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Ismail Osman^a; Keyf Seyfullah^a; Corbacioglu Burcu^a

^a Department of Chemical Engineering, Yıldız Technical University, Davutpasa Campus, Faculty of Chemical-Metallurgical, Istanbul, Turkey

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The Effect of PEG on the Water Absorption Capacity and Rate of Superabsorbent Copolymers Based on Acrylic Acid

Ismail Osman
Keyf Seyfullah
Corbacioglu Burcu

Yıldız Technical University, Davutpasa Campus, Faculty of
Chemical-Metallurgical, Department of Chemical Engineering,
Istanbul, Turkey

In this study, acrylic acid-based superabsorbent copolymers were synthesized with different comonomers (PEGs) by inverse suspension polymerization technique. The said superabsorbent copolymers were obtained from the aqueous solution of monomers dispersed in a continuous organic phase. The inverse suspension polymerization was accomplished by the use of aqueous solutions of partly neutralized acrylic acid with a predetermined crosslinker agent/monomer ratio in cyclohexane. Sorbitan monostearate was used as a surfactant, ethylene glycol dimethacrylate (EGDM) either as a comonomer or a crosslinking agent, potassium persulfate as the initiator, and polyethylene glycols (PEG 200, PEG 1000, PEG 4000) were used as pore-making agents. The copolymers (SAPs) were characterized by IR spectroscopy and SEM micrographs. Effect of PEGs on the water absorption capacity, rate of absorption to reach equilibrium, and the time needed to release the water absorbed from the gels were investigated. It was observed that the copolymer based on EGDM-Na acrylate + PEG4000 (A3) gave better results than the other copolyemrs.

Keywords: swelling, water-soluble polymers, radical polymerization, gels, crosslinking

INTRODUCTION

Water absorbent polymers or hydrogels, defined as superabsorbent polymers (SAPs) in the literature, are water-insoluble hydrophilic polymers able to swell and absorb amounts of water, saline solutions,

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Address correspondence to Ismail Osman, Yıldız Technical University, Davutpasa Campus, Faculty of Chemical-Metallurgical, Department of Chemical Engineering, 34210 Eseuler, Istanbul, Turkey. E-mail: ismail@yildiz.edu.tr

or physiological fluids as high as 10–1000 times their own weight. These polymers generally contain carboxylic groups [1, 2].

Because of their excellent characteristics, the superabsorbents [3] are widely used in many applications for manufacture of personal care and hygienic products such as disposable diapers, sanitary napkins, towels, drug delivery systems, surgical pads, and so on [4–7]. The influence of various reaction parameters on the water absorption capacity of SAPs has been investigated by various researchers [8–13]. SAPs should have high water absorbency, high water absorption rate, and good mechanical strength after absorption. For some applications the fluid absorption should be reversible (the polymer should be able to absorb and release the fluid several times). In practice, it is difficult for a polymer to meet all these requirements simultaneously. For instance, by increasing the crosslinking degree, the absorption rate and the gel strength increase but the absorption capacity decreases. Hence, the efforts of industrial research groups are addressed to find a better compromise among the several properties and effects.

In this work, EGDM-Na acrylate copolymers were investigated in the presence of PEG 200, PEG 1000, and PEG 4000. Their absorption capacity, the rate of absorption, and release of water were compared, too.

EXPERIMENTAL

Material and Methods

Preparation of the Aqueous Phase

Acrylic acid was 80% neutralized by caustic solution at $\sim 5^{\circ}\text{C}$. To prepare the pre-suspension, crosslinker (EGDM), which is also used as comonomer in the reaction, and initiator (potassium persulfate) were added and agitated until a clear solution was obtained. PEGs (PEG 200, PEG 1000, PEG 4000) were added into this solution and mixed until dissolution was completed. The PEGs had three different molecular weights of 200, 1000, 4000, and one of them was used in each polymerization.

Preparation of the Continuous Phase

Cyclohexane was selected as the medium for inverse suspension polymerization because of the reaction temperature range ($50\text{--}70^{\circ}\text{C}$). The surfactant (sorbitan monostearate) was dissolved in cyclohexane.

Synthesis of Superabsorbent Polymers

Polymerizations were performed in a 1L, five-necked glass reactor. Reaction temperature was held constant at $52 \pm 1^{\circ}\text{C}$ using a

temperature-regulated water bath. After agitating the organic phase at 52°C for 1 h, the solution phase prepared previously was added into reactor dropwise (10 g/min). The reactor was continuously supplied with nitrogen, so that the polymerization was performed under inert atmosphere. The speed of the mechanical stirrer was set to 650 rpm. Upon monitoring the increase of the reactor internal temperature, it was observed that the polymerization started after 2 min. The reaction temperature was held constant at 52°C for 3 h and then the temperature was increased to 72°C in order to achieve maximum conversion and the polymerization proceeded for a further 1 h. Then, the cooled dispersion was slowly precipitated in a large excess of methanol under vigorous agitation. After settling, the supernatant liquid was separated from the tiny superabsorbent beads.

