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Acrylamide-Potassium Methacrylate Superabsorbent Copolymers: Synthesis, Swelling Kinetics And Effect Of Reaction Parameters On Swelling Behaviour

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ABSTRACT

A series of superabsorbent copolymers based on acrylamide, potassium methacrylate were synthesized by simultaneous free radical aqueous polymerization using diallyl phthalate (DP) and N,N′-methylene-bis-acrylamide (MBA), ammonium persulfate (APS), N,N′-tetramethylethylenediamine (TMEDA) as crosslinkers, initiator and activators, respectively. The influence of reaction parameters such as AAM/KMA ratio, crosslinker, initiator, activator concentrations and polymerization temperature on the swelling behaviour was investigated. The swelling kinetics of the copolymers was studied at different temperatures The swelling studies revealed that DP crosslinked superabsorbent copolymers have shown higher swelling capacity in all compositions than the MBA crosslinked superabsorbent copolymers. In the present investigation, two different crosslinking agents were employed to study their influence on swelling capacity. The effect of crosslinker concentration on morphology of the copolymers also supports the swelling studies. In addition the salt sensitivity of the copolymers was observed at different crosslinker concentrations. To attain maximum swelling capacity to the AAM-KMA copolymers optimization of reaction conditions were investigated. The copolymers de-swelling as well as pH effect on the copolymers were also studied in detail.   © 2006 Trade Science Inc. - INDIA

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INTRODUCTION

In the recent years there has been considerable interest in water swellable "Superabsorbing" polymers capable of absorbing and holding large amounts of water. Superabsorbents are one of the most promising types of polymers being used for the new developments in the area of functional polymers[1]. The hydrophilicity to the polymers is attributed due to the presence of hydrophilic functional groups such as hydroxyl, carboxylic acid, and amines[2,3]. The excellent properties such as, lack of toxicity, high swelling capacity in aqueous environment, hydrophilicity, and biocompatibility have enhanced their wide spread applications in different fields including agriculture and horticulture, health, bioengineering, pharmaceutical, drug delivery, food industry, and other advanced technologies[4-10]. Further the superabsorbents were also used for the adsorption of some cationic dyes, uranyl ions, and serum albumin[11-13].

Our earlier studies revealed the effective utilization of the acrylamide based tercopolymers for agriculture and horticultural applications. The optimization of the reaction conditions also studied in detail by varying the reaction parameters such as monomer, crosslinkers and initiator concentrations as well as the reaction temperatures[4,5,14-17]. In many of the reports the water absorbency and water retention capacities were investigated as one of the most important properties of the SAPs for the above applications. The authors have contributed significant work for the modification of SAPs to enhance their absorbency, gel strength and sharp absorption rate[18-24]. The kaolinite grafted superabsorbent copolymer was a suitable method to reduce the cost of the production and also the way to get an increase in the swelling properties of superabsorbents[20]. Wu et al[25-27] have reported the synthesis as well as the properties of starch-g-acrylamide/kaolinite and poly(acrylic acid)/mica superabsorbent composites and also the influence of the hydrophilic groups on water absorbency of starch-g-acrylamide/kaolinite superabsorbent composites in detail. Recently much attention is focused on the development of composite[20,25-27] and porous superabsorbents[28-30] in the superabsorbent research field to obtain sharp and high swelling

capacity as well as to reduce the cost of the production of the material.

A wide range of synthetic methods are available in the literature for the preparation of superabsorbent polymers but the simultaneous polymerization with redox- initiation method was recognized as a significant method in the preparation of SAPs due to its simplicity. A few series of copolymers were synthesized by simultaneous polymerization and their swelling and diffusion parameters were investigated[31-35]. W.J.Zhou and co-workers have studied the synthesis and swelling properties of copolymers of acrylamide with anionic monomers[31] and crosslinked poly [acrylamide sodium methallylsulfonate-sodium acrylate], poly(AM-MSAS-AA) gels[32]. S.K.Bajpai et al. have reported the synthesis and characterization of AAM/Itaconic acid for oral drug delivery of peptide[33] as well as studied the swelling and de-swelling behaviour of poly(acrylamide-co-maleic acid) [34]. E.Karadag et al. have reported the synthesis as well as the swelling behaviour, and swelling/diffusion characteristics of poly(acrylamide-co-crotonic acid)[35]. B.Isik has reported the swelling behaviour and the diffusion characteristics of acrylamide-acrylic acid hydrogels[27]. The relationship between water absorbency and reaction time of neutralized acrylic acid was investigated by J.Chen and Y.Zhao using potassium persulfate/TMEDA initiating system[36].

In our earlier studies, we have reported the synthesis of superabsorbent tercopolymers by free radical polymerization using ammonium persulfate as initiator at 80^0C[14-17]. More recently we reported the synthesis and swelling/diffusion kinetics of AAM-KMA copolymers prepared by APS/TMEDA redox-initiation[37]. In the present investigation, we report the influence of various reaction parameters such as, monomer concentration, crosslinker concentration, initiator concentration and temperature on water absorbency of the AAM-KMA superabsorbent copolymers. The swelling kinetics of superabsorbents prepared by varying the composition of KMA was studied at different temperatures. The saline sensitivity, pH effect and de-swelling studies were also investigated.

