

## Fast-swelling superabsorbent polymers with polymerizable macromolecular surfactant as crosslinker

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**ABSTRACT:** Fast-swelling superabsorbent polymers (SAPs) have been prepared by micellar cross-linking copolymerization of acrylic acid and acrylamide using polymerizable macromolecular surfactants (PMSs) as multifunctional crosslinkers, with a foaming technique. Sodium bicarbonate (NaHCO<sub>3</sub>)/acetone and Pluronic F127 were used as foam (or porosity) generators and stabilizer, respectively. It has been found that the PMSs with different polyethylene glycol (PEG) chain length have little effect on the swelling rate of the SAPs, while the foam generators are crucial. In addition, the monomer concentration and the concentration of the PMS are important to the fast swelling rate of the SAPs. For a balance of a relatively fast-swelling rate, high water absorbency under load and low cost in drying, a monomer concentration of above 50%, 0.08% PMS, and 5% NaHCO<sub>3</sub> are preferable. The water absorbency of SAP prepared under optimal conditions could reach half of equilibrium water absorbency in 30 seconds and the saline water absorbency under load could reach 18.4 g/g. © 2016 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2016**, *133*, 44173.

**KEYWORDS:** gels; surfactants; swelling

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### INTRODUCTION

Superabsorbent polymers (SAPs) used in hygiene area need to satisfy the features such as high saline (0.9 wt % NaCl) water absorbency under load (AUL), desirable swollen gel strength, especially fast swelling rate.<sup>1–4</sup> Conventional unmodified acrylic-based SAPs could not satisfy the above demands, and numerous work has been carried out to improve their properties.<sup>4–26</sup> Studies have demonstrated that the swelling rate of the SAPs may be increased through the creation of porosity in the SAPs via the porogen technique, phase separation technique, and foaming technique, and so forth,<sup>24</sup> because the pores in the SAPs generate large surface area to accommodate large amount of water in a short time.

Micellar copolymerization technique has been approved to be a simple and straightforward method to prepare high-toughness hydrogels.<sup>27–32</sup> In the present article, following our previous work,<sup>31,32</sup> fast-swelling SAPs have been prepared by micellar cross-linking copolymerization of acrylic acid and acrylamide using polymerizable macromolecular surfactant (PMS) as multifunctional crosslinker through the foaming technique. Sodium bicarbonate (NaHCO<sub>3</sub>)/acetone and Pluronic F127 were used as foam (or porosity) generators and stabilizer, respectively. The influence of different components, including foam generator, foam stabilizer, monomer concentration, PMS concentration,

and the PEG chain length of PMSs on the swelling behavior of the SAPs were investigated in detail.

### EXPERIMENTAL

#### Materials

3-isocyanatomethyl-3,5,5-trimethylcyclohexyl isocyanate (IPDI), polyethylene glycol [PEG-600 ( $M_w = 600 \text{ g mol}^{-1}$ ), PEG-1000 ( $M_w = 1000 \text{ g mol}^{-1}$ ), PEG-2000 ( $M_w = 2000 \text{ g mol}^{-1}$ )], 4-methoxyphenol, acrylamide (AM), acrylic acid (AA), ammonium persulfate (APS), sodium bicarbonate (NaHCO<sub>3</sub>), acetone, methanol were purchased from Sinopharm Chemical Reagent Co. Ltd., Shanghai, China. Pluronic F127 (PF127, a poly(ethylene oxide)-poly(propylene oxide)-poly(ethylene oxide) tri-block copolymers) was purchased from BASF company, Shanghai, China. 2-isocyanatoethyl acrylate (AOI) was kindly donated by Showa Denko company, Shanghai, China. Dibutyltin dilaurate (DBTDL) was purchased from TCI, Shanghai, China. *N,N,N',N'*-tetramethylethylenediamine (TEMED) was from Aldrich Chemical Company, Shanghai, China. All reagents were used without any further purification.