Extraction

Copolymer beads were extracted in soxhlet with methanol for 24 h. By this way, any possible residual monomers within the copolymer beads were eliminated.

Drying

Upon the elimination of monomers, the copolymers beads were dried at 80°C in a drying oven for 24 h. Four kinds of superabsorbent copolymers were obtained and coded as A (EGDM-Na acrylate), A1 (EGDM-Na acrylate + PEG 200), A2 (EGDM-Na acrylate + PEG 1000), A3 (EGDM-Na acrylate + PEG 4000).

RESULTS AND DISCUSSION

Characterization of Copolymers

The functional groups of the copolymers were investigated using a MATTSON 1000 Fourier-Transform Infrared Spectrophotometer. The copolymer was ground with dried KBr powder. The spectrum of the copolymer confirms the existence of carboxylate functionalities, evidenced by absorption peaks at 1719, 1560, and 1400 cm^{-1} (See Figures 1 and 2). The surface morphology of the copolymers was investigated using a JSM-T 220 A JEOL Scanning Electron Microscope. SEM micrographs of A, A1, and A3 copolymers are given in Figures 3, 4, and 5. As seen in Figure 3, the surface of EGDM-Na acrylate copolymer is rough. As PEG is added to the copolymers their surfaces are rougher than before. It was observed that by adding PEG the porosity increased (Figures 4 and 5).

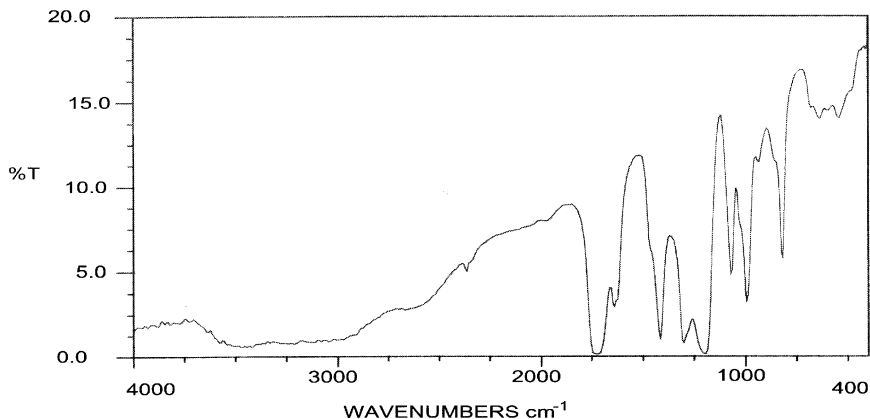


FIGURE 1 FTIR spectrum of acrylic acid.

Determination of Water Absorption Capacity of Copolymers and Absorption Rate Equilibrium

300 g water added to 1 g copolymer samples placed in a 500 ml flask for 48 h were allowed to reach equilibrium absorption. Absorption capacity of the copolymer was determined from the weight differences between swollen and dry samples. Wet samples were weighed after drying by blotting with a piece of filter paper.

The water absorbency, Q (g H₂O/g sample), was calculated by using the following equation [3]:

$$Q(\text{g H}_2\text{O/g copolymer}) = [(m - m_0)/m_0]$$

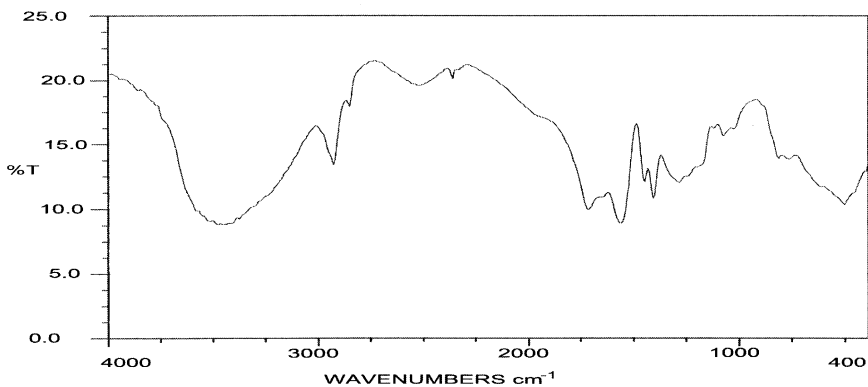


FIGURE 2 FTIR spectrum of A.