EXPERIMENTAL

Materials

Acrylamide (AM), ammonium persulfate (APS), and N,N′-methylenebisacrylamide were received from S.D. Fine Chem (Bombay, India). Methacrylic acid (MA), Diallyphthalate (DP) and N,N,N′,N′-tetramethylethylenediamine (TMEDA) were obtained from Aldrich (Sigma Aldrich Chemicals Private Limited, India). All the chemicals were used as received. Potassium methacrylate was prepared by complete neutralization of methacrylic acid with potassium hydroxide[37]. Double distilled water was utilized for the copolymerizations as well as for the swelling studies.

SAP preparation

AAM-KMA SAPs were prepared according the experimental procedure reported by us[37]. The method follows the simultaneous free-radical polymerization. The preparation involves the addition of potassium methacrylate, crosslinker, ammonium persulfate and TMEDA sequentially to the acrylamide solution dissolved in 2 ml of distilled water at room temperature. After adding all reaction ingredients the polymerization mixture was pored in PVC straws (3 mm dia) to obtain the superabsorbent polymers in the form of rods. Then the superabsorbents were cut into pieces of 3-4 mm length and dried in air and then under vacuum.

Swelling measurements[38]

Accurately weighed dry superabsorbent polymers (M_d) (40-50 mg) were soaked in a 100 ml beaker containing double distilled water or saline solution or pH solutions until they reach the equilibrium at room temperature. After attaining the swelling equilibrium the gels were separated from the solvent and the residual water was removed superficially with filter paper and then the swollen superabsorbents were weighed (M_e). The swelling ratio (S%) (g/g) or equilibrium swelling percentage of superabsorbents was calculated using the equation given below (1).

Swelling ratio (S%) = [(M_e − M_d)/M_d]   (1)

where M_e and M_d denote the weight of the swol-

len superabsorbent at equilibrium and the weight of the dry superabsorbent at time 0, respectively.

Preparation of pH solutions[38]

Buffer solution 1 was prepared by mixing 12.3 g of anhydrous boric acid (0.20M) and 10.51 g of citric acid (0.05M) in 1000 ml distilled water and the buffer solution 2 was prepared by dissolving 38.01 g of tri-sodium phosphate in 1000 ml distilled water. In order to prepare a specific buffer solution the two solutions (pH, solution 1 and 2) were mixed at different volumes based on Shugar and Dean[38].

RESULTS AND DISCUSSION

The diffusion process represents the affinity between the polymer networks and external solution. The absorption mechanism of superabsorbent polymers was elucidated in the form of their swelling behaviour, which is caused by diffusion process. The absorption of superabsorbent polymers depends on crosslinking density, polymer network behaviour, strength of the hydrophilic groups, elasticity of the polymer networks and the type of solvent and strength of the external solution as well as the characteristics of the external solution etc. The absorption of external solutions can be balanced by following three main forces, (1) free energy between chain networks of the polymers and external solvent; (2) electrostatic repulsion (domain effect); and (3) elastic retractile response of the networks. The first two forces promotes the swelling capacity and the third one causes to decrease the swelling capacity of the superabsorbent polymer.

Effect of monomer concentration

Among the influencing factors, the hydrophilic monomer concentration plays a pivotal role to improve the swelling properties of superabsorbent copolymer[4,5,14-17]. As shown in TABLE 1, with increase of potassium methacrylate content in the acrylamide superabsorbent copolymer the swelling ratio value increased enormously. It was noticed that the superabsorbent copolymers crosslinked with DP have shown higher swelling ratio than the MBA crosslinked superabsorbent copolymers at every com-

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TABLE 1: Influence of KMA content on the swelling behaviour of AAM-KMA superabsorbent copolymer[a]

SAP code	KMA (g)	Swelling ratio (g/g)	Gelation time
DP 1	-	06.71	11 min
DP 2	0.10	87.00	37 min
DP 3	0.15	107.88	38 min
DP 4	0.20	184.71	40 min
DP 5	0.25	236.50	42 min
DP 6	0.30	196.85	48 min
DP 7	0.5	202.75	64 min
MBA 1	-	09.80	30 sec
MBA 2	0.10	55.75	52 sec
MBA 3	0.20	50.54	125 sec
MBA 4	0.25	80.35	158 sec
MBA 5	0.30	57.46	184 sec
MBA 6	0.40	57.68	255 sec
MBA 7	0.50	46.91	285 sec
MBA 8	1.00	11.38	-

[a]Reaction conditions: AAM = 1.00 (g) 0.014 mole; DP = 8.12×10^{-5} mole (or) MBA = 7.45×10^{-5} mole; APS = 4.38×10^{-5} ; TMEDA = 8.60×10^{-5}; Temperature = room temperature (=25^0C)

position of AAM/KMA. In both series of superabsorbent copolymers, it was observed that the swelling ratio increases with increase of potassium methacrylate content upto an optimum level and then decreases slowly with further increase in the potassium methacrylate content. Our earlier studies also revealed similar effect on absorbency behaviour of the superabsorbent copolymers with increment of hydrophilic units in the AAM copolymer[14-17]. It is identified that the copolymers prepared having AAM:KMA 1.00:0.25% composition have shown maximum absorbency.