#### Synthesis of Fast-Swelling SAPs

The PMSs (PMS-600, PMS-1000, PMS-2000) (Figure 1) used in this work were synthesized according to the procedure described

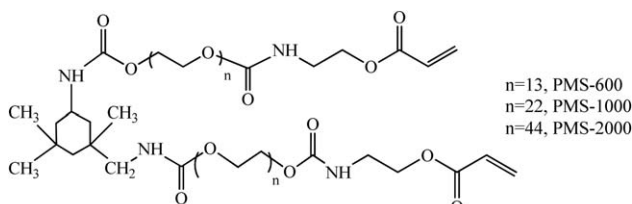
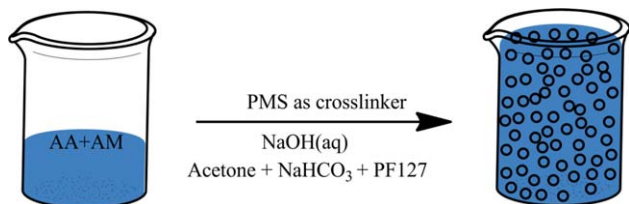


Figure 1. The chemical structure of PMSs.

earlier.<sup>31</sup> The SAPs were prepared in a beaker as shown in Scheme 1. A predetermined amount of 30 wt % NaOH aqueous solution was added dropwise to the stirring 70 wt % AA aqueous solution in a 100 mL beaker cooled with an ice bath for partial neutralization. A proper amount of 10 wt % aqueous solution of PMS-600, PMS-1000, PMS-2000 was added to the above AA solution. Then a given amount of the comonomer AM (the molar ratio of AA and AM = 1:1) and deionized water were carefully added to the monomer mixture. The monomer solution was titrated with 30 wt % of NaOH solution to raise the pH to 5–6. After that, nitrogen ( $N_2$ ) was slowly purged into the beaker to drive off the remaining oxygen while fully stirring. Then appropriate amount of acetone and a proper amount of 10 wt % aqueous solution of PF127 was added to the monomer mixture. After mild agitation for a few minutes, a predetermined amount of 10 wt % of APS solution and 10 wt % of TEMED aqueous solution were added to the mixture. Finally, a given amount of sodium bicarbonate was poured into the mixture with mild agitation until the viscosity increased quickly. The volume of the final product in the beaker usually expanded to 2–10 times the original solution volume. The products were removed from the beaker, and dehydrated first by applying about 5–10 mL of absolute methanol per each gel and finally dried in a forced-draft oven for 24 h at 60°C to reach constant weight. The dried SAPs were ground and screened in the range of 30 to 60 mesh. The detailed polymerization conditions are shown in Table I.

### Water Absorbency Measurements

The accurately weighed 0.050 g sample was immersed into a beaker containing 100 mL 0.9 wt % NaCl aqueous solution and allowed to swell at room temperature and atmospheric pressure. The swollen hydrogel was filtrated through a 100 mesh sieve to remove the non-absorbed water and weighed. Surface water of the filtered swollen particles was dried carefully using filter paper. This measurement was repeated at least three times for each sample. The error range for water absorbency was  $\pm 0.5$  g/



Scheme 1. Schematic synthesis procedure of fast-swelling SAPs. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

Table I. Components for Preparing SAPs

Component	Percentage (%)
Monomer <sup>a</sup>	20–55 <sup>b</sup>
PMS	0.02–0.20 <sup>c</sup>
APS	1.5 <sup>d</sup>
TEMED	1.5 <sup>d</sup>
PF127	1 <sup>d</sup>
Acetone	10 <sup>d</sup>
NaHCO <sub>3</sub>	0–30 <sup>d</sup>

<sup>a</sup>The molar ratio of AM/AA = 1.

<sup>b</sup>Based on the total mass of the solution.

<sup>c</sup>Based on the total mole number of AM and AA.