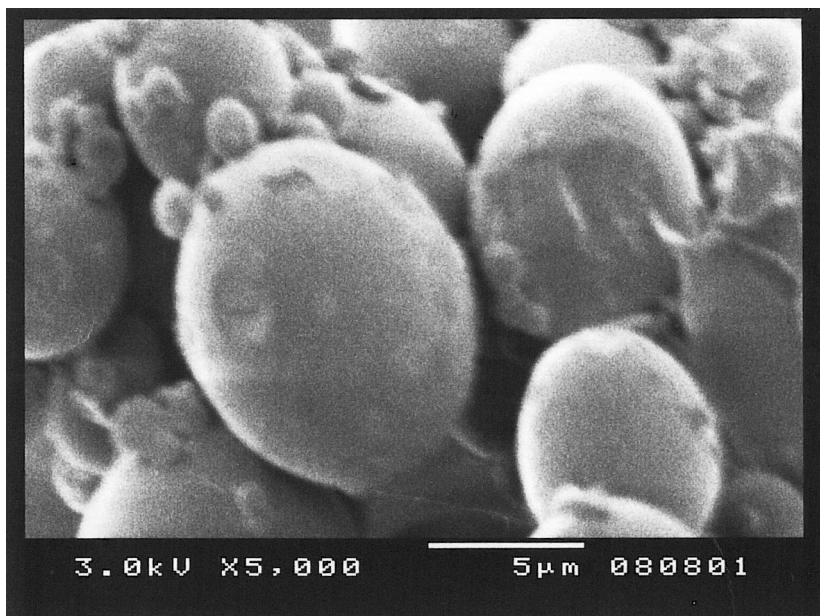


FIGURE 3 SEM micrograph of A.



FIGURE 4 SEM micrograph of A1.

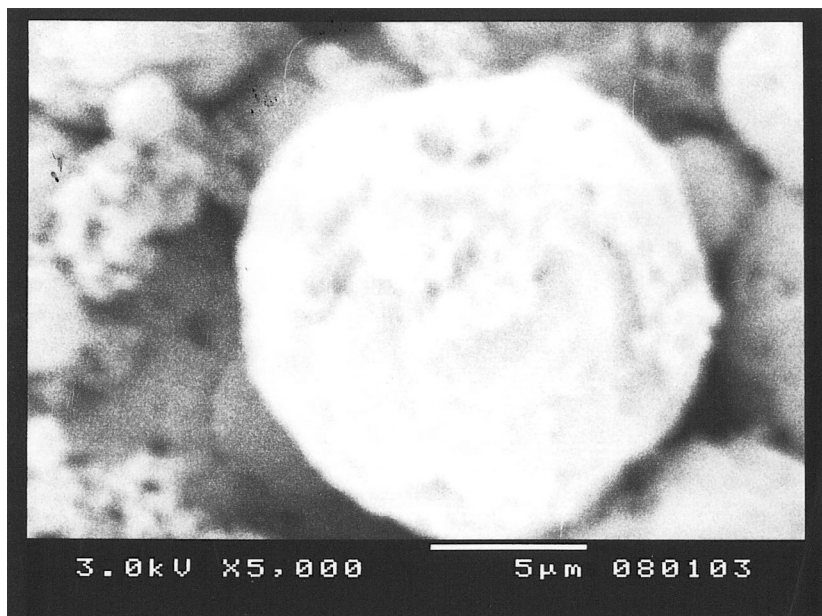


FIGURE 5 SEM micrograph of A3.

Absorbency is expressed as a gram of water retained in the gel per gram of dried gel; in this equation m and m_0 denote the weight of the gel swollen by water and the weight of the absorbent, respectively.

Weight loss of the samples under ambient conditions was determined by weighing them at various time intervals. The absorption capacities and equilibrium absorption rates of the copolymers are given in Table 1. For example, 1 g copolymer A (EGDM-Na acrylate) absorbed 193.49 g water in 1020 s to reach equilibrium concentration.

It was found that there is a good agreement between the values calculated and obtained for absorption capacity and equilibrium

TABLE 1 Absorption Capacity (g Water/g Copolymer) and Equilibrium Absorption Rate (g water/sec) of Copolymers

Copolymers	Absorption capacity I (g)	Water equilibrium absorption rate I (sec.)
A	193.49	1020
A1	201.19	948
A2	209.32	890
A3	234.69	840

