upto pH 8.8 while beyond this pH a drop in the swelling ratio is noticed. The results can be attributed to the fact that with increasing pH of the external solution the amide groups of crosslinked PAM chains undergo hydrolysis and are converted into carboxylic groups which begin to ionize as the pH increases. This obviously produces anionically charged polymer chains which because of the mutual repulsion among carboxylate ions ($-COO^-$) results in an expansion of the network chains leading to an increase in swelling. However, beyond a certain pH (8.8 in this case)

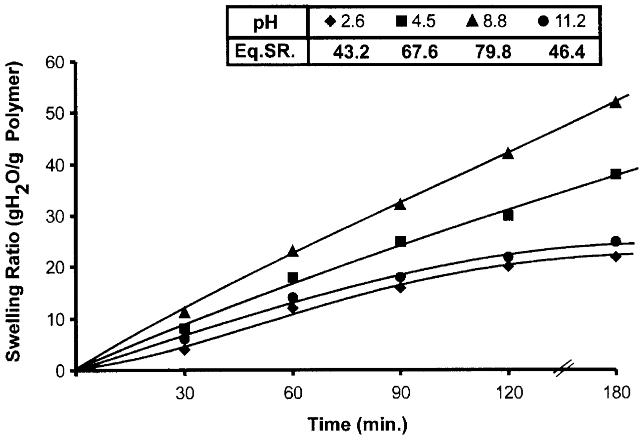

Fig. 8. Effect of pH of the bathing medium on the swelling ratio of the hydrogel.

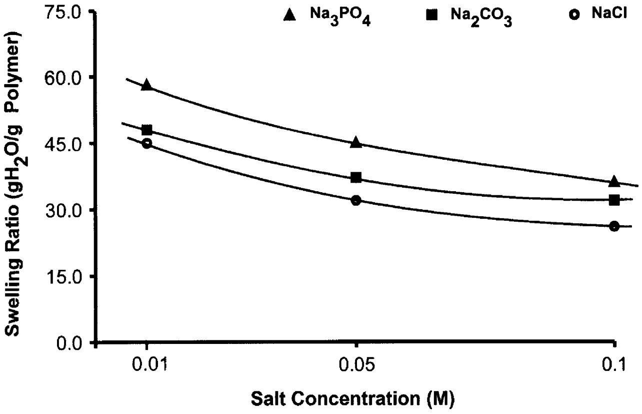

Fig. 9. Effect of ionic strength of the medium on the swelling ratio of the hydrogel.

swelling is found to decrease. The reason for the observed decrease may be that at higher pHs the ionic concentration becomes large in the external medium and, therefore, swelling decreases.

3.5. Effect of ionic strength

The presence of ions in the swelling medium has a profound effect on the swelling behaviour of the hydrogel. The underlying principal behind this ionic dependence of swelling is that it is the balance between the osmotic pressure of the swelling system and elastic response of the polymeric material that controls the extent of swelling. The osmotic pressure results from the difference between the mobile ion concentrations between the interior of the hydrogel network and the external immersion medium. Increasing the ionic concentration reduces the mobile ion concentration difference between the polymer gel and external medium (osmotic swelling pressure) which, in turn, reduces the gel volume, i.e. the gel shrinks.

In the present context the effect of salt concentration on the swelling ratio of the hydrogel has been investigated by increasing the ionic strength of the external solution by adding sodium salts of anions to the external solution in the concentration range 0.01–0.10 M. The results shown in Fig. 9 reveal that the swelling ratio decreases as the ionic strength of the medium increases.

The results also reveal that the relative effect of the added anions follows increases in the following order:

$$PO_4^{3-} < CO_3^{2-} < Cl^-$$

This sequence may be explained by the fact that PO_4^{3-} and CO_3^{2-} ions, being trivalent and divalent, respectively, will form ion-pairs with sodium ions and, therefore, will reduce their effective concentrations in external solution. This results in a fall in the water sorption capacity but is still less than that produced by Cl^- ions which because they are monovalent have greater effective concentration in the external solution. This explains the observed order of relative effectiveness of the added salts.

3.6. Analysis of dynamic sorption data

The phenomenon of water sorption by hydrogel depends mechanistically on the diffusion of water molecules into the gel matrix and subsequent relaxation of macromolecular chains of the hydrogel.

Water transport mechanism in a swelling hydrogel is greatly contributed to by numerous factors such as equilibrium water content, swelling rate, chemical composition of the hydrogel system, etc. In the present study with the help of Eqs. (2) and (3) the values of the swelling exponent '*n*' and diffusion constant '*D*' have been calculated under varying experimental condition and the respective data is summarized in Table 3.