<sup>d</sup>Based on the total mass of AM and AA.

g. The water absorbency at atmospheric pressure was calculated by the following equation:

$$Q_t = \frac{M - M_0}{M_0}$$

where  $Q_t$  is the water absorbency at time  $t$ .  $M$  is the weight of the swollen hydrogel at time  $t$ .  $M_0$  is the weight of dried sample.

### Water Absorbency under Load (AUL)

The AUL test procedure in saline (0.9 wt % NaCl) solution was accomplished according to a previous report.<sup>16</sup> The accurately weighed 0.100 g sample was spread uniformly on the surface of polyester gauze which was located on a macro-porous iron filter plate placed in a Petri dish. A cylindrical solid load (Teflon,  $d = 28$  mm, 123 g) was placed on the sample ( $P \approx 2000$  Pa). Then, 0.9 wt % saline solution was added to the Petri dish, and swelled for 2 h at room temperature. After that, nonabsorbed solution was removed and the filter plate was weighed again after 45 min. This measurement was repeated at least three times for each sample. The error range for AUL was  $\pm 0.5$  g/g. The value of AUL was calculated according to the following equation:

$$AUL = \frac{M_2 - M_1 - M_0}{M_0}$$

where  $M_0$  denotes the weight of dry sample,  $M_1$  is the weight of the iron filter plate,  $M_2$  is the weight of iron filter plate with the swollen hydrogel.

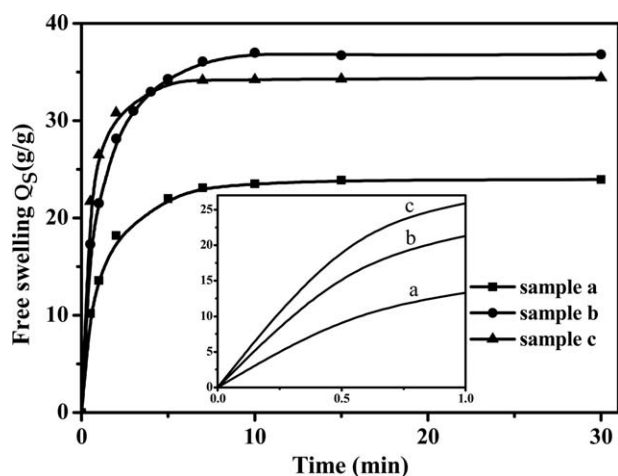
### Morphology

Morphology of the dried gel was examined with a scanning electron microscopy (SEM). Dried SAP powders were coated with a thin layer of palladium gold alloy in a Hummer I Sputter Coater and imaged in a SEM instrument (FE-SEM, S-4700, Hitachi, Japan).

## RESULTS AND DISCUSSION

### Effect of Porosity Generators and Pore Stabilizer on the Fast-Swelling Property of SAPs

To make fast-swelling SAPs, the construction of porous structure is crucial. It has been shown that when acetone and sodium bicarbonate are used as porosity generators consecutively in



**Figure 2.** Swelling kinetics of different SAPs. PMS-600 was used as the crosslinker. The detailed polymerization condition was described in Table II. Sample (a), prepared without porosity generators or pore stabilizer. Sample (b), prepared under porosity generators only. Sample (c), prepared under porosity generators and pore stabilizer PF127.

the process, a remarkable synergistic effect was observed in the swelling rate of the products.<sup>3</sup> Acetone and sodium bicarbonate are also used as porosity generators in this study. The SAP samples b and c prepared under porosity generators absorb water more quickly than sample a prepared under no porosity generators (Figure 2, Table II). The water absorbency of SAP samples b and c could reach half of equilibrium water absorbency less than 30 seconds. The increase of swelling rate under porosity generators can be interpreted by the creation of porous structure which generates much larger surface area to quickly accommodate larger amount of water.<sup>24</sup> The SEM micrographs of the SAPs prepared under different conditions are presented in Figure 3. The particle surface of