The swelling kinetics of the superabsorbent copolymers on temperature dependency in distilled water was studied at different temperatures and the results were plotted in figure 1 and 2 for DP and MBA series, respectively. For both crosslinked AAM-KMA superabsorbent copolymer series, the highest swelling ratio was observed at 45^0C and the lowest at 10^0C. The reason to increase in the swelling ratio at higher temperatures can be explained due to expansion of polymer chains, which can hold large

Figure 1: Swelling isotherms of AAM-KMA superabsorbent copolymers crosslinked by DP
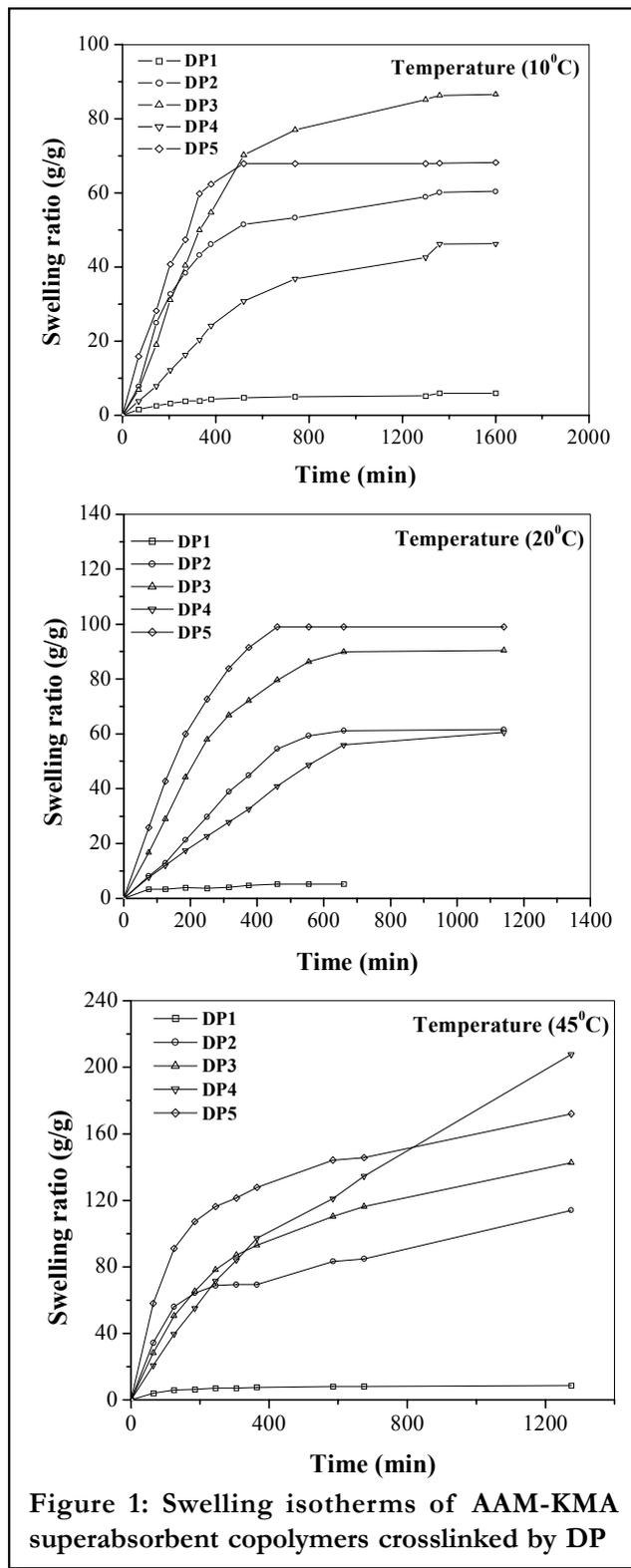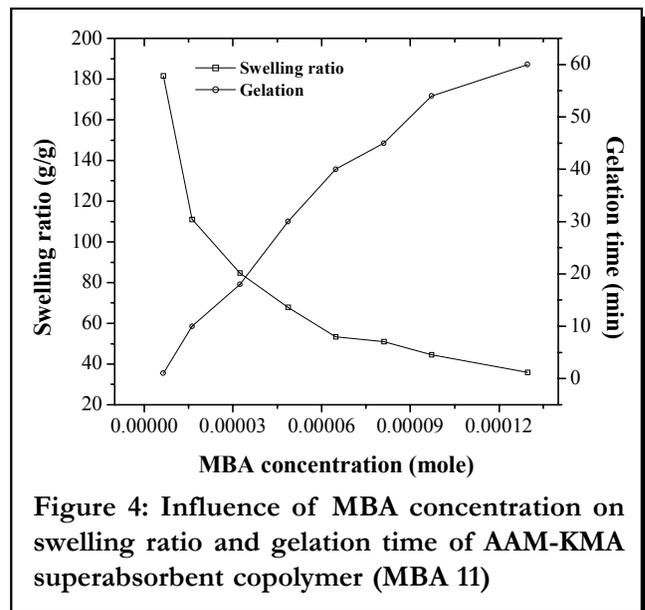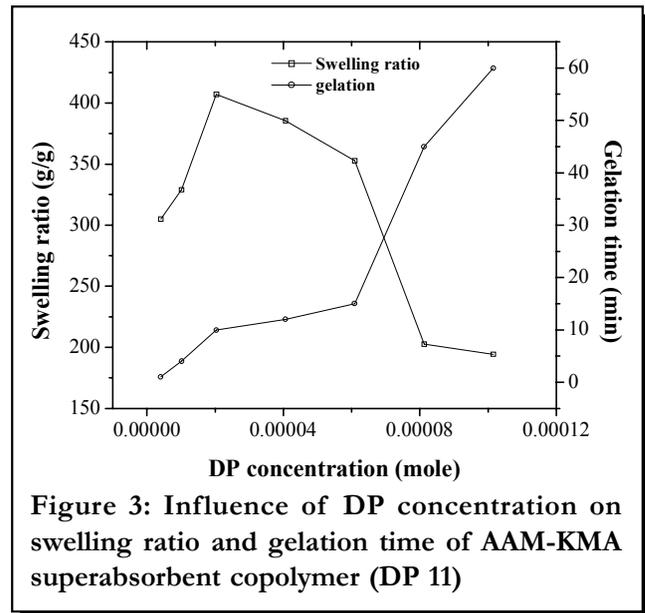
amount of water. This behaviour of SAPs may be due to their association/dissociation of the hydrogen bonding by the hydrophilic groups in the copolymer.