It is clear from the Table that on increasing the concentration of CMC in the range 0.2–0.5 g, the swelling exponent '*n*' decreases from 0.71–0.57 while the diffusion constant increases from 12.2 to 24.4 $cm^2 s^{-1}$. The decrease in the swelling exponent suggests that the swelling process tends to shift from a more non-Fickian to a less non-Fickian nature. This suggests that with increasing CMC content chain relaxation becomes less important in controlling swelling. In other words, at higher CMC content the hydrogel chains undergo more rapid relaxation than at lower CMC concentration. This may be because the bulky CMC chains do not pack efficiently and, therefore, show more freedom for chain relaxation. The diffusion constant is found to increase as due to the greater chain relaxation the water molecules can diffuse more rapidly.

With increasing an acrylamide concentration in the feed mixture (7.03–21.1 mM) the value of *n* is found to decrease in the non-Fickian range and finally attain a Fickian value. The reason for such an observation is quite obvious as increasing PAM chains in the network will certainly gives rise to a compact arrangement which consequently permits a slow diffusion of the water molecules into the gel matrix. Thus, with increasing PAM content the hydrogel displays

Table 3
Various kinetic parameters of the swelling process at different hydrogel compositions

Swelling ratio equilibrium	CMC (g)	AM (mM)	MBA (mM)	KPS (mM)	n	$D \times 10^8$ $cm^2\,s^{-1}$	Mechanism
52.8	0.2	10.5	0.13	0.037	0.71	12.2	More non-Fickian
64.2	0.3	10.5	0.13	0.037	0.63	17.0	More non-Fickian
92.6	0.4	10.5	0.13	0.037	0.60	18.3	More non-Fickian
76.4	0.5	10.5	0.13	0.037	0.57	24.4	Less non-Fickian
110	0.4	7.03	0.13	0.037	0.75	24.4	Non-Fickian
92.6	0.4	10.5	0.13	0.037	0.60	18.3	Non-Fickian
78.4	0.4	14.06	0.13	0.037	0.58	17.0	Less non-Fickian
46.6	0.4	21.1	0.13	0.037	0.50	10.8	Fickian
66.8	0.4	10.5	0.064	0.037	0.56	19.2	less non-Fickian
92.6	0.4	10.5	0.13	0.037	0.60	18.3	Less non-Fickian
52.0	0.4	10.5	0.39	0.037	0.80	17.6	Non-Fickian
42.2	0.4	10.5	0.39	0.037	0.83	17.0	More non-Fickian

decreasing non-Fickian or relaxation controlled swelling process. At the greatest AM content, the swelling process becomes truly diffusion controlled, showing an exponent of 0.5. As is clear from the Table as the gel compacts the diffusion of water molecules into the hydrogel will becomes more difficult and as a consequence the diffusion coefficient decreases.

When the concentration of crosslinker (MBA) increases in the feed mixture the crosslink density obviously increases in the hydrogel (Table 1). It is clear from the Table 3 that increase in the MBA concentration in the hydrogel results in an increase in the swelling exponent n. This means that a hydrogel with a higher crosslink density shows a more relaxation controlled swelling process. In other words, the swelling mechanism shifts from less non-Fickian value to more non-Fickian value as shown in Table 2. This is because as in a densely crosslinked hydrogel the chain relaxation will be relatively more difficult than the water diffusion process. It can also been seen from the Table that with increasing crosslinker concentration the diffusion constants continually decrease. The observed fall in the diffusion coefficient is quite expected because with increasing crosslink density in the hydrogels the diffusion of water molecules will become more difficult.

3.7. Release study

The release kinetics of a loaded hydrogel is closely related to its water sorption kinetics (Colombo et al., 1999a, b) since a highly swelling hydrogel should release a greater amount of solute entrapped within the gel. Our results on release also support the above fact and are consistent with the water-sorption results. In the following sections we will discuss the effects of composition of the hydrogel on the release kinetics.

3.7.1. Effect of CMC

The effect of CMC content in the loaded hydrogel on its release behaviour is shown in Fig. 10 which reveal that the released amount of KNO_3 (in mg) increases when the CMC content increases in the feed mixture in the range 0.2–0.4 g. However, beyond 0.4 g, a fall in the released amount is noticed. The results are quite obvious and can be explained on the basis of the swelling behavior of the hydrogels which also obey the same pattern and have already been explained in the earlier part of the paper.

3.7.2. Effect of AM

As has been stated earlier the swelling ratio decreases with increasing AM content in the feed mixture in the range 7.03–21.1 mM. When the release results are compared with those of the swelling then it is noticed that the released amount of KNO_3 increases from 7.03–10.3 mM while beyond it a decrease in the released amount is observed (Fig. 11). The reason for the lower release rate at 7.03 mM of AM content in the hydrogel may be because for a low network density of the hydrogel the PAM chains are well separated and, therefore, the available free volume between the chains is quite large. In this situation, therefore, one can not rule out the possibility of re-loading of released KNO_3 from the solution phase to the interior of the hydrogel