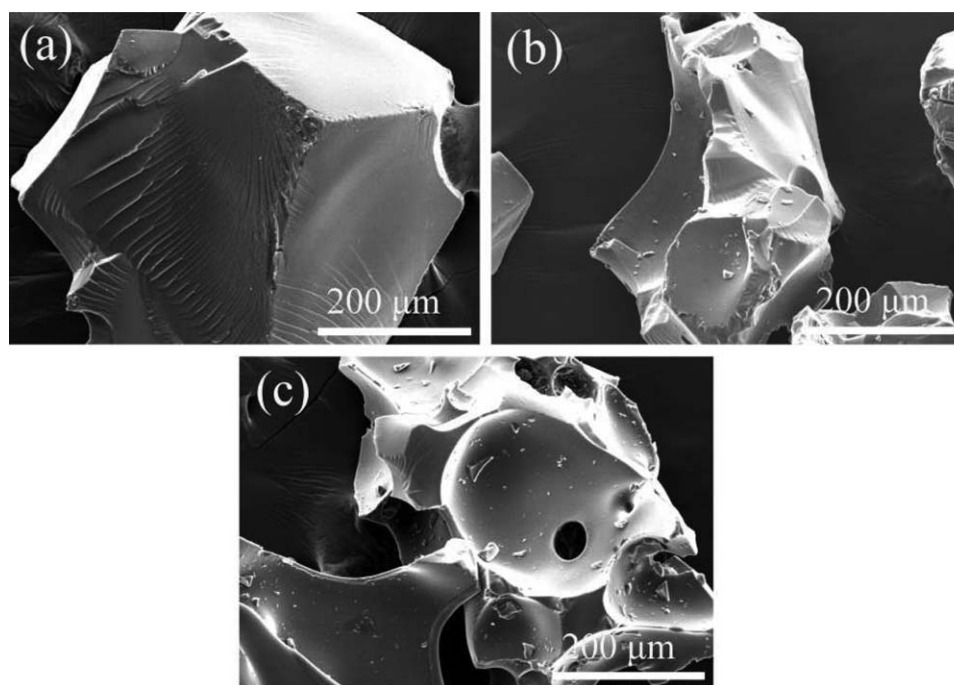
**Table II.** The Components for the Preparation of Different SAPs

Sample	[NaHCO <sub>3</sub> ] (wt %)	[Acetone] (wt %)	[PF127] (wt %)	[Monomer] (wt %)
a	0	0	0	45
b	20	10	0	45
c	20	10	1	45

sample a [Figure 3(a)] is quite smooth. In contrast, samples prepared with porosity generators have clear rough surfaces resulting from the collapse of the pores.

In addition to porosity generators, the stabilizer of the pores formed is also important. Comparing the swelling rate of sample b and c (Figure 2), it can be found that the swelling rate of sample c prepared with pore stabilizer PF127 is slightly higher than that of sample b prepared with no pore stabilizer. This result is consistent with the SEM photographs shown in Figure 3, where sample c has a much rougher surface than sample b.

The effect of pore stabilizer (PF127) concentration on the swelling capacity of the SAP samples was listed in Table III. With the increase of pore stabilizer (PF127) concentration from 0 wt % to 2 wt %, saline-absorbency of the SAP samples within 30 s of swelling increases from 17.3 g/g to 23.8 g/g and the AUL value increases from 17.2 g/g to 19.2 g/g, but the equilibrium saline-absorbency decreases from 37 g/g to 29.2 g/g. Increase of the swelling rate with increasing pore stabilizer (PF127) concentration is related to the foam stabilizing effect of pore stabilizer (PF127).<sup>24</sup> On the other hand, pore stabilizer (PF127) may solubilize PMS-600 to a higher degree which may eliminate the large consumption of PMS-600 at the early stage of the polymerization and increase the cross-link density of SAPs. The increased cross-link density contributes to