Figure 3: Influence of DP concentration on swelling ratio and gelation time of AAM-KMA superabsorbent copolymer (DP 11)

Figure 4: Influence of MBA concentration on swelling ratio and gelation time of AAM-KMA superabsorbent copolymer (MBA 11)

Figure 2: Swelling isotherms of AAM-KMA superabsorbent copolymers crosslinked by MBA
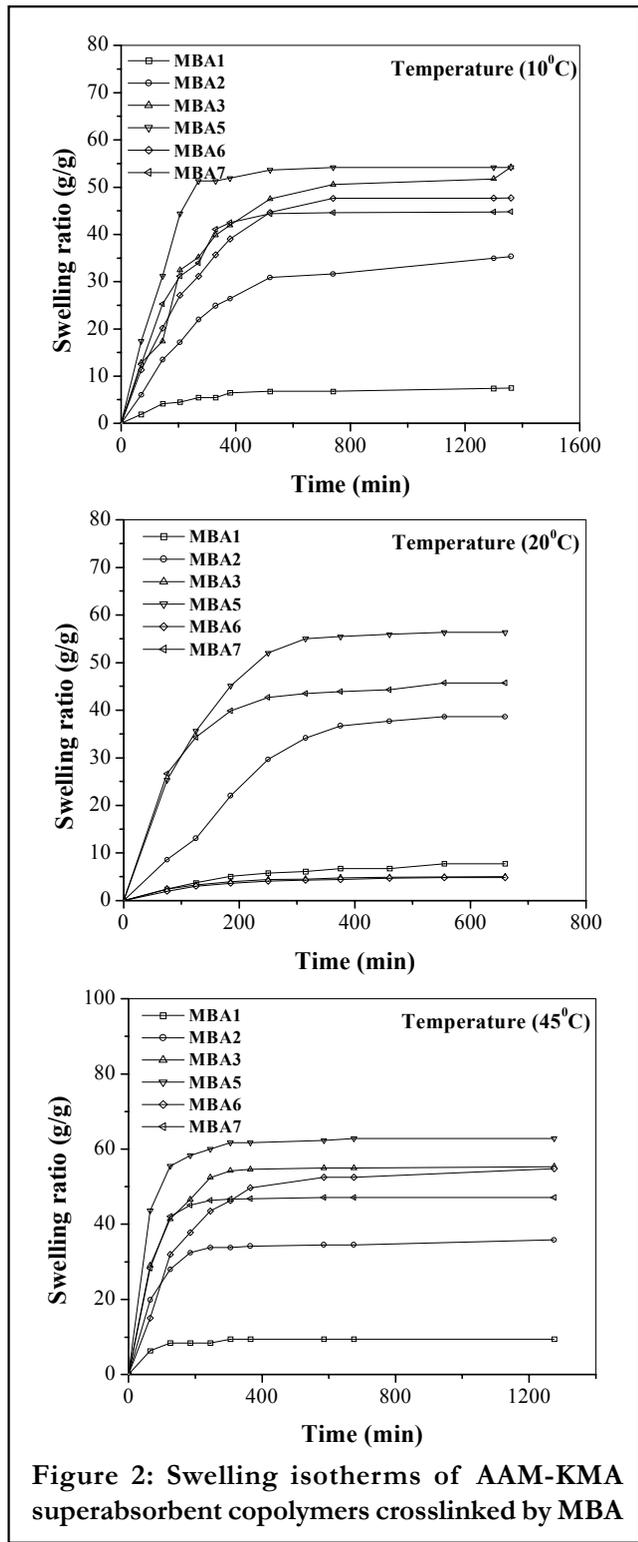
Effect of crosslinking agent type and concentration

The crosslinking agent contains two or more double bonds, which can participate in the free radical polymerization with the acrylic monomers and form crosslinking networks permanently between the polymeric chains[35,39]. The type of crosslinking agent directly affect the swelling behaviour of any superabsorbent polymer. TABLE 1 reveals that DP crosslinked copolymers have shown higher swelling ratios than the MBA crosslinked copolymers. This may be due to the difference in the formation of crosslinking networks in the copolymers. Figure 3 and 4 shows the swelling ratio of superabsorbent copolymers as a function of crosslinker concentration and gelation time. It was observed from the figure 3, with increase of DP concentration the swelling ratio was increased from 305.00 g/g (4.06 x 10^{-6}
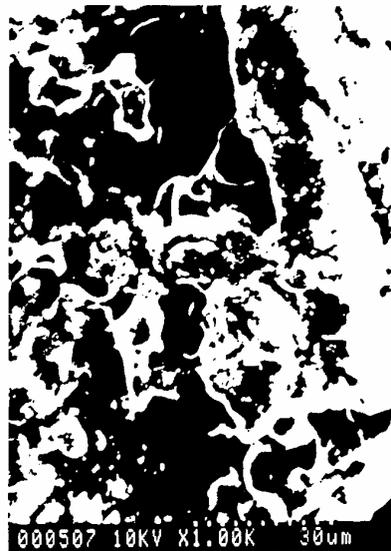
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TABLE 2: AAM-KMA superabsorbent copolymer reaction parameters variation