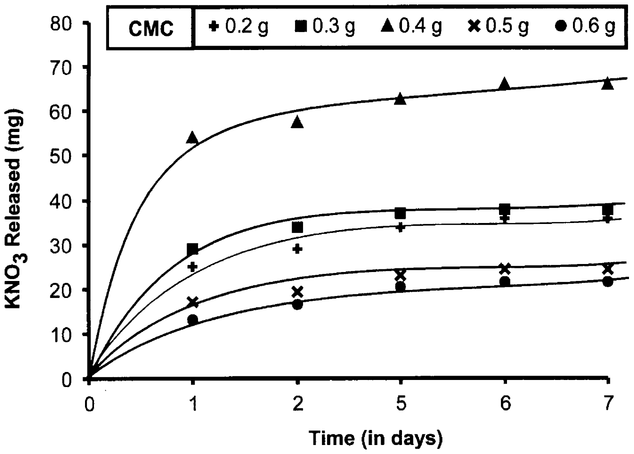

Fig. 10. Effect of CMC variation in the hydrogel on the released amount of KNO_3.

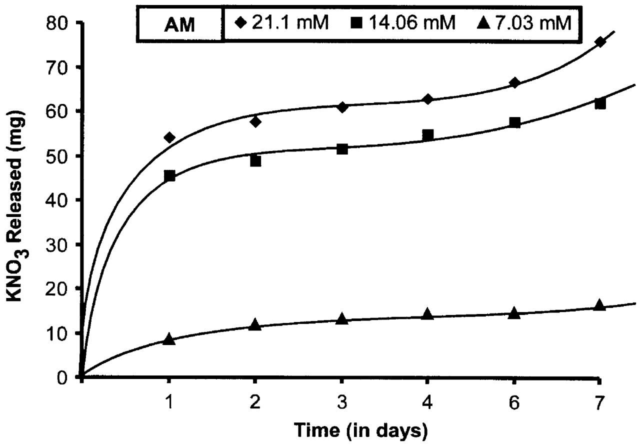

Fig. 11. Effect of acrylamide content in the hydrogel on the released amount of KNO_3.

matrix. This will obviously result in a lower amount of released KNO_3.

3.7.3. Effect of crosslinker

The crosslink density of a hydrogel is intimately related to the degree of swelling and consequently, the release behavior of the hydrogel. In the present study the effect of crosslinker on the release rate of KNO_3 has been investigated by adding MBA to the feed mixture in the concentration range 0.064– 0.39 mM. The release kinetics of the respective gels were then measured. The results are shown in Fig. 12 which are consistent with the swelling behaviour. It reveals from the Figure that a maximum release rate and release amount of KNO_3 is observed at 0.13 mM of the crosslinker while with increasing MBA the released amount decreases. The results are quite obvious as for a densely crosslinked gel the release of a solute is more difficult then from a loosely crosslinked network. Similar results have also been reported by other workers (Cohn, Aronhime, & Abdo, 1992).

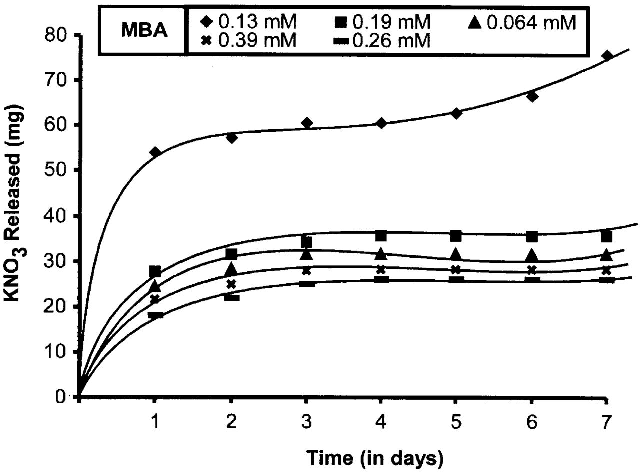

Fig. 12. Effect of crosslinker (MBA) content in the hydrogel on the released amount of KNO_3.

4. Conclusions

CMC, in conjugation with crosslinked polyacrylamide (PAM) forms a novel type of highly swelling hydrogel which imbibes as much as 60 g water per g of dry gel. The hydrogels display interesting swelling results. It is found that the swelling ratio increases in the concentration range (0.2–0.4 g) of CMC while beyond it a fall in the swelling ratio is observed. The sorption process also shifts from more non-Fickian to a less non-Fickian nature. The increase in monomer (acrylamide) content in the hydrogel lowers the swelling ratio while the kinetic data suggests that the swelling process tends to become more and more non-Fickian in nature. The crosslinker (MBA) has an inverse effect on swelling ratio and its increasing content in the hydrogel results in a more non-Fickian water sorption. The swelling of the hydrogel also varies with the pH and ionic strength of the immersion medium.

The dry hydrogel loaded with potassium nitrate swells in aqueous medium releasing the salt entrapped within the matrix. The release results correlate well with the swelling results.

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