**Figure 3.** SEM micrographs of dried SAP samples: (a) sample a, (b) sample b, (c) sample c.

**Table III.** Saline-Absorbency of the Samples Prepared with Different Pore Stabilizer Concentration

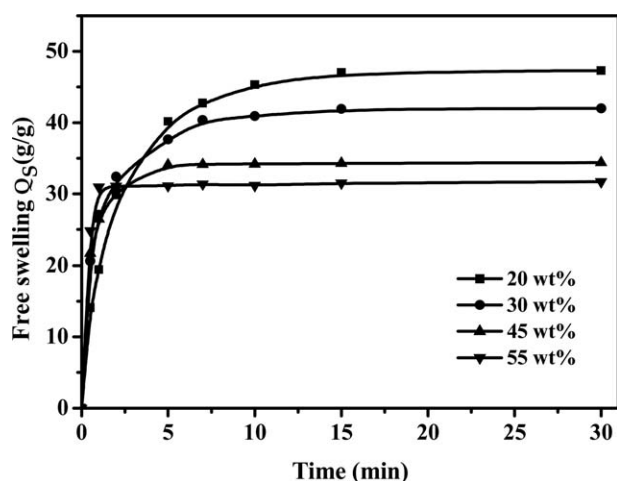
PF127 Concentration (wt %)	Saline-absorbency within 30 s (g/g)	Saline-absorbency after equilibrium (g/g)	AUL (g/g)
0	17.3	37.0	17.2
0.5	19.3	35.6	17.4
1	21.7	34.4	18.4
2	23.8	29.2	19.2

the decrease of the equilibrium saline-absorbency of SAP and the increase of the AUL value. For a balance of a relatively fast-swelling rate and the equilibrium saline-absorbency, 1 wt % PF127 was used in this study to make fast-swelling SAPs.

#### Effect of Different Monomer Concentration on Swelling Properties

Figure 4 displays the swelling kinetics of the samples which were produced from different monomer concentration. Variation of the monomer concentration caused considerable difference in the swelling properties. With the increase of the monomer concentration from 20 wt % to 30 wt %, the swelling rate increases significantly. However, a further increase of monomer concentration has little effect on the swelling rate. Increase of the swelling rate with increasing monomer concentration is most probably related to the gelation time of the solution (Table IV). The higher monomer concentration leads to a higher viscosity of the solution. This will give rise to a shorter gelation time, thereby more foams could be trapped in the gel.

The swelling capacity of the SAP samples produced from different monomer concentration is listed in Table IV. With increasing monomer concentration from 20 wt % to 55 wt %, load-free saline-absorbency decreases from 47.3 g/g to 31.7 g/g, but the AUL value maintains at about 18 g/g, that is, the higher monomer concentration has little effect on the AUL value. A

**Figure 4.** Swelling kinetics of the SAP samples produced from different monomer concentration. Polymerization condition:  $[\text{NaHCO}_3] = 20$  wt %,  $[\text{Acetone}] = 10$  wt %,  $[\text{PF127}] = 1$  wt %,  $[\text{PMS-600}] = 0.16$  mol %,  $[\text{M}] = 20\text{--}55$  wt %.**Table IV.** Gelation Time, Saline-Absorbency of the SAP Samples Prepared with Different Monomer Concentration

Monomer Concentration (wt %)	Gelation time (s)	Saline-Absorbency <sup>a</sup> (g/g)	AUL (g/g)
20	50	47.3	18.0
30	35	42.0	18.3
45	31	34.4	18.4
55	28	31.7	18.4