SAP code	AAM	KMA	DP	MBA	APS	TMEDA
DP 11	1.40×10^{-2}	4.00×10^{-3}	-	-	4.38×10^{-5}	8.60×10^{-5}
DP 12	1.40×10^{-2}	4.00×10^{-3}	2.03×10^{-5}	-	-	8.60×10^{-5}
DP 13	1.40×10^{-2}	4.00×10^{-3}	2.03×10^{-5}	-	4.38×10^{-5}	-
MBA 11	1.40×10^{-2}	2.00×10^{-3}	-	-	4.38×10^{-5}	8.60×10^{-5}
MBA 12	1.40×10^{-2}	2.00×10^{-3}	-	6.48×10^{-6}	-	8.60×10^{-5}
MBA 13	1.40×10^{-2}	2.00×10^{-3}	-	6.48×10^{-6}	1.75×10^{-5}	-

Figure 5: SEM micrographs of AAM-KMA superabsorbent copolymers crosslinked by (a) 1.01 x 10⁻⁵ moles (b) 1.01 x 10⁻⁴ moles of DP and (c) 1.621 x 10⁻⁵ moles (d) 9.7 x 10⁻⁵ moles of MBA

moles) to 406.98 g/g (2.03 x 10^{-5} moles) and gradually decreased with further increase in DP concentration. The higher swelling ratio at 2.30 x 10^{-5} moles of DP crosslinked copolymer can be explained as at this concentration there may be formation of well defined network structure in the copolymer. The swelling ratio decreases significantly with increase of the MBA concentration as shown in figure 4. With increase of MBA concentration in the copolymer network the difference in the formation of structure leads to decrement of the swelling ratio of the copolymer. This was identified in the graph that at lower concentration of MBA crosslinker, the formed copolymer have shown higher swelling capacity. This may be due to higher reactivity and higher solubility of MBA crosslinker in the reaction mixture. This behaviour can be explained on the basis of formation of more crosslinks in the copolymer chains with increase in the concentration of crosslinker. This increase in the crosslink density decreases the space between the polymer chains and thereby suppressing the swelling of the superabsorbents[14-17]. It is also observed that the MBA crosslinked copolymers have very stiff gels in all the concentrations where as the DP crosslinked copolymers shows smooth gels. The gelation time was also increased in both the series of copolymers as the crosslinker concentration increases. The gelation studies indicate that the higher concentration of crosslinkers require higher polymerization times to form crosslinks in the entire polymeric chains.

The formation of higher crosslink density in the copolymer chains at higher concentrations of the crosslinker was verified by scanning electron microscopic (SEM) studies. The gold-coated surfaces of the superabsorbent copolymers were analyzed using Hitachi-520 Scanning Electron Microscope, Japan. The SEM micrographs of AAM-KMA superabsorbent copolymers are presented with two different concentrations of DP and MBA crosslinkers as shown in figure 5. It is a clear evidence from the SEM micrographs of figure 5 (a) and (c) that the copolymers prepared with lower concentrations of crosslinkers have loose crosslink structure with lower crosslink density, where as the copolymers crosslinked with higher concentrations have shown higher crosslink

density as shown in figure 5 (b) and (d).

The salt concentration and charge valencies significantly affects the swelling behaviour of the absorbents[14-17,31,32]. The influence of different concentrations of sodium chloride solution on swelling behaviour of AAM-KMA superabsorbent polymers was investigated. Figure 6 and 7 represents the swelling ratio of AAM-KMA superabsorbent copolymers as a function of crosslinker concentration in different concentrations of saline solutions. From figures
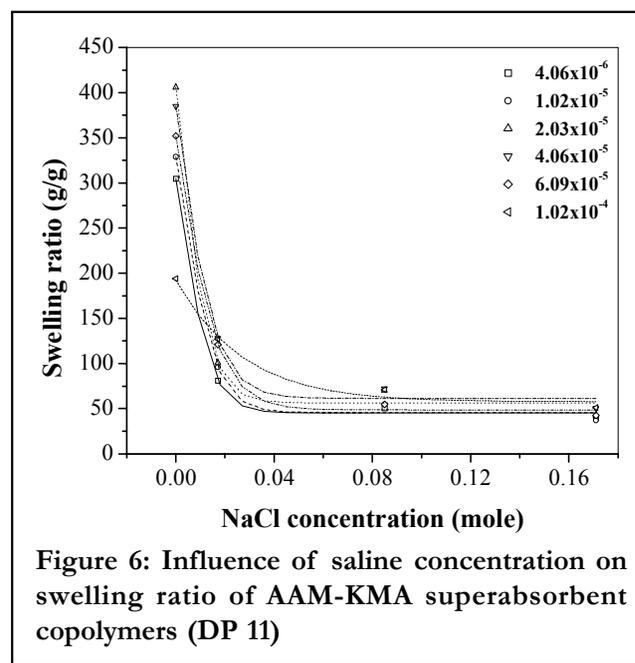