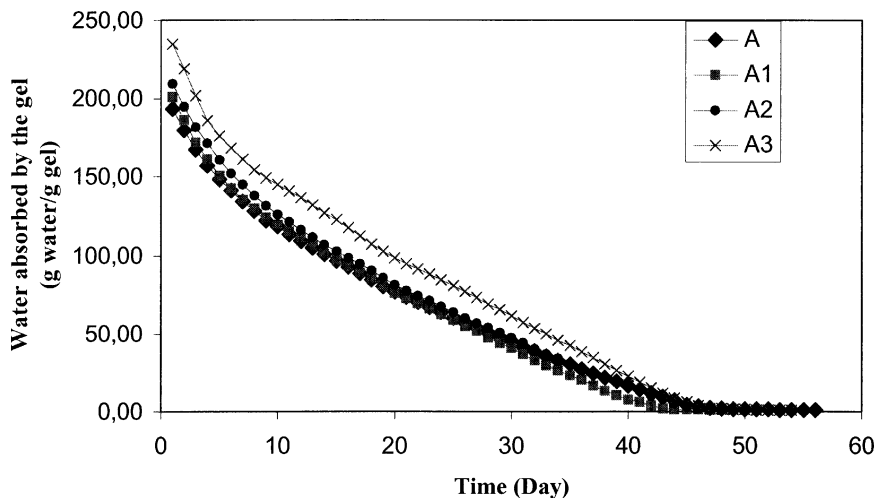


FIGURE 6 Water release of copolymers.

absorption rate of copolymers. It was observed that the absorption capacity of the copolymers increased, but their equilibrium absorption rate decreased with the addition of PEGs.

Figure 6 illustrates how the copolymers released the absorbed water day by day under laboratory conditions. As seen in Figure 6, copolymers obtained by adding PEGs, absorbed much more water than the copolymer including only EGDM-Na acrylate, but they released the water in a shorter time.

CONCLUSIONS

The absorption capacities and equilibrium absorption rates of the copolymers that were synthesized by using PEGs, were found to be higher than the one prepared only from EGDM-NA acrylate (A). The porosities of A1 and A3 are higher than that of EGDM-Na acrylate copolymer according to SEM micrograph. The absorption capacities of the copolymers that were obtained by the addition of polyethylene glycol to EGDM-Na acrylate copolymer (A), increased by about 3.98, 8.27, and 21.3% for A1, A2, A3, respectively.

It was observed that A3 (EGDM-Na acrylate + PEG4000) released the absorbed water more slowly than A1 and A2. It was also observed that the absorption capacities and the absorption rates of the copolymers were affected by the structure of the copolymers and their grain

size. The smaller grain size increased the surface area as well as the absorption capacities and the absorption rates of the copolymers.

It was also seen that high temperature and high humidity lower the release of water from the copolymers.

SYMBOLS

PEG	Polyethylene glycol.
SAP	Super absorbent polymers.
EGDM	Ethylene glycol dimethacrylate.
A	EGDM-Na acrylate copolymer.
A1	(EGDM-Na acrylate + PEG 200) copolymer.
A2	(EGDM-Na acrylate + PEG 1000) copolymer.
A3	(EGDM-Na acrylate + PEG 4000) copolymer.
Na	Sodium.

REFERENCES

- [1] Buchholz, F. L. (1994). CHEMTECH, 1994, 38; Buchholz, F. L. and Peppas, N. A., Ed.; *Superabsorbent Polymers: Science and Technology*, ACS symposium series 573, American Chemical Society, Washington, DC, p.7.
- [2] Ricardo, P. Q., *Macromolecular Chemical Physics.*, **34**, 607, (1994).
- [3] Omidian, H., Hashemi, S. A., Sammes, P. Q., and Meldrum, I., *Polym.* **40**, 1753 (1999).
- [4] Sakiyama, T., Chu, C. H., Fujii, T., and Yano, T., *J. Appl. Polym. Sci.* **50**, 2021 (1993).
- [5] Yoshida, M., Asano, M., and Kumakura, M., *Eur. Polym. J.* **25**, 1197 (1989).
- [6] Shiga, T., Hirose, Y., Okada, A., and Kurauchi, T., *J. Appl. Polym. Sci.* **47**, 113 (1993).
- [7] Trijasson, P. Q., Pith, T., and Lambla, M., *Macromol. Chem., Macromol. Symp.* **35/36**, 141 (1990).
- [8] Askari, F., Nafisi, S., Omidian, H., and Hashemi, S. A., *J. Appl. Polym. Sci.* **50**, 1851 (1993).
- [9] Kiatkamjorwong, S. and Pphunchareon, P., *J. Appl. Polym. Sci.* **72**, 1349 (1999).
- [10] Lee, W.-F. and Tu, Y.-M., *J. Appl. Polym. Sci.* **72**, 1221 (1999).
- [11] Lokhande, H. T., Varadarjan, P. V., and Iyer, V., *J. Appl. Polym. Sci.* **45**, 2031 (1992).
- [12] Padmanabha Raju, M. and Mohana Raju, K., *J. Appl. Polym. Sci.* **80**, 2635 (2001).
- [13] Padmanabha Raju, M., Mohana Raju, K., and Murali, Y., *J. Appl. Polym. Sci.* **85**, 1795 (2002).