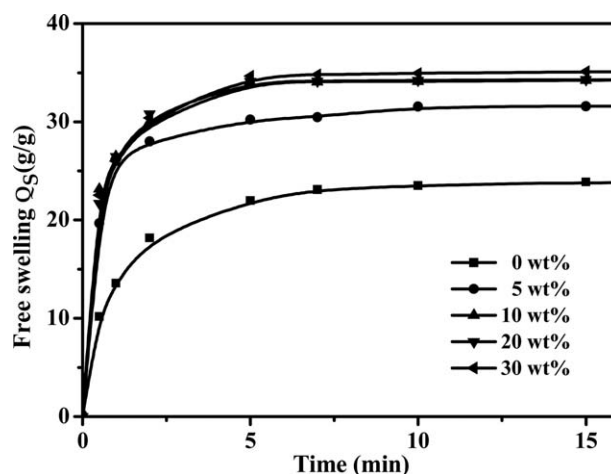
<sup>a</sup> Load-free swelling.

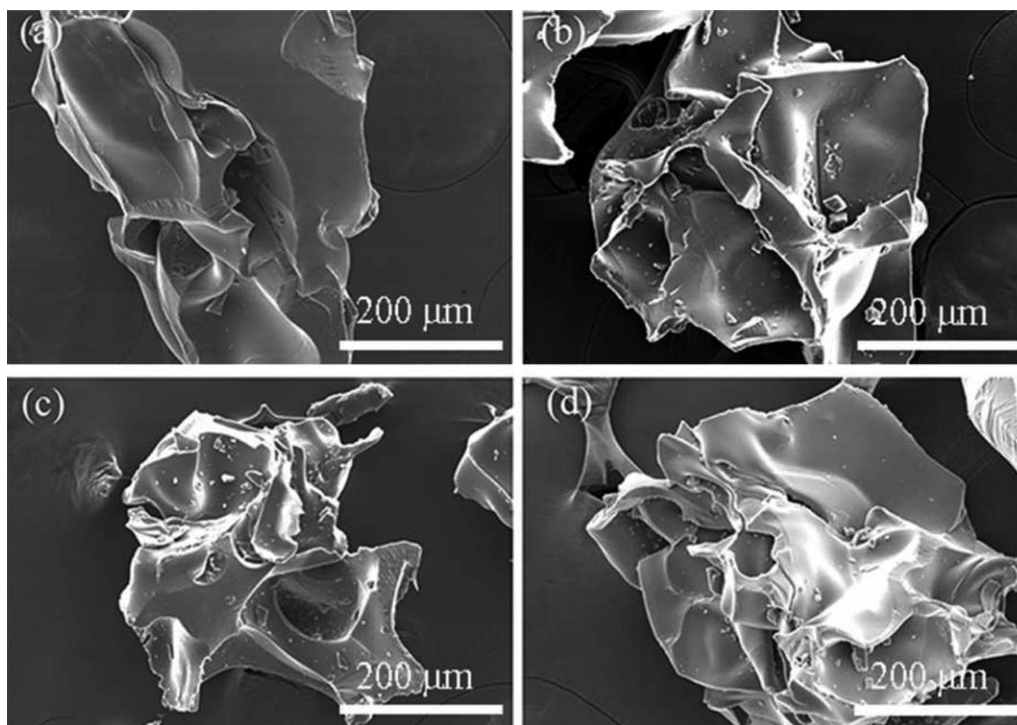
higher monomer concentration causes a more vigorous heat release and self-cross-linking reactions, thereby a higher cross-link density,<sup>25</sup> which contributes to the decrease of the load-free saline-absorbency of SAP. On the other hand, the increased cross-link density increases the strength of the hydrogel which should increase the AUL value. For a balance of fast swelling capability, high AUL value and low cost in drying, a monomer concentration of above 50% is preferable.