Figure 6: Influence of saline concentration on swelling ratio of AAM-KMA superabsorbent copolymers (DP 11)
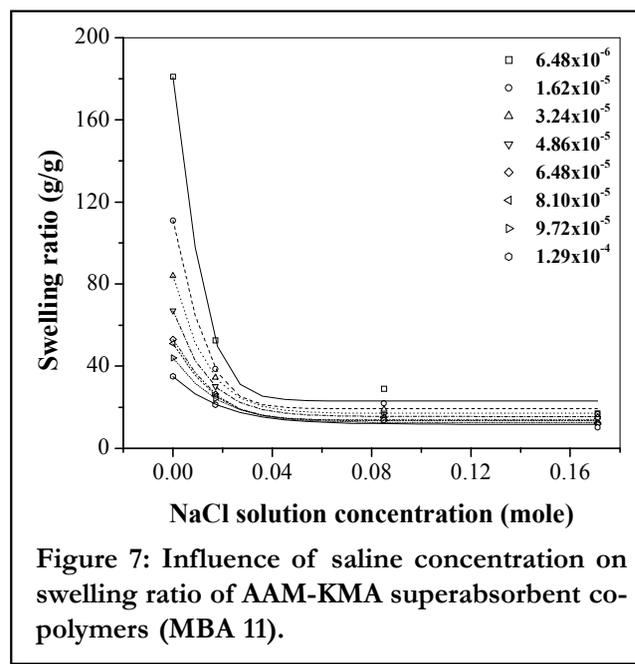

Figure 7: Influence of saline concentration on swelling ratio of AAM-KMA superabsorbent copolymers (MBA 11).

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6 and 7, it is clearly observed that the swelling ratio of SAPs decreased in salt solution as ionic concentration of the salt solution increases. This is because of the decrement in expansion of the gel networks due to repulsive forces of counter ions on the polymeric chain shielded by the bound ionic charge. Due to this interaction, the osmatic pressure difference between the gel network and the external solution decreased with an increase in the ionic strength of the saline concentration[14-17,31,32]. Similar behaviour was verified in the saline swelling behaviour with the copolymers such as, poly(sodium acrylate-sodium 2-acrylamido-2-methyl propane sulfonate), poly(SA-NaAMPS), poly(sodiumacrylate-hydroxylethyl methacrylate), poly(SA-HEMA), poly[sodium acrylate-3,3-dimethyl(methacryloyloxy ethyl) ammonium propane sulfonate] poly(SA-DMAPS) copolymers[40-42]. From this study it is clearly indicated that the AAM-KMA superabsorbent copolymers shows saline sensitivity. The salt sensitivity can be evaluated by dimensionless factor (α). The dimensionless factor (α) is the ratio of absorption at a given salinity to salt free water[43]. The α values for different saline concentrations are given in TABLE 3. The results indi-

TABLE 3: Dependency of dimensionless swelling factor (α), on the crosslinker concentration at various saline concentrations

Crosslinker concentration (mole)	Dimension less factor (α)		
	($\alpha_{0.0171}$)	($\alpha_{0.085}$)	($\alpha_{0.171}$)
DP			
4.06×10^{-6}	0.265	0.165	0.131
1.02×10^{-5}	0.290	0.166	0.111
2.03×10^{-5}	0.248	0.172	0.104
4.06×10^{-5}	0.332	0.187	0.131
6.09×10^{-5}	0.344	0.155	0.120
1.02×10^{-4}	0.655	0.365	0.264
MBA			
6.48×10^{-6}	0.290	0.160	0.094
1.62×10^{-5}	0.347	0.197	0.150
3.24×10^{-5}	0.410	0.225	0.180
4.86×10^{-5}	0.449	0.245	0.218
6.48×10^{-5}	0.495	0.285	0.234
8.10×10^{-5}	0.501	0.294	0.236
9.72×10^{-5}	0.548	0.304	0.267
1.29×10^{-4}	0.605	0.390	0.290

cate that the sensitivity of absorbance to changes in salinity is decreased, as the crosslinker concentration decreases.