#### Effect of the Amount of $\text{NaHCO}_3$ on Swelling Properties

The swelling kinetics of the samples synthesized using different amount of  $\text{NaHCO}_3$  is shown in Figure 5. The swelling rate of the SAP made in the absence of  $\text{NaHCO}_3$  is much slower than the ones with  $\text{NaHCO}_3$ . With the addition of only 5 wt %  $\text{NaHCO}_3$ , the swelling rate of the SAP increased significantly. The swelling rate does not change much with a further increase in the amount of  $\text{NaHCO}_3$ . The SEM micrographs of the samples prepared using different amount of  $\text{NaHCO}_3$  shown in Figure 6 verified the above result. It is observed from Figure 6 that particle surfaces of the dried samples with larger amount of  $\text{NaHCO}_3$  are clearly rougher than the one with only 5% of  $\text{NaHCO}_3$ .

The swelling capacity of the samples synthesized using different amount of  $\text{NaHCO}_3$  is listed in Table V. With the increase of

**Figure 5.** Swelling kinetics of the SAP samples synthesized using different amount of  $\text{NaHCO}_3$ . Polymerization condition:  $[\text{Acetone}] = 10$  wt %,  $[\text{PF127}] = 1$  wt %,  $[\text{PMS-600}] = 0.16$  mol %,  $[\text{M}] = 45$  wt %,  $[\text{NaHCO}_3] = 0\text{--}30$  wt %.



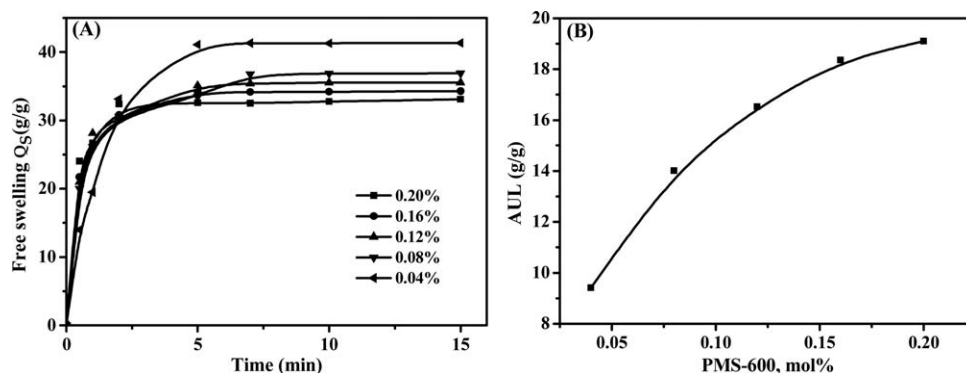
**Figure 6.** SEM micrographs of dried SAP samples synthesized using different amount of  $\text{NaHCO}_3$ . (a) 5 wt %, (b) 10 wt %, (c) 20 wt %, (d) 30 wt %.

**Table V.** Saline-Absorbency of the SAP Samples Synthesized Using Different Amount of  $\text{NaHCO}_3$

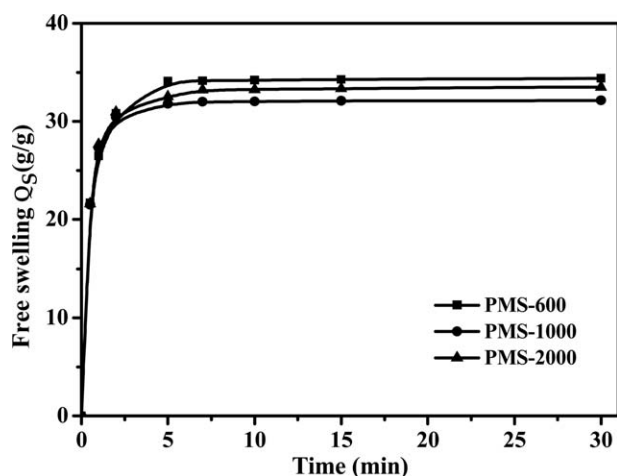
Amount of $\text{NaHCO}_3$ (wt %)	Saline-absorbency (g/g)	AUL (g/g)
0	24.0	19.7
5	31.7	20.1
10	34.5	18.8
20	34.3	18.4
30	35.2	18.8

AUL, water absorbency under load.