Effect of initiator and activator

It is known that initiator or initiating system will have influence on crosslink network structure and phase behaviour of superabsorbent polymers. Further they also contribute for the inhomogeneity in the polymer system[44]. In the present studies, the polymerization reaction of AAm, KMA starts with the reaction between APS and TMEDA and then follows simultaneous free-radical polymerization in the presence of crosslinker. In recent years, similar type of studies was carried out for starch-g-acrylamide-kaolinite SAP composites (very high water absorbency value of 4000 g/g) using ceric ammonium nitrate/starch[20], acrylamide/acrylic acid using KPS/TMEDA[22], acrylamide/crotonic acid using APS/TMEDA[35], poly(vinyl alcohol) and poly(isopropyl-acrylamide) with APS/TMEDA[45], N-isopropyl-acrylamide sponge gels with KPS/TMEDA[46]. As shown in figure 8, as APS concentration varies in the polymerization reactions, the swelling ratio and gelation times varies and at one concentration of APS the polymer formed showed higher swelling ratio than other concentrations. The optimum concentration for obtaining higher swelling ratio was found to be 5.69×10^{-5} moles (406 g/g). Gelation time of
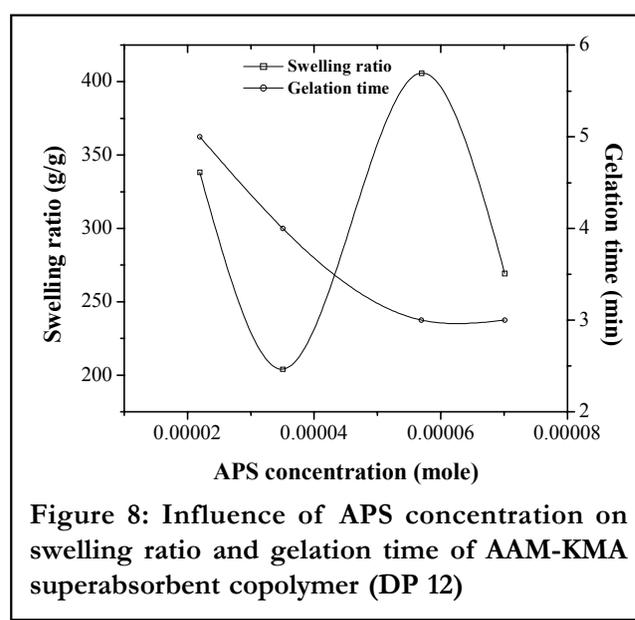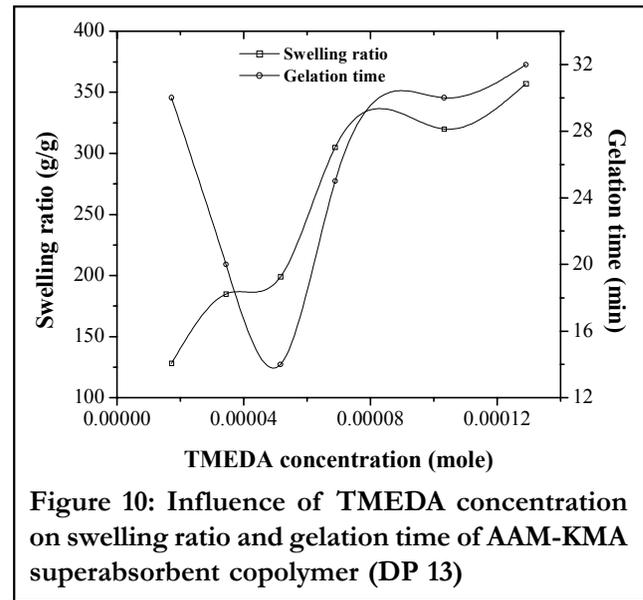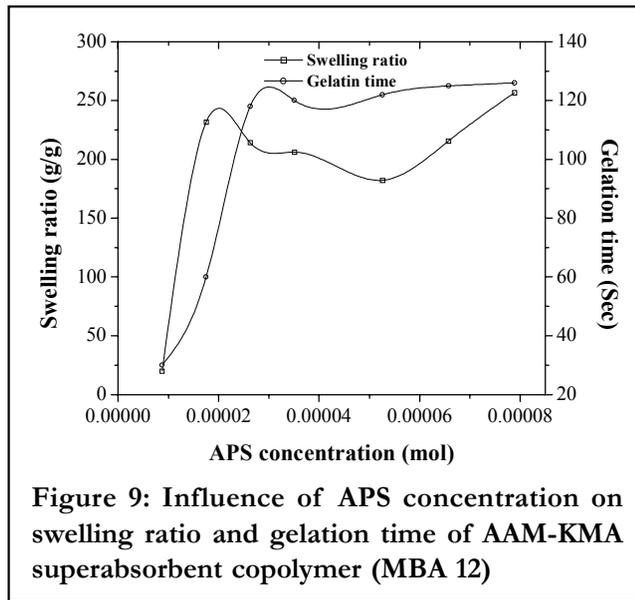

Figure 8: Influence of APS concentration on swelling ratio and gelation time of AAM-KMA superabsorbent copolymer (DP 12)

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Figure 9: Influence of APS concentration on swelling ratio and gelation time of AAM-KMA superabsorbent copolymer (MBA 12)

Figure 10: Influence of TMEDA concentration on swelling ratio and gelation time of AAM-KMA superabsorbent copolymer (DP 13)


Figure 11: Influence of TMEDA concentration on swelling ratio and gelation time of AAM-KMA superabsorbent copolymer (MBA 13)

the reactions decreased as the APS concentration increased. Similar behaviour was also observed in the case of MBA crosslinked superabsorbent copolymers. Figure 9 shows the swelling ratio and gelation time of the copolymers as a function of APS concentration. The swelling ratio of the MBA crosslinked copolymers was increased with increase of APS concentration from 8.76×10^{-6} moles to 1.75×10^{-5} moles (19.94 to 231.81 g/g) and further increase in the concentration of APS the swelling ratio decreased slightly and found higher swelling ratio 256.61 g/g at 7.88×10^{-5} moles of APS. Very low gelation time i.e., 30-126 sec was noted for MBA crosslinked copolymers than DP crosslinked copolymers.