$\text{NaHCO}_3$  from 0 to 5%, the saline absorbency increases rapidly from 24.0 g/g to 31.7 g/g, then slowly to 34.5 g/g when the amount of  $\text{NaHCO}_3$  added was 10%. Above 10%, the increase of  $\text{NaHCO}_3$  has no effect on saline absorbency. The AUL value slightly decreases from 20.1 g/g to 18.8 g/g when the amount of  $\text{NaHCO}_3$  added increases from 5% to 10%, then has essentially no change after that. The above observation is apparently a result of increasing porosity created when the amount of  $\text{NaHCO}_3$  increases from 5% to 10%. Above 10%, probably no more porosity can be created. Besides, the porosity structure slightly reduced the strength of the gel, giving rise to a reduction of AUL value. For a balance of a relatively fast-swelling and high AUL value, 5%  $\text{NaHCO}_3$  is preferable.



**Figure 7.** (A) Swelling kinetics of SAP samples synthesized using various concentration of PMS-600. (B) The influence of PMS-600 concentration on saline-absorbency under load. Polymerization condition:  $[\text{NaHCO}_3] = 20$  wt %,  $[\text{Acetone}] = 10$  wt %,  $[\text{M}] = 45$  wt %,  $[\text{PF127}] = 1$  wt %,  $[\text{PMS-600}] = 0.04\text{--}0.20$  mol %.



**Figure 8.** Swelling kinetics of the SAP samples synthesized using PMS-600, PMS-1000 and PMS-2000. Polymerization condition:  $[\text{NaHCO}_3] = 20$  wt %,  $[\text{Acetone}] = 10$  wt %,  $[\text{PF127}] = 1$  wt %,  $[\text{M}] = 45$  wt %,  $[\text{PMS}] = 0.16$  mol %.

#### Effect of PMS Concentration on Swelling Properties

Swelling kinetics of SAP samples synthesized using various concentration of PMS-600, ranging from 0.04–0.20 mole % based on the total mole number of AA and AM, is shown in Figure 7(A). It can be seen that when the amount of PMS-600 is larger than 0.04%, the swelling rate increases significantly and then almost has no change. Obviously, the increase of PMS-600 concentration leads to a reduction of gelation time, and more bubbles can be trapped in the hydrogel. As a result, higher porosity can be created, leading to higher absorption rate. But this effect is not measurable when the concentration of PMS-600 is larger than 0.08%. On the other hand, higher PMS-600 concentration gives rise to higher cross-link density, which is apparently the reason of saline-absorbency reduction as shown in Figure 7(A) and the increase of AUL value, as shown in Figure 7(B).<sup>33,34</sup> From the above results, it is clear that the concentration of PMS-600 plays a crucial role as in SAPs without porosity generators.

#### Effect of PMSs with Different PEG Chain Length on Swelling Properties

The swelling kinetics of the SAP samples synthesized using PMSs (PMS-600, PMS-1000, PMS-2000) with different hydrophilic PEG chain length at the same mole fraction is shown in Figure 8. With the increase of the hydrophilic PEG chain length, the swelling rate does not have measurable change, indicating that the PMSs with different hydrophilic PEG chain have little effect on the foaming process. This result also suggests, from another side, that the use of porosity generator is a dominant factor controlling the swelling rate.

#### CONCLUSIONS

In the present work, polymerizable macromolecular surfactants (PMSs) were used as multifunctional crosslinker in the micellar cross-linking copolymerization of acrylic acid and acrylamide to prepare fast-swelling superabsorbent SAPs, in the presence of

foam (or porosity) generators and stabilizer. In addition to the dominant factor of porosity generators, monomer concentration and the concentration of PMS are also important to the fast swelling rate of the SAPs. It has also found that the PMS with different PEG chain length at the same mole fraction does not have measurable influence on the swelling rate of the SAPs.

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