The influence of activator, namely, N,N,N′,N′-tetramethylethylenediamine (TMEDA) concentration on the swelling behaviour of superabsorbent copolymers was also studied. Figure 10 shows the swelling behaviour of AAM-KMA superabsorbent copolymers as a function of TMEDA concentration and gelation time. Figure 10 shows that swelling ratio was increased as the TMEDA concentration increased where as the gelation time differed as the TMEDA concentration varies. This may be due to the difficulty in the formation of crosslinking networks (incompatibility in the polymerization system) in the simultaneous polymerization because the TMEDA activates both the polymerization and crosslinking reactions. Figure 11 illustrates the effect of TMEDA concentration on the swelling
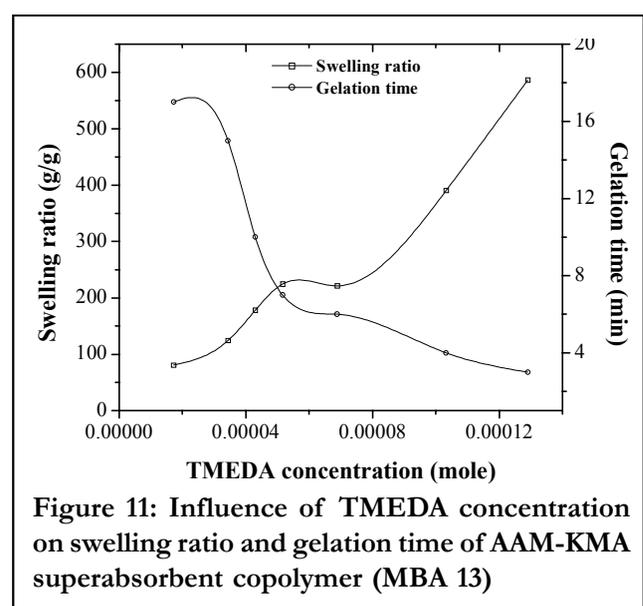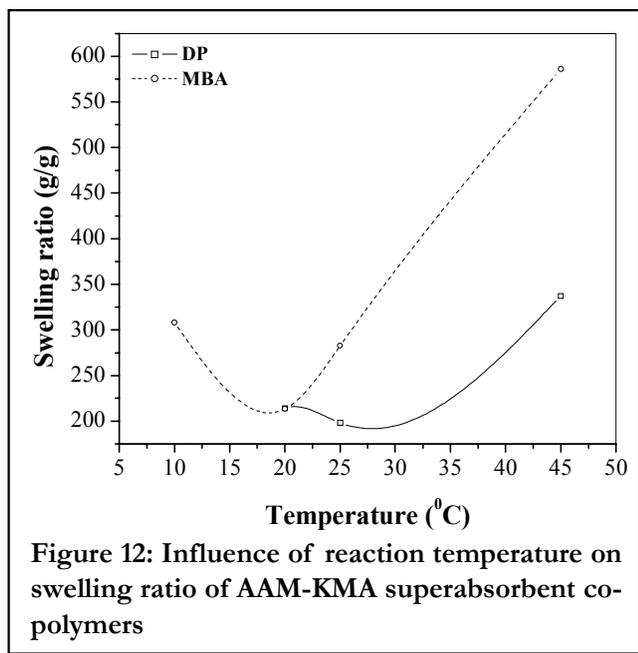
behaviour and gelation time of the AAM-KMA superabsorbent copolymers. The results indicated that the swelling ratio of the superabsorbent copolymers was influenced to a great extent as the concentration of activator varied. As the TMEDA concentration increased the gelation time as well as the swelling ratio was improved enormously. It is noticed that for MBA crosslinked AAM-KMA superabsorbent copolymers, the swelling ratio increased enormously from 80.66 g/g to 586.50 g/g by varying the activator concentration to a small extent from 1.72×10^{-5} moles to 1.29×10^{-4} moles.

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TABLE 4: Influence of pH solution (2-12) on the swelling ratio of the copolymers

Desired pH	Solution 1 (ml)	Solution 2 (ml)	Ionic strength (mol ion dm^{-3})	Swelling ratio of DP copolymer[a] (g/g)	Swelling ratio of MBA copolymer[b] (g/g)
2	97.50	2.50	0.1866	16.04	19.04
3	88.00	12.00	0.1762	12.69	17.54
5	67.00	33.00	0.1521	24.69	36.69
7	49.50	50.50	0.1243	25.28	49.00
11	22.00	78.00	0.0886	Viscous liquid	52.08
12	8.50	91.50	0.0711	Dissolved	48.73

Figure 12: Influence of reaction temperature on swelling ratio of AAM-KMA superabsorbent copolymers

Effect of polymerization temperature

To study the effect of temperature of polymerization reaction on the swelling behaviour of the superabsorbent copolymers, the reactions were conducted at different temperatures ranging from 10^{0}C to 45^{0}C. From this studies it is noted that the copolymers formed at 45^{0}C showed higher swelling ratio than the copolymers prepared at lower temperatures. This behaviour is due to the fact that APS/TMEDA reaction is favourable at 45^{0}C than other temperatures. This variation was clearly observed as shown in figure 12.

Effect of pH effect on swelling ratio of SAPs

To study the behaviour of the AAM-KMA superabsorbent copolymers to changes in the external pH conditions, the copolymer samples were allowed to swell to equilibrium in an aqueous swelling media from pH 2 to 12 and the swelling ratio was calculated by using equation 1. TABLE 4 gives the details of preparative buffer solutions with their ionic strengths along with the influence of pH solution (2-12) on the swelling ratio of the copolymers. From TABLE 4, it is observed that the swelling behaviour of the copolymers is dependent on the pH of external solution. At higher pH values, the samples containing AAM-KMA repeating units, showed highest swelling ratio values than at lower pH values. Increase of buffer solution concentration from pH 7 to 12, the DP crosslinked copolymers dissolved in the buffer solution.

CONCLUSION

The superabsorbent copolymers based on acrylamide and potassium methacrylate were successfully synthesized in aqueous solution under normal atmospheric conditions using APS/TMEDA initiating system. The swelling kinetics of the AAM-KMA superabsorbent copolymers are revealed at different temperatures. The effect of crosslinker type and concentration on the superabsorbent swelling behaviour was studied with DP and MBA. The morphological changes are observed with the variation of crosslinker concentration in the copolymers. The effect of variables such as monomer ratio, crosslinker concentration, initiator, activator, and polymerization temperature on the swelling ratio has been investigated in detail. Further the copolymers saline sensitivity as well as the pH effect was also studied